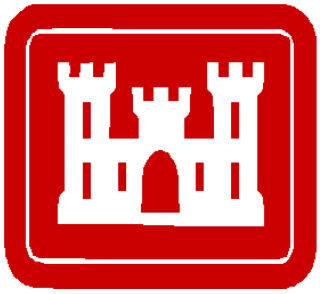
FINAL PROGRAMMATIC UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN

REMEDIAL INVESTIGATION AT FOUR KNOWN PFAS SITES AND PRELIMINARY ASSESSMENT/SITE INSPECTION AT SUSPECTED PFAS SITES

FORMER SENECA ARMY DEPOT
ROMULUS, SENECA COUNTY, NEW YORK



Prepared for:

U.S. Army Corps of Engineers
Huntsville District

Contract W912DY-20-D-0017
Delivery Order: W912DY21F0310

Version 0.2
May 2023

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FINAL PROGRAMMATIC UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN

REMEDIAL INVESTIGATION AT FOUR KNOWN PFAS SITES AND PRELIMINARY ASSESSMENT/SITE INSPECTION AT SUSPECTED PFAS SITES FORMER SENECA ARMY DEPOT ROMULUS, SENECA COUNTY, NEW YORK

Prepared for:

U.S. Army Corps of Engineers
Huntsville District
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LIST OF ATTACHMENTS

LIST OF ACRONYMS AND ABBREVIATIONS

°C	degree Celsius
AFFF	aqueous film-forming foam
AOC	Area of Concern
B.A.	Bachelor of Arts
BRAC	Base Realignment and Closure
B.S.	Bachelor of Science
CA	corrective action
CAS	Chemical Abstracts Service
CASRN	CAS Registry Number
CV	calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CHMM	Certified Hazardous Materials Manager
CIH	Certified Industrial Hygienist
CoC	chain of custody
COCs	contaminants of concern
COR	Contracting Officer Representative
CQCSM	Contractor Quality Control Systems Manager
CQM	Construction Quality Manager
cy	cubic yards
DERP	Defense Environmental Restoration Program
DL	detection limit
DoD	Department of Defense
DQI	data quality indicator
DQO	data quality objective
EIS	Extracted Internal Standard
ELAP	Environmental Laboratory Accreditation Program
ELLE	Eurofins Lancaster Laboratories Environment Testing, LLC
EOD	Explosive Ordnance Disposal
EPA	U.S. Environmental Protection Agency
ESI	expanded site investigation
FS	Feasibility Study
ft	foot/feet
FTS	fluorotelomer sulfonate
HDPE	high-density polyethylene
HGL	HydroGeoLogic, Inc.
HRR	historical record review
ICAL	initial calibration
ICV	initial calibration verification
ID	identification

ISC	instrument sensitivity check
LC/MS/MS	liquid chromatography/tandem mass spectrometry
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDC	Laboratory Data Consultants
LOD	limit of detection
LOQ	limit of quantitation
LTM	long-term monitoring
M.S.	Master of Science
MEC	munitions and explosives of concern
MD	matrix duplicate
mL	milliliter
MS	matrix spike
MSD	matrix spike duplicate
mV	millivolt
MW	monitoring well
NA	not applicable
NCP	National Contingency Plan
NE	not established
NEtFOSAA	n-ethylperfluorooctanesulfonamidoacetic acid
ng/L	nanograms per liter
NMeFOSAA	n-methylperfluorooctanesulfonamidoacetic acid
NYSDEC	New York State Department of Environmental Conservation
ORP	oxidation reduction potential
OU	Operable Unit
PA	Preliminary Assessment
PAH	polynuclear aromatic hydrocarbons
PAL	Project Action Limit
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
Parsons	Parsons Corporation
P.E.	Professional Engineer
PFAS	perfluoroalkyl and polyfluoroalkyl substances
PFBA	perfluorobutanoic acid
PFBS	perfluorobutanesulfonic acid
PFDA	perfluorodecanoic acid
PFDoA	perfluorododecanoic acid
PFDS	perfluorodecanesulfonic acid
PFHpA	perfluoroheptanoic acid
PFHpS	perfluoroheptanesulfonic acid
PFHxA	perfluorohexanoic acid

PFHxS	perfluorohexanesulfonic acid
PFNA	perfluorononanoic acid
PFNS	perfluorononanesulfonic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctanesulfonic acid
PFOSA	perfluorooctanesulfonamide
PFPA	perfluoropentanoic acid
PFPeA	perfluoropentanoic acid
PFPS	perfluoropentanesulfonic acid
PFTeDA	perfluorotetradecanoic acid
PFTrDA	perfluorotridecanoic acid
PFUnA	perfluoroundecanoic acid
P.G.	Professional Geologist
Ph.D.	Doctor of Philosophy
PM	Project Manager
PP	proposed plan
PWS	Performance Work Statement
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality Systems Manual
RI	Remedial Investigation
RPD	relative percent difference
SEAD	Former Seneca Army Depot
SEDA	Former Seneca Army Depot Activity
SI	Site Inspection
SOP	standard operating procedure
SPP	Systematic Project Planning
SSHO	Site Safety and Health Officer
SVOCs	Semi-volatile organic compounds
TBD	to be determined
UFP	Uniform Federal Policy
USACE	U.S. Army Corps of Engineers
UXO	Unexploded Ordnance

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FINAL PROGRAMMATIC UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN REMEDIAL INVESTIGATION AT FOUR KNOWN PFAS SITES AND PRELIMINARY ASSESSMENT/SITE INSPECTION AT SUSPECTED PFAS SITES FORMER SENECA ARMY DEPOT ROMULUS, SENECA COUNTY, NEW YORK

EXECUTIVE SUMMARY

Introduction

This Programmatic Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP) was prepared by HydroGeoLogic, Inc. (HGL) for a Remedial Investigation (RI) at Four Known Per- and Polyfluoroalkyl Substances (PFAS) Sites and Preliminary Assessment (PA)/Site Inspection (SI) at 34 Suspected PFAS Sites. The RI will be conducted at four sites where PFAS have been detected, while the SI will be performed at the 34 Former Seneca Army Depot Activity (SEDA) sites to determine if there is evidence of PFAS release. The SI will address the 34 suspected PFAS sites and any new sites discovered during the PA/Historical Records Review (HRR). These investigations will assess whether the SEDA has been impacted by the release of aqueous film-forming foam (AFFF) during on-base firefighting or and other Department of Defense (DoD) related site-use or other non-DoD related disposal activities of materials containing perfluorooctanoic acid (PFOA) and/or PFAS that occurred in the past. HGL has prepared this UFP-QAPP under contract with the U.S. Army Corps of Engineers (USACE) Huntsville District, Contract Number W912DY-20-D-0017, Delivery Order W912DY21F0310. The work is being executed in accordance with the Performance Work Statement issued with task order award.

This UFP-QAPP is specific to the SEDA sites and meets the requirements and elements set forth in the U.S. Environmental Protection Agency (EPA) guidance document titled *Uniform Federal Policy for Quality Assurance Project Plans* (Intergovernmental Data Quality Task Force, 2005) with the optimized worksheets developed in 2012 (Intergovernmental Data Quality Task Force, 2012). It also includes supplemental information and requirements as necessary to support site-specific objectives.

Background

The HGL team, including our teaming partner Parsons Corporation (Parsons), will be providing environmental investigation services through the Performance Work Statement (PWS), which falls within the Defense Environmental Restoration Program (DERP) Installation Restoration Program at various sites on SEDA. SEDA was closed under the Base Realignment and Closure (BRAC) Program in 1995. Work will be performed in accordance with the Comprehensive Environmental

Response, Compensation, and Liability Act (CERCLA), and the National Contingency Plan. In January of 2016, EPA requested the Army sample for PFAS in groundwater in two areas of SEDA. Additional sampling was conducted through PFAS SIs confirming the presence of PFAS contamination in 2018 and an Expanded SI (ESI) was conducted in phases in those areas between May 2019 and March 2021. Additional background information is included in Worksheet #10.

Scope of Work

The project objectives established in the PWS are as follows:

- Perform a PA for the entire SEDA to determine if there is historical evidence of release of PFAS at additional sites. A HRR Report will present the results of the PA;
- Perform SIs on the sites listed in Table 1 below. Any sites identified during the PA will be added to this list; and
- Perform an RI at SEAD-25, SEAD-26, SEAD-122E/122D and the former Fire House (Building. 103). The purpose of the RI is to confirm/define the nature and extent of PFAS, to collect sufficient data to identify and quantify exposure pathways through development of a Risk Assessment and for these sites.

Table 1. Site Inspection Sites

SEAD-002-R-01, EOD Area #2 and #3; OU11	SEAD-22, Sewerage Treatment Plant # 314, OU14
SEAD-003-R-01, EOD #1 (SEAD-57), OU11	SEAD-23, Open Burning Grounds, OU2
SEAD-007-R-01, Grenade Range, OU11	SEAD-24, Abandoned Powder Burning Pits, OU13
SEAD-3, Incinerator Cooling Water Pond, OU1	SEAD-45, Open Detonation Grounds, OU17
SEAD-5, Sewage Sludge Storage Pile, OU13	SEAD-46, Small Arms Range, (aka 3.5-inch Rocket Range), OU11
SEAD-6, Abandoned Ash Landfill, OU1	SEAD-58, Debris Area Near Booster Station 2131, OU14
SEAD-7, Shale Pit. OU14	SEAD-59, Fill Area West of Building 315, OU6
SEAD-8, Non-Combustible Fill Area, OU1	SEAD-64A, Garbage Disposal Area South of Storage Pad, OU12
SEAD-9, Old Scrap Wood Site, OU14	SEAD-64B, Garbage Disposal Area South of Classification Area, OU14
SEAD-10, Scrap Wood Site, OU14	SEAD 64C, Garbage Disposal Area, OU14
SEAD-11, Old Construction Debris Landfill, OU8	SEAD 64D, Garbage Disposal Area West of Building 2203, OU14
SEAD-14, Refuse Burning Pits (2 units), OU1	SEAD-67, Dump Site East of Sewage Treatment Plant #4, OU14
SEAD-15, Abandoned Solid Waste Incinerator (Building 2207), OU1	SEAD-68, Old Pest Control Shop (Building S-335), OU14
SEAD-16, Building S311, Abandoned Deactivation Furnace, OU4	SEAD-69, Building 606 Disposal Area, OU14
SEAD-17, Building 367, Active Deactivation Furnace, OU4	SEAD-70, Former Building T-2110, Filled Area, OU11
SEAD-20, Sewage Treatment Plant #4, OU14	SEAD-122D, Airfield Hot Pad Spill
SEAD-21, Sewage Treatment Plant # 715, OU14	Fire House Building 722

OB = Open Burning OU = Operable Unit UXO = Unexploded Ordnance EOD = Explosive Ordnance Disposal

WORKSHEETS #1 AND #2 TITLE AND APPROVAL PAGE

Programmatic UFP-QAPP Seneca Army Depot
Document Title

U.S. Corps of Engineers (USACE)
Lead Organization

Denise Rivers, HGL
Preparer's Name and Organizational Affiliation

2405 N. Courtenay Parkway, Suite 203, Merritt Island, FL 32953, 910-233-8460, drivers@hgl.com
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May 2023
Preparation Date (Month/Year)

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Signature / Date
Alexandria Lambert / USACE
Printed Name / Organization

USACE, Huntsville District PM:

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Charles H. Heaton, P.E. / USACE
Printed Name / Organization

USACE, New York District PM:

Digitally signed by GALLO.CHRISTOPHER.T.1604778820 Date: 2023.05.23 09:22:09 -04'00'

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Christopher Gallo, USACE
Printed Name / Organization

Federal Regulatory Agency:

ROBERT MORSE Digitally signed by ROBERT MORSE Date: 2023.05.24 11:45:22 -04'00'

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Robert Morse, EPA Remedial Project Manager
Printed Name / Organization

Federal Regulatory Agency:

Lynn E Arabia Digitally signed by Arabia, Lynn Date: 2023.05.24 12:19:40 -04'00'

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Lynn Arabia, EPA CHMM
Printed Name / Organization

HGL PM:

Blaum, John Digitally signed by Blaum, John Date: 2023.05.23 08:36:09 -04'00'

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John Blaum, P.E. / HGL
Printed Name / Organization

HGL Project Chemist:

Denise M. Rivers

Signature / Date
Denise Rivers, Ph.D. / HGL
Printed Name / Organization

WORKSHEET #1 AND #2 (CONTINUED)
TITLE AND APPROVAL PAGE

Site Name/Project Name: Seneca Army Depot, New York
Site Location: Romulus, Seneca County, New York
Site Number/Code: not applicable
Operable Unit: not applicable
Contract Number: W912DY20F0017
Work Assignment Number (optional): W912DY21F0310

1. Identify guidance used to prepare the UFP-QAPP: UFP-QAPP; DoD Quality Systems Manual (QSM) Version 5.4 or more recent version.
2. Identify regulatory program**:** CERCLA
3. Identify approval entities: USACE
4. The UFP-QAPP is programmatic.
5. List dates of scoping sessions that will be held:
   - November 3 – Systematic Project Planning (SPP) Meeting No. 1
   - Additional SPP Meetings TBD
6. List dates and titles of UFP-QAPP documents written for previous site work, if applicable: Not applicable.
7. List organizational partners (stakeholders): USACE, EPA, New York State Department of Environmental Conservation (NYSDEC), New York State Department of Health.
8. List data users: HGL, USACE, EPA, NYSDEC and New York State Department of Health.

WORKSHEETS #3 AND #5
PROJECT ORGANIZATION AND UFP-QAPP DISTRIBUTION

Distribution:

The following is the distribution list for this UFP-QAPP.

Name	Organization	Role	Phone	Email Address
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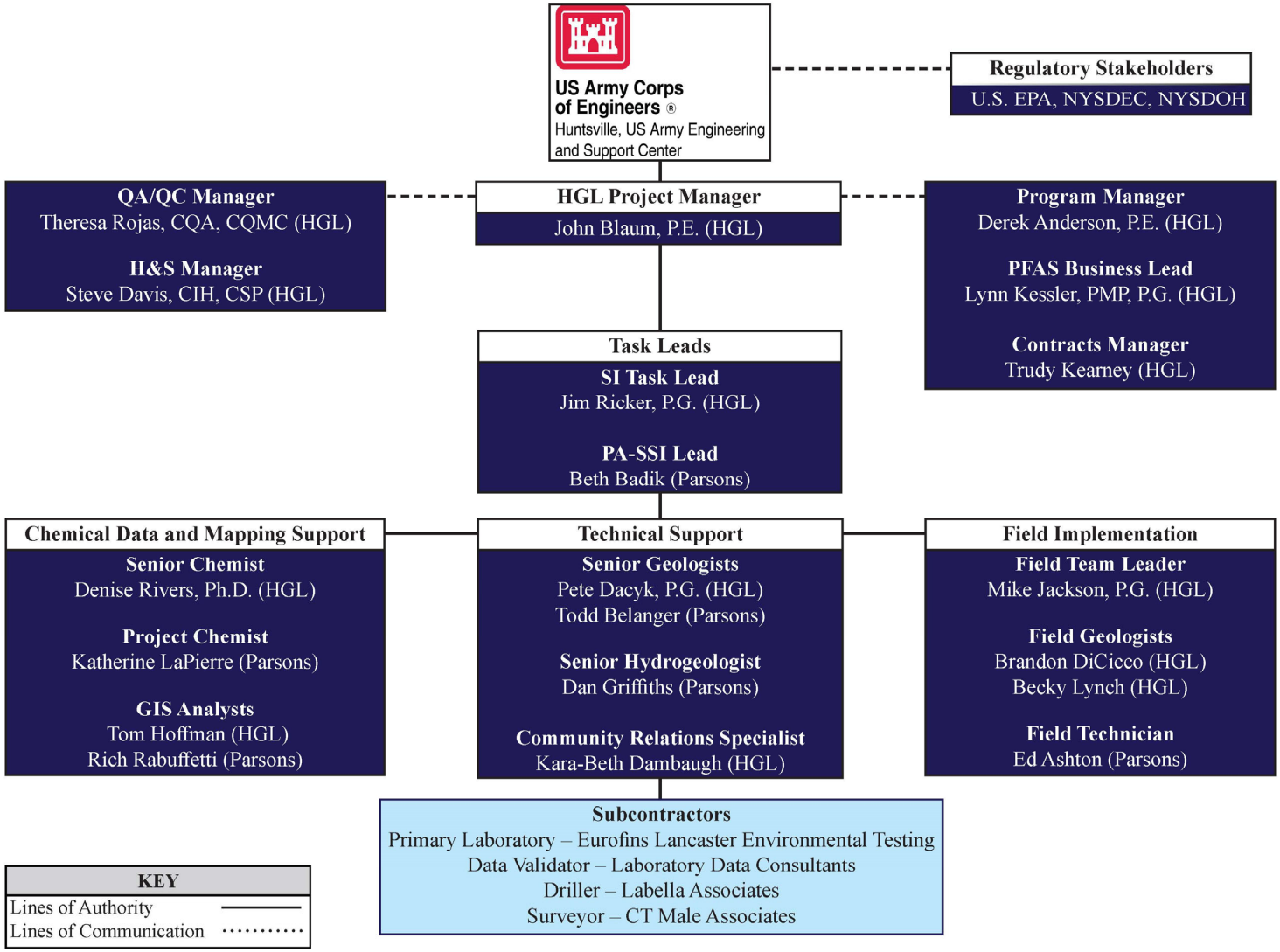
COR = Contracting Officer Representative ELLE = Eurofins Lancaster Laboratories Environment Testing, LLC LDC = Laboratory Data Consultants PM = Project Manager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Field Team Leader
Mike Jackson, P.G. (HGL)
Field Geologists
Brandon DiCicco (HGL)
Becky Lynch (HGL)
Field Technician
Ed Ashton (Parsons)
Subcontractors
Primary Laboratory – Eurofins Lancaster Environmental Testing
Data Validator – Laboratory Data Consultants
Driller – Labella Associates
Surveyor – CT Male Associates
KEY
Lines of Authority
Lines of Communication

WORKSHEETS #4, #7, AND #8
PROJECT PERSONNEL QUALIFICATIONS AND SIGN-OFF SHEET

ORGANIZATION: HGL

Name/Organization	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
John Blaum, P.E.	HGL PM	B.S., Civil Engineering Experience: 34 years	P.E. NYS	
Eric Dambaugh	CQCSM	B.S., Earth Science Experience: 30 years	P.G.	
Edie Scala-Hampson	Health and Safety Manager	B.S., Biology Experience: 43 years	CIH #2929CP CHMM #06859	
Denise Rivers, PhD	Project Chemist	Ph.D., Environmental Chemistry B.A., Chemistry Experience: 15 years		

CHMM = Certified Hazardous Materials Manager
CIH = Certified Industrial Hygienist
CQMSM = Contractor Quality Control Systems Manager
B.S. = Bachelor of Science
Ph.D. = Doctor of Philosophy
P.E. = Professional Engineer
P.G. = Professional Geologist
QC = quality control

WORKSHEETS #4, #7, AND #8 (CONTINUED)
PROJECT PERSONNEL QUALIFICATIONS AND SIGN-OFF SHEET

ELLE (Primary Laboratory)

Name	Project Title/Role	Education/Experience	Signature/Date
Richard Karam	Laboratory Director	B.S., Environmental Studies Experience: 20 years	
Kenneth Boley	Quality Assurance (QA) Manager	B.S., Chemistry Experience: 20 years	
Vanessa Badman	PM	B.S., Biology Experience: 19 years	

B.S. = Bachelor of Science

LDC (Data Validation)

Name	Project Title/Role	Education/Experience	Signature/Date
Pei Geng	PM	M.S., Chemistry Experience: 28 years	

M.S. = Master of Science

WORKSHEET #6
COMMUNICATION PATHWAYS

Communication Drivers	Responsible Affiliation	Name	Telephone Number	Procedure (Timing, Pathway to & from, etc.)
Prime Contractor point of contact	HGL	John Blaum	jblaum@hgl.com 518-877-3793	The PM will communicate project-related issues, including changes in schedule, changes in scope of field works or delays, and recommendation to stop work, to USACE by phone or email within 24 hours. The PM will provide project information to USACE through monthly progress calls and reports, email updates, teleconference calls, or other progress meetings.
Field Progress Report	HGL	John Blaum	jblaum@hgl.com 518-877-3793	The PM will provide daily updates to USACE while in the field.
QAPP changes prior to and during fieldwork	HGL	Denise Rivers	Drivers@hgl.com 910-233-8460	If errors or changed conditions require the modification of the QAPP before fieldwork begins, the Project Chemist will prepare revised text in collaboration with the PM. All changes to the QAPP will require final approval from USACE.
Stop Work due to safety issues	HGL	Edie Scala--Hampson	escala-hampson@hgl.com 847-409-6384	If unsafe work conditions are noted, the Site Safety and Health Officer (SSHO) will stop work immediately. Work will not be allowed to resume until the unsafe condition is corrected. The SSHO will notify the Health and Safety Manager and PM immediately when a stop work situation is encountered. The PM will notify USACE immediately.
Field Corrective Actions (CA)	HGL	Eric Dambaugh	edambaugh@hgl.com 518-877-3785	CAs resulting from either failure to follow QAPP requirements or due to changes in site conditions will be documented by the Field Team Leader; the Field Team Leader will communicate the need for CA to the PM and CQCSM on the same business day. The Field Team Leader may initiate an interim CA in the field subject to final approval by the PM and CQCSM.
Sample Receipt Discrepancies	Laboratory	Vanessa Badman	Vanessa.Badman@ET.EurofinsUS.com 717-556-9762	The laboratory PM will communicate discrepancies in the sample receipt to the Project Chemist on the same business day that the discrepancy is identified. The Project Chemist will instruct the laboratory PM on the appropriate course of action.
Laboratory QC discrepancies	HGL	Denise Rivers	Drivers@hgl.com 910-233-8460	The Project Chemist will prepare variance request in collaboration with the laboratory PM and the PM for transmittal to USACE for approval.

WORKSHEET #6 (CONTINUED)
COMMUNICATION PATHWAYS

Communication Drivers	Responsible Affiliation	Name	Telephone Number	Procedure (Timing, Pathway to & from, etc.)
Analytical CAs	HGL	Denise Rivers	drivers@hgl.com 910-233-8460	Need for laboratory CAs will be determined by the Project Chemist and/or laboratory PM and will be documented in memoranda to the PM and USACE.
Data Verification issues (e.g., incomplete records)	HGL	Denise Rivers	drivers@hgl.com 910-233-8460	The Project Chemist will verify that the laboratory data complies with the requirements of the QAPP. The Project Chemist will work with the laboratory PM to ensure missing information are delivered to on a timely basis.
Data Validation issues (e.g., noncompliance with procedures)	LDC	Pei Geng	pgeng@lab-data.com 760-827-1100	LDC will perform data validation and provide data validation reports to the Project Chemist within 21 business days. Severe issues, such as noncompliance with procedures, will be communicated via email to the Project Chemist within 1 week of data package receipt.
Data Review CAs	HGL	Denise Rivers	drivers@hgl.com 910-233-8460	Final analytical data cannot be released until any required validation is complete and the Project Chemist has approved the release.
Daily Field Reports	HGL	Mike Jackson	mjackson@hgl.com 518-877-3789	Field Team Leader will provide HGL PM with daily reports of progress, potential issues, and corrective actions taken.

WORKSHEET #9
PROJECT SCOPING SESSION PARTICIPANTS SHEET

Date of planning session: 02 December 2021

Location: Virtual – Cisco WebEx

Purpose: Review proposed approach for RI and SI through SPP process

Participants:

Name	Organization	Title/Role	Email
Jim Moore	USACE	Base Environmental Coordinator	James.T.Moore@usace.army.mil
Chris Gallo	USACE	Project Manager	Christopher.T.Gallo@usace.arm.mil
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John Blaum	HGL	PM	jblaum@hgl.com
Jim Ricker	HGL	Technical Leader – SI	jricker@hgl.com
Beth Badik	Parsons	Technical Leader – RI	Beth.Badik@parsons.com
Todd Belanger	Parsons	Senior Geologist	Todd.Belanger@parsons.com
Denise Rivers	HGL	Project Chemist	drivers@hgl.com

COR = Contracting Officer Representative
PM = Project Manager

Notes/Comments: Review of the RI and SI sample locations

Consensus decisions made: ESI will most likely move to and RI. Modifications to current figures and also include better representation of the RI and SI soil, surface water and sediment samples on figures.

Action Items: Further develop the figures and tables based on discussions for inclusion in ESI and SI site specific work plans and Programmatic UFP-QAPP.

WORKSHEET #10
CONCEPTUAL SITE MODEL

Environmental Problem

In 1995, SEDA was designated for closure under the DoD BRAC process. Between 1941 and 2000, SEDA was owned by the U.S. Government and operated by the Department of the Army. SEDA began its primary mission of the receipt, maintenance, and supply of ammunition in 1943. After World War II, the primary mission of the facility was the receipt, storage, maintenance, and disposal of military items. PFAS-containing material were suspected of being used at SEDA, and four sites were known to have PFAS contamination and subject to an RI. At this time, 34 sites are suspected of having PFAS contamination based on past use and will undergo an SI. All sites were identified through a review of the Solid Waste Management Unit classification report (Parsons, 1994). Currently, all 34 sites listed in the PWS will proceed to the SI stage.

The HGL team also will conduct a PA for any additional sites that may have had PFAS use and prepare, submit, and gain acceptance of a detailed HRR report in accordance with the requirements outlined in the PWS. The team will review the relevant historical documents provided by the Government in the Administrative Record and aerial photographs to determine the likelihood of PFAS impacts based on historical use at each site. The site documents will be evaluated to determine if there are additional suspect PFAS sites and document this in the HRR. These data will be used to further refine the SI requirements. HGL will prepare the HHR report, which will document the following:

- Historical site uses;
- Potential sources of PFAS;
- Available information on possible fate and transport mechanisms; and
- Recommendations for SI.

The HGL team will complete the SIs at 34 suspected sites to determine if PFAS are present in surface soil, surface water, sediment, and/or groundwaters. The NYSDEC has focused its PFAS investigations on three site types: existing hazardous waste sites; landfills or disposal areas; and wastewater treatment facilities or sludge disposal areas. The proposed sampling locations and selected media (groundwater, surface water, soil, or sediment) described in the following subsection and shown on the figures were selected based on the following:

- Previous site operations;
- The size of the site, so adequate data could be gathered to determine presence or absence of PFAS and groundwater flow direction, if not known; and
- Proximity to other SI or RI sites and existing wells.

The 34 sites, by site location type, are identified as follows:

Munitions and Explosives of Concern (MEC) Sites (10)

- SEAD-002-R-01 EOD Area #2 and #3, OU11;
- SEAD-003-R-01, EOD #1, (SEAD-57); OU11
- SEAD-16, Building S311, Abandoned Deactivation Furnace, OU4;
- SEAD-17, Building 367, Active Deactivation Furnace, OU4;
- SEAD-23, Open Burning Grounds, OU2;
- SEAD-24, Abandoned Powder Burning Pits, OU13;
- SEAD-45, Open Detonation Grounds, OU17;
- SEAD-46, Small Arms Range, (aka 3.5-inch Rocket Range), OU11;
- SEAD-007-R-01, Rifle Grenade Range, OU11; and
- Fire House Bldg. 722.

Ash Landfill Sites (5)

- SEAD-3, Incinerator Cooling Water Pond, OU1;
- SEAD-6, Abandoned Ash Landfill, OU1;
- SEAD-8, Non-Combustible Fill Area, OU1;
- SEAD-14, Refuse Burning Pit (2 Units), OU1; and
- SEAD-15, Abandoned Solid Waste Incinerator (Building 2207), OU1.

Sewage Related Sites (4)

- SEAD-5, Sewage Sludge Storage Pile, OU13;
- SEAD-20, Sewerage Treatment Plant #4, OU14;
- SEAD-21, Sewerage Treatment Plant #715, OU14; and
- SEAD-22, Sewerage Treatment Plant #314, OU14.

Disposal/Spill Sites (15)

- SEAD-7, Shale Pit, OU14;
- SEAD-9, Old Scrap Wood Site, OU14;
- SEAD-10, Scrap Wood Site, OU14;
- SEAD-11, Old Construction Debris Landfill, OU8;
- SEAD-58, Debris Area Near Booster Station 2131, OU14;
- SEAD-59, Fill Area West of Building 315, OU6;
- SEAD-64A, Garbage Disposal Area South of Storage Pad, OU12;

- SEAD-64B, Garbage Disposal Area South of Classification Area, OU14;
- SEAD 64C, Garbage Disposal Area, OU14;
- SEAD 64D, Garbage Disposal Area West of Building 2203, OU14;
- SEAD-67, Dump Site East of Sewerage Treatment Plant #4, OU14;
- SEAD-68, Old Pest Control Shop (Building S-335), OU14;
- SEAD-69, Building 606 Disposal Area, OU14;
- SEAD-70, Former Building T-2110, Filled Area, OU11; and
- SEAD-122D, Airfield Hot Pad Spill.

The fieldwork associated with the SIs will include the installation of overburden monitoring wells, monitoring well development and survey, and the collection of soil, groundwater, surface water, and sediment samples to characterize the nature and extent of potential contamination. Samples will be analyzed for PFAS compounds by draft EPA method 1633, and all samples will be submitted to ELLE Laboratory. It is important to note that the proposed sample locations shown on the figures are preliminary and subject to change based on the viability of existing wells and their accessibility in the field.

Where there is the potential for encountering material potentially presenting an explosive hazard (e.g., MEC), which includes UXO and/or discarded military munitions, anomaly avoidance shall be implemented as applicable at the sites.

Site Location and History

SEDA is located approximately 40 miles south of Lake Ontario in Seneca County, New York. SEDA lies immediately west of Romulus, New York, approximately 12 miles south of the villages of Waterloo and Seneca Falls, and 2.5 miles north of Ovid, New York. The two closest major cities are Rochester and Syracuse, New York. Before the U.S. Government acquired the land in 1941, the property was privately owned and was used principally as homesteads and for agriculture.

SEDA is in an uplands area, where the ground elevation ranges from approximately 600 feet (ft) above mean sea level (amsl) along the western boundary of the former installation to nearly 760 ft amsl in the central portion of the eastern boundary. The uplands area where SEDA is located forms a divide that separates two of the New York Finger Lakes: Cayuga Lake to the east and Seneca Lake to the west. Sparsely populated farmland covers most of the surrounding area. New York State Highways 96 and 96A border SEDA to the east and west, respectively.

SEDA geology is characterized by gray Devonian shale with a thin weathered zone, where it contacts the overlying mantle of Pleistocene glacial till. The predominant surficial geologic unit is dense glacial till. The till is distributed across the entire facility and ranges in thickness from less than 2 ft to as much as 15 ft, although generally it is only a few feet thick. The till is characterized by brown to gray-brown silt, clay, and fine sand with a few fine-to-coarse gravel-sized inclusions of weathered shale. The geologic cross-sections suggest that a groundwater divide exists halfway between the two lakes. SEDA is located on the western slope of this divide; therefore, regional

groundwater flow is expected to be primarily westward toward Seneca Lake. Local hydrogeology is generally consistent with regional hydrogeology.

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

Environmental Setting

Climate

A cool climate exists at SEDA with temperatures ranging from an average of 31°F in January to 69°F in July. Marked temperature differences are found between daytime highs and nighttime lows during the summer and portions of the transitional seasons. Precipitation is well distributed, averaging approximately 3 inches per month. This precipitation is derived principally from cyclonic storms, which pass from the interior of the county through the St. Lawrence Valley. Seneca, Cayuga, and Ontario Lakes provide a significant amount of winter precipitation and moderate the local climate. The annual average snowfall is approximately 100 inches. Wind velocities are moderate, but during the winter months, there are numerous days with sufficient winds to cause blowing and drifting snow. The most frequently occurring wind directions are westerly and west southwesterly (Parsons, 2021).

Topography and Surface Water

SEDA is located in an uplands area, where the elevation ranges from approximately 600 feet (ft.) National Geodetic Vertical Datum (NGVD 1929) along the western boundary of the Depot to nearly 760 feet NGVD 1929 in the central portion of the eastern boundary. The uplands area where SEDA is located forms a divide separating two of the New York Finger Lakes: Cayuga Lake on the east and Seneca Lake on the west. Sparsely populated farmland covers most of the surrounding area. In general, the Airfield AOCs are located on the western side of the topographic divide and SEAD-25 and SEAD-26 are on the eastern side. The former Firehouse, SEAD-25 and SEAD-26 are located in the east-central portion of the former SEDA. The topography has low relief and slopes to the southwest (Firehouse, SEAD-25) and west (areas west of SEAD-25 and SEAD-26). The Airfield is located in the southwest corner of SEDA and is generally level with a slight slope to the west.

Geology

The typical geology beneath the local area is a thin mantle of glacial till overlying shale bedrock. Generally, the overburden consists of a thin layer of high fines content soils (where undisturbed) underlain by glacial till (unsorted clay, silt, sand and gravel) a few feet thick to approximately 15

feet in thickness that drains poorly. Minor amounts of fill are present, but the fill is difficult to distinguish from the native till and is likely the same material only reworked. Bedrock is soft, fissile, shale bedrock of the Moscow Formation for the AOCs in the east. The Ludlowville and Moscow Formation bisects the airfield with the northern and central SEAD-AOCs within the Ludlowville Formation and the southern SEADs within the Moscow Formation. The shales within both formations have poor intergranular porosity and the flow of groundwater is expected to move through millimeter scale horizontal and vertical zones of porosity (bedding plane fractures and joints) on a localized scale (inches to several feet) (Merin, 1992; Parsons ES, 1998). The upper 10 feet of the bedrock typically has low rock quality designations (RQD) of less than 30%. RQD typically increases with depth (Parsons ES, 1998; Parsons, 2022b).

Hydrogeology
Groundwater is found seasonally in the overburden/weathered bedrock zone (subject to precipitation); however, the water in the wells is not considered potable due to low well yield. Wells installed in the area would not meet the requirements for a standard well yield test which includes a minimum 4-hour period of stabilized (± 0.5 feet) drawdown while pumping at a constant flow rate (NYSDOH, 2021). Recharge of the underlying shallow saturated zone is dependent on precipitation. Rainwater or snow melt slowly infiltrates into the till/weathered bedrock water bearing zone; however, during larger precipitation events, the infiltration rate is likely not high enough, and overland flow transports excess precipitation to local drainage ditches and low areas. During the PFAS ESI (Parsons, 2022a), wells installed in the upper water bearing zone were installed to depths typically 15 feet bgs or less and wells installed in the lower water bearing zone (shallow fractured bedrock) were to depths of approximately 60 feet bgs although two wells (MW26-28D and MW26-32D) were extended to a depth of 100ft and 80ft bgs, respectively, due to a lack of recharge. Within the lower water bearing zone, well yields were observed to be poor with slow recharge and are not considered potable based on their inability to meet the state regulations for water wells. Based on discussions with local drillers in the areas, wells at a depth of greater than 150 feet are typically needed to obtain sufficient well yields.

Previous Investigations

Examples of additional site-specific information may include but are not limited to:

- Parsons Engineering Science, Inc., 1995. *Final Expanded Site Inspection – Seven High Priority Solid Waste Management Units SEAD 4, 16, 17, 24, 25, 26, and 45. Seneca Army Depot Activity, Romulus, New York.* December.
- Parsons Engineering Science, Inc., 1998. *Final Remedial Investigation Report for the Fire Training and Demonstration Pad (SEAD-25) and the Fire Training Pit and Area.* May 1998.
- Parsons, 2004. *Record of Decision (ROD) for the Fire Training and Demonstration Pad (SEAD-25) and the Fire Training Pit and Area (SEAD-26)*. July.
- Parsons, 2005. *Final Remedial Design Work Plan and Design Report (RDR) for the Fire Training and Demonstration Pad (SEAD-25) and the Fire Training Pit and Area (SEAD-26)*. November.

- Parsons, 2006a. *Final Construction Completion Report (CCR) for the Fire Training and Demonstration Pad (SEAD-25) and the Fire Training Pit and Area (SEAD-26), Seneca Army Depot Activity*. November.
- Parsons, 2006b. *Final Land Use Control Remedial Design for SEAD 27, 66, 64A.*
- Parsons, 2006c. *Round 1 – Long-Term Monitoring Results for SEAD-25 and SEAD-26; ContractFA8903-04-D-8675, Delivery Order 0012, CDRL A001H. Technical Memorandum.* May.
- Parsons, 2006d. *Round 2 – Long-Term Monitoring Results for SEAD-25 and SEAD-26 at Seneca Army Depot Activity, Romulus, New York. Technical Memorandum.* December.
- Parsons, 2007. *Draft Annual Report for the Fire Training and Demonstration Pad (SEAD-25) and the Fire Training Pit and Area (SEAD-26), Seneca Army Depot Activity*. February.
- Parsons, 2011a. *Round 7 (3Q2010) – Long-Term Monitoring Results for SEAD-25 at the Seneca Army Depot Activity, Romulus, New York. Technical Memorandum*. January.
- Parsons, 2011b. *Round 8 (1Q2011) – Long-Term Monitoring Results for SEAD-25 at the Seneca Army Depot Activity, Romulus, New York. Technical Memorandum*. March.
- Parsons, 2011c. *Draft Fourth Long-Term Monitoring and Site Assessment Report, Fire Training and Demonstration Pad (SEAD-25), Seneca Army Depot Activity*. May.
- Parsons, 2013a. *Final Well Decommissioning Report. Ash Landfill Operable Unit, SEAD-4, SEAD-5,SEAD-11, SEAD-12, SEAD-13, SEAD-24, SEAD-25, SEAD-26, SEAD-27, SEAD-48, SEAD-59, SEAD-63,SEAD-67, SEAD-70, SEAD-71, SEAD-119B, SEAD-121C, & SEAD-122B Seneca Army Depot Activity*. March.
- Parsons, 2013b. *Final 2012 Annual Long-Term Monitoring Report. Fire Training and Demonstration Pad (SEAD-25), Seneca Army Depot Activity.* April.
- Parsons, 2014. *Draft 2013 Annual Long-Term Monitoring Report. Fire Training and Demonstration Pad (SEAD-25), Seneca Army Depot Activity*. April.
- Parsons, 2015a. *Final 2014 Annual Long-Term Monitoring Report. Fire Training and Demonstration Pad (SEAD-25), Seneca Army Depot Activity*. February.
- Parsons, 2015b. *Draft 2015 Annual Long-Term Monitoring Report. Fire Training and Demonstration Pad (SEAD-25), Seneca Army Depot Activity*. August.
- Parsons, 2016. *Draft 2016 Annual Long-Term Monitoring Report. Fire Training and Demonstration Pad (SEAD-25), Seneca Army Depot Activity*. October.
- Parsons, 2017. *Final UFP-QAPP, Seneca Army Depot Activity, Long-Term Monitoring.* May.
- Parsons, 2018. *Draft 2017 Annual Long-Term Monitoring Report. Fire Training and Demonstration Pad (SEAD 25), Seneca Army Depot Activity*. June.
- Parsons, 2019. *Draft 2018 Annual Long-Term Monitoring Report. Fire Training and Demonstrations Pad (SEAD 25), Seneca Army Depot Activity*. February.

- Parsons, 2021. Final Five-Year Review, Seneca Army Depot. SEAD 1, 2, 5, 12, 13, 16, 17, 23, 25, 26, 27, 39, 40, 41, 43, 44A, 44B, 46, 52, 56, 59, 62, 64A, 64B, 64C, 64D, 66, 67, 69, 71, 121C, 121I, 122B, 122E, 002-R-01, 003-R-01, 007-R-01, and the Ash Landfill Operable Unit (SEADs 3, 6, 8, 14, and 15). Seneca Army Depot Activity. August 2021.
- Parsons, 2018. Final 2017 PFAS Site Inspection Report. SEAD 25 (Fire Training and Demonstration Pad), SEAD 26 (Fire Training Pit and Area), and SEAD 122E (Airfield and Refueling Pads). Seneca Army Depot Activity. January 2018.
- Parsons, 2022. Final PFAS Expanded Site Investigation Report. Former Fire House (Building 103), SEAD 25 (Fire Training and Demonstration Pad), SEAD 26 (Fire Training Piet and Area), Seneca Army Depot Activity. March 2022.

Previous Remedial Actions

SEAD-23 Open Burning Grounds

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

Currently, the long-term monitoring (LTM) component of the remedy is being implemented by Parsons. LTM began in November 2007, and 10 sampling events have been completed; the most recent of which was conducted in October 2015. LTM at the OB Grounds site was initially scheduled to occur on a quarterly basis. The results of the first four LTM rounds were combined and summarized in an annual report, in which the recommended frequency of monitoring was recommended to change from quarterly to annually. Based on comments received from EPA and NYSDEC in 2009, the Army authorized the performance of an inspection of Reeder Creek. The monitoring frequency of groundwater was agreed upon by EPA and NYSDEC in February 2010 to be conducted annually. Subsequent to Round 5, investigations at the OB Grounds have included yearly groundwater sampling and inspection of both the soil caps and Reeder Creek.

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

applicable screening criteria. LTM will continue until closure is negotiated between the Army and the regulators.

SEAD-25 FIRE TRAINING AND DEMONSTRATION PAD

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

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

A total of 1,722 cy (2,600 tons) of soil were excavated from the pad and the swale at SEAD-25 and disposed off-site at Ontario County Landfill. The pad excavation was backfilled with approximately 793 cy of on-site fill material and 168 cy of fill material obtained from an off-site source and restored to the existing grade. The onsite soil borrow source is believed to have been generated from excavated underground utility work completed by New York State Electric and Gas (NYSEG) at uncontaminated locations in the Administration Area of SEDA. The soil was excavated along East Patrol Road; between 2nd Steet and South Street; along Quarters Drive; a segment of 1st Avenue; and 3rd Avenue.

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

SEAD-25 was investigated for PFAS during the PFAS SI (Parsons, 2018) and PFAS ESI (Parsons, 2022a). During the SI, the investigation focused on the former training area where groundwater from 12 existing wells was analyzed for a targeted suite of 14 PFAS compounds. Twelve of 14 PFAS compounds were detected at SEAD-25. PFOS and PFOA were detected in all 12 wells

sampled at SEAD-25. The combined concentrations of PFOS and PFOA exceeded EPA lifetime health Seneca Army Depot Activity Work Plan for the PFAS Remedial Investigation June 2022 15 advisory level (70 ng/L) in all 12 wells. The maximum detection of PFOS was 8,300 ng/L in well MW25-8. The maximum detection of PFOA was 89,000 J ng/L in well MW25-2. During the PFAS ESI, additional perimeter wells were added to delineate the extent of PFAS contamination and soil and surface water samples were collected (Parsons, 2022a). The PFAS impacts to shallow groundwater are concentrated around the former SEAD-25 site boundary and fire training pad area and approximately 500 ft downgradient to the west and southwest of the site. The ESI defined a shallow groundwater plume extending southwest of SEAD-25 with plume extents bounded to the west, southwest, and south by wells with PFAS concentrations below the New York state (NYS) MCL. Impacts were not observed in the deeper water bearing zone at the source area or downgradient of the source area.

ASH LANDFILL SITES – (SEAD-3, Incinerator Cooling Water Pond; SEAD-6, Abandoned Ash Landfill; SEAD-8, Non-Combustible Fill Area; SEAD-14, Refuse Burning Pit; and SEAD-15, Abandoned Solid Waste Incinerator (Building 2207).

Prior to the listing of SEDA on the National Priorities List, two removal actions were performed at the Ash Landfill. The first action was the removal of a former 1,000-gallon underground storage tank (UST) that was used to store heating oil and was located on the east side of the abandoned Incinerator Building. The second, a Non-Time Critical Removal Action (NTCRA), was conducted by the Army in 1994/1995 and consisted of the excavation and thermal treatment of soil impacted with volatile organic compounds (Parsons, 2005c).

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

Following the completion of the ROD and during the Remedial Design phase, permeable biowalls were selected as the remedy in place of implementing large scale ZVI walls, which are still in place from the demonstration study. Since a wall material other than iron was selected, the Army conducted a review of the biowall remedy effectiveness one year after the walls are installed. Subsequent annual reviews were performed until the first Five-Year Review. The first four rounds of groundwater sampling were performed in the first year of LTM and were completed in January 2007, March 2007, June 2007, and November 2007. As part of the Year 1 report, the Army recommended that the frequency of LTM events at the Ash Landfill OU be reduced from quarterly to semi-annually, which was approved by EPA and NYSDEC. Ten years of groundwater monitoring and 21 sampling events have been completed; the most recent sampling event was conducted in June 2016.

SEAD-16 Building S311, Abandoned Deactivation Furnace and SEAD-17 Building 367, Active Deactivation Furnace

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

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

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

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

SEAD-122E PLANE DEICING AREA

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

SEAD-26 FIRE TRAINING PIT AND AREA

At SEAD-26, the primary contaminants detected included SVOCs and metals in the soil and sediments. In addition, low levels of volatiles also were detected in the groundwater at levels above NYSDEC Class GA Standards; however, the contaminants that exceeded NYSDEC GA Standards in the groundwater were no longer found in the soil of SEAD-26 due to attenuation of the contaminants in the soil (Parsons ES, 1998). The initial excavation at SEAD-26 began on

November 9, 2005 and was completed on November 15, 2005. Five distinct areas at SEAD-26 were excavated to a depth of one-foot bgs, and a total of 828 cubic yards (1,248 tons) of soil was excavated and disposed off-site. Confirmatory soil samples were collected from the perimeter and the base of each of the five excavation areas and were analyzed for PAHs. The edges of the five excavation areas were smoothed. All confirmatory samples representative of soil remaining on-site met the soil cleanup goals. Additional remediation of soils at SEAD-26 was not required.

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

SEAD-26 was also investigated for PFAS during the PFAS SI (Parsons, 2018) and PFAS ESI (Parsons, 2022a). During the SI, groundwater from eight temporary one-inch wells was analyzed for PFAS compounds at SEAD 26. Nine of 14 PFAS compounds were detected at SEAD 26. PFDoA, PFTriA, PFUnA, NEtFOSAA and NMeFOSAA were not detected at SEAD 26. PFOS and PFOA were detected in all eight wells sampled at SEAD 26. Combined PFOS/PFOA concentrations exceeded EPA advisory level in four wells (TMW-26-2, -3, -6 and -7) with a maximum concentration of 580 ng/L in well TMW-26-3. Well locations TMW-26-3, -6, and -7 are located directly downgradient of the main former fire training area at SEAD 26. Similar to SEAD 25, the PFOA concentrations were higher than the PFOS concentrations except for the concentrations at TMW-26-1. During the ESI, five soil borings were advanced. Locations SB26-13, SB26-15, and SB26-17 had one sample each collected at a depth of 2.5 to 3 feet bgs. Locations SB26- 14 and SB26-16 encountered fill and samples were collected from 0.2 to 2 feet and 2.5 to 3 feet bgs

Land Use Considerations

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

Potential Receptors and Exposure Pathways

Potential receptors of concern are within the former SEDA boundary and include known or potential exposure pathways for both human exposure (e.g., public water supply wells and private wells) and environmental receptors (e.g., surface water bodies, wetlands). These receptors are potential human and environmental endpoints for exposure. One of the primary objectives of this

RI is to determine if a contaminant migration pathway exists between identified PFAS source areas and receptors.

There are no known public or private water supply wells within one-mile of the Fire House, SEAD-25, or SEAD-26 AOCs, nor are there known private groundwater wells within the former SEDA boundary. A water distribution building/reservoir (formerly Building 334R), located 1,700 ft south of SEAD-25, is now used by the Seneca County Water Department. This structure is partially below grade and was formerly uncovered. This building is outside the expected extent of the SEAD-25 PFAS plume bound to the north of Building 334R by non-detect data at wells MW25-23 and MW25-26 and does not provide water from within the SEDA. The nearest residential receptors are at the Spring Meadows Apartments located east of the Fire House AOC. These apartments are connected to the Seneca County Water District and do not use the local groundwater or have surface water bodies that exit the ESI AOCs. The nearest known downgradient drinking water wells are located along Route 96A approximately 2.5 miles west of the ESI AOCs (Parsons ES, 1994).

The Fire House and SEAD-25, and the known extents of their PFAS plumes, are located within the Planned Industrial/Office Development and Warehousing Area. The future land use of this area is light industry/commercial and currently has a groundwater restriction in place (Parsons, 2021b). The projected land use for the area west of SEAD-25 and SEAD-26 is farming with potential residential use. The landowner of this area was contacted, and they noted the land immediately west of SEAD-26 was poor for farming (marshy, wetlands) and was more likely to be used for hunting or other recreation.

Exposure pathways for environmental receptors (e.g., surface water bodies and wetlands) include groundwater discharge to unnamed drainage ditches adjacent to SEAD-25 and SEAD-26 and an unnamed pond and wetland area west of SEAD-26. Past sampling indicates that PFAS was detected in the drainage ditches southwest of SEAD-25 and in the drainages and pond west of SEAD-26. The results indicate an exposure pathway exists between the activities at SEAD-25 and the PFAS observed in the drainage ditches southwest of SEAD-25, via groundwater discharge and ephemeral stormwater discharge from the Administration Area (Fire House). The analytical results also indicate an exposure pathway is present between the activities at SEAD-26 and the drainage ditches, pond and wetland area west of SEAD-26. No human receptors are likely to be exposed to the water in the drainage ditches; however, farming activities (i.e., grazing livestock) were observed west of SEAD-26. The livestock may ingest water in the drainage ditches or eat vegetation exposed to contaminated groundwater. Groundwater discharge to natural wetland areas and plant uptake of PFAS contaminated groundwater is a potentially complete pathway as these plants may serve as a local food source for deer or other wildlife which may be targets for hunting activities within the former Depot.

WORKSHEET #11
PROJECT/DATA QUALITY OBJECTIVES

DATA QUALITY OBJECTIVES

The data quality objectives (DQO) specify the project objectives, the data collection boundaries and limitations, the most appropriate type of data to collect, and the level of decision error that will be acceptable for the decision. The quality and quantity of data required to implement a response action to treat PFAS contaminated groundwater are also defined. The project-specific DQOs, as defined through the EPA seven-step process (2006), are as follows:

- State the problem;
- Identify the goals of the study;
- Identify information inputs;
- Define the boundaries of the study;
- Develop an analytic approach;
- Specify performance or acceptance criteria; and
- Develop the plan for obtaining data.

General discussions of each step of the DQO process are presented below.

STATE THE PROBLEM

PFAS are an emerging contaminant and have a potential impact on human health and the environment.

IDENTIFY THE GOALS OF THE STUDY

The project objectives are as follows:

1. Perform a PA for the entire SEDA to determine if there is historical evidence of the use, disposal or release of materials containing PFAS at additional sites. A Historical Records Review Report will present the results of the PA.
2. Perform SIs on the sites listed in Table 1 to determine presence or absence of PFAS. Any sites identified during the PA may be added to this list.
3. Perform a RI at SEAD 25, SEAD 26, SEAD-122E/122D and Fire House Bldg. 103 to support FS, PP and ROD.

General activities will include the installation of monitoring wells and sampling of groundwater, soil, surface water, sediment, and biota (deer tissue).

IDENTIFY INFORMATION INPUTS

Analytical PFAS data for groundwater, surface and subsurface soil, sediment and surface water.

DEFINE THE BOUNDARIES OF THE PROJECT

The RI will be at four known PFAS sites and data collected to support the RI will be used to define the vertical and horizontal extent, to collect sufficient data to support conducting human health and ecological risk assessments, and to support identification and evaluation of potential remedial alternatives in the FS. The SIs are to determine if PFAS are present. If PFAS concentrations exceed the acceptance criteria, further action may be proposed.

The groundwater sampling program will include new monitoring wells installed to address potential data gaps at the SEADs, as well as existing monitoring wells that are located in areas that provide adequate spatial coverage. The sample collection will take place during the spring, when seasonal water levels are the highest, and no sooner than 1 week after installation of the new wells to allow for the equilibration of site-specific groundwater field parameters. Development of the newly installed wells will take place at least 48 hours after completion of the well construction activities.

DEVELOP THE ANALYTIC APPROACH

All groundwater, surface and subsurface soil, sediment and surface water will be submitted for the off-site analysis of the PFAS analytes listed in Worksheet #12 by a DoD Environmental Laboratory Accreditation Program (ELAP) accredited laboratory compliant with DoD QSM Version 5.4 (or most current). Laboratory analytical results will be subject to data verification and data validation against criteria established within the analytical method, the DoD QSM, the DoD General Data Validation Guidelines, and this Programmatic UFP-QAPP. Validation will be performed to a 90 percent (%) Stage 2b standard and a 10% Stage 4 standard with recalculation of appropriate data (including DoD QSM, Table B-24 requirements).

Field data from each investigation phase will be evaluated to determine if the nature and lateral/vertical extent of PFAS in groundwater, surface and subsurface soil, sediment and surface water resulting from past DoD-related releases has been defined. The need for additional sample locations to bound PFAS extent (i.e., “step-out samples”) will be based on the presence or lack of sample results less than PALs located laterally outward, or vertically beneath, other sample results greater than PALs. Site-specific conditions, including physical barriers that limit or prevent PFAS migration, will also be considered. Examples of physical barriers include but are not limited to low-permeability geologic strata; surface topography (drainage ditches/swales/streams/storm sewers; and infrastructure (roads, buildings, etc.). The Conceptual Site Model will be revised to incorporate the field investigation results and evaluations after each field investigation phase. The Conceptual Site Model will also include additional proposed sampling recommendations for the subsequent phase, if applicable. After the initial evaluation of field data, additional scoping sessions will be conducted with the project team and decisions from those scoping sessions will be documented in UFPQAPP Worksheet #9 addenda.

SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA

The DoD has adopted a policy within the CERCLA process to compare analytical results for PFAS to risk-based human health screening levels (SLs) for soil and groundwater, as described in a memorandum from the OSD dated 06 July 2022. The 2022 OSD memorandum recommends using the May 2022 EPA RSLs for screening soil and groundwater to be protective of human receptors. The EPA RSLs were updated in November 2022, but there were no changes to the PFAS RSLs. The program under which this RI is being performed follows this DoD policy. The EPA RSLs (presented to 2 significant figures) are consistent with the EPA RSL table format rather than the values as presented in the memorandum. The SLs established in the OSD memorandum apply to six compounds: PFOS, PFOA, PFBS, PFNA, PFHxS, and HFPO-DA. Risk-based human health screening levels for surface water and sediment were also calculated using the May 2022 RSL calculator.

There are two types of decision errors: sampling design and measurement. Sampling design errors are a function of the selection of sample locations and analytical methods used to characterize the site to be studied. The sampling design error will be controlled by careful planning conducted in consultation with USACE and regulatory agencies.

Measurement errors are a function of the procedures used to collect and analyze the samples. Measurement errors that arise during the various steps of the sample-measurement process (e.g., sample collection, sample handling, sample preservation, sample analysis, data reduction, and data handling) are possible regardless of the sample design. Neither measurement error nor variability can be eliminated, but they can be controlled by selecting appropriate procedures. Measurement and performance criteria are presented in Worksheet #12 and the analytical methods, method reporting limits, and project-specific screening limits are discussed in Worksheet #15. Measurement error is further managed by using standard procedures, review of data records, and data quality management.

Decision error will be limited by a careful evaluation of the data and by adherence to established data collection procedures. Analytical method requirements and the programmatic DQOs were established to limit decision error. Published analytical methods and requirements in the DoD QSM (DoD, 2021) are the primary determinants of DQOs by establishing limits for precision and accuracy.

The main source of existing data being considered for use are the PAs and SIs previously completed for each RI and SI site. The acceptance criteria for these data are USACE approvals of these documents.

Field sampling personnel will review the UFP-QAPP before samples are collected and sign off on QAPP Worksheet #4. A copy of the UFP-QAPP will be provided to the laboratory. In addition, third-party data validation by LDC will be performed.

DEVELOP THE PLAN FOR OBTAINING DATA

Worksheets #19 and #30 specify the analytical method, Worksheet #20 identifies the field quality control sampling, and Worksheets #24, #25, #26, #27, and #28 specify the analysis design requirements. The project-specific sampling design and rationale will be presented in Worksheet #17 of the installation-specific UFP-QAPP addenda based on the identified sampling needs.

WORKSHEET #12
MEASUREMENT PERFORMANCE CRITERIA

Table 12-1 Measurement Performance Criteria for PFAS

Matrix	**Water/Solid**	
Concentration	**Low**	
Analytical Group or Method	**PFAS (**Draft Method 1633**)**	
DQI	**QC Sample or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for analytes detected in both samples ≥ sample-specific LOQ.
Analytical accuracy/bias (laboratory)	Laboratory Control Sample (LCS) or Ongoing Precision and Recovery Standard (OPR) and Low-Level Laboratory Control Sample (LLLCS) or Low-Level Ongoing Precision and Recovery Standard (LLOPR)	Analyte specific (see Tables 12-2 through 12-4)
Analytical precision/accuracy/bias (matrix interference)	Matrix spike (MS)/matrix spike duplicate (MSD)	RPD ≤30% (between MS and MSD); Analyte specific (see Tables 12-2 through 12-4)
Analytical precision	Matrix Duplicate (MD)	RPD ≤ 30% (between sample and MD; RPD criteria only applies to analytes whose concentration in the sample is ≥ LOQ.)
Overall accuracy/bias (contamination)	Instrument blank	Concentration of each analyte must be ≤ ½ LOQ
Overall accuracy/bias (contamination)	Field Blank, equipment blank, method blank	No analytes detected >½ LOQ or > 1/10th the amount measured in any associated sample or 1/10th the regulatory limit, whichever is greater.
Sensitivity	Instrument sensitivity check	All analyte concentrations must be at LOQ; concentrations must be within ±30% of their true values. Signal-to-noise ratio must be ≥ 3:1.
Sensitivity	Bile Salt Standards	The retention time of the bile salt(s) peak must fall outside of the retention time window of PFOS by at least 1 minute.
Completeness	See Worksheet #37	See Worksheet #37

DQI = data quality indicator
LOQ = limit of quantitation
RPD = relative percent difference

Table 12-2 Measurement Performance Criteria for PFAS (Aqueous Matrix)

Chemical	CASRN	Acronym	Lower Control Limit (%)	Upper Control Limit (%)
4:2 Fluorotelomer sulfonate	75124-72-4	4:2 FTS	40	150
6:2 Fluorotelomer sulfonate	27619-97-2	6:2 FTS	40	150
8:2 Fluorotelomer sulfonate	39108-34-4	8:2 FTS	40	150
N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	40	150
N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	40	150
Perfluorobutanesulfonic acid	375-73-5	PFBS	40	150
Perfluorobutanoic acid	375-22-4	PFBA	40	150
Perfluorodecanesulfonic acid	335-77-3	PFDS	40	150
Perfluorodecanoic acid	335-76-2	PFDA	40	150
Perfluorododecanoic acid	307-55-1	PFDoA	40	150
Perfluoroheptanoic acid	375-85-9	PFHpA	40	150
Perfluoroheptanesulfonic acid	375-92-8	PFHpS	40	150
Perfluorohexanesulfonic acid	355-46-4	PFHxS	40	150
Perfluorohexanoic acid	307-24-4	PFHxA	40	150
Perfluorononanoic acid	375-95-1	PFNA	40	150
Perfluorononanesulfonic acid	68259-12-1	PFNS	40	150
Perfluorooctanesulfonamide	754-91-6	PFOSA	40	150
Perfluorooctanesulfonic acid	1763-23-1	PFOS	40	150
Perfluorooctanoic acid	335-67-1	PFOA	40	150
Perfluoropentanoic acid	2706-90-3	PFPeA	40	150
Perfluoropentanesulfonic acid	2706-91-4	PFPeS	40	150
Perfluorotetradecanoic acid	376-06-7	PFTeDA	40	150
Perfluorotridecanoic acid	72629-94-68	PFTrDA	40	150
Perfluoroundecanoic acid	2058-94-8	PFUnA	40	150
Perfluorododecanesulfonic acid	79780-39-5	PFDoS	40	150
N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA	40	150
N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA	40	150
N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMeFOSE	40	150
N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtFOSE	40	150
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	40	150
4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	40	150

Table 12-2 Measurement Performance Criteria for PFAS (Aqueous Matrix) (Continued)

Chemical	CASRN	Acronym	Lower Control Limit (%)	Upper Control Limit (%)
Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	40	150
Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	40	150
Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	40	150
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	40	150
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	40	150
Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	40	150
3-Perfluoropropyl propanoic acid	356-02-5	3:3FTCA	40	150
2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3FTCA	40	150
3-Perfluoroheptyl propanoic acid	812-70-4	7:3FTCA	40	150

CASRN = Chemical Abstracts Service Registry Number
FTS = fluorotelomer sulfonate

Table 12-3 Measurement Performance Criteria for PFAS (Solid Matrix)

Chemical	CASRN	Acronym	Lower Control Limit (%)	Upper Control Limit (%)
4:2 Fluorotelomer sulfonate	75124-72-4	4:2 FTS	40	150
6:2 Fluorotelomer sulfonate	27619-97-2	6:2 FTS	40	150
8:2 Fluorotelomer sulfonate	39108-34-4	8:2 FTS	40	150
N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	40	150
N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	40	150
Perfluorobutanesulfonic acid	375-73-5	PFBS	40	150
Perfluorobutanoic acid	375-22-4	PFBA	40	150
Perfluorodecanesulfonic acid	335-77-3	PFDS	40	150
Perfluorodecanoic acid	335-76-2	PFDA	40	150
Perfluorododecanoic acid	307-55-1	PFDoA	40	150
Perfluoroheptanoic acid	375-85-9	PFHpA	40	150
Perfluoroheptanesulfonic acid	375-92-8	PFHpS	40	150
Perfluorohexanesulfonic acid	355-46-4	PFHxS	40	150
Perfluorohexanoic acid	307-24-4	PFHxA	40	150
Perfluorononanoic acid	375-95-1	PFNA	40	150
Perfluorononanesulfonic acid	68259-12-1	PFNS	40	150
Perfluorooctanesulfonamide	754-91-6	PFOSA	40	150
Perfluorooctanesulfonic acid	1763-23-1	PFOS	40	150
Perfluorooctanoic acid	335-67-1	PFOA	40	150
Perfluoropentanoic acid	2706-90-3	PFPeA	40	150
Perfluoropentanesulfonic acid	2706-91-4	PFPeS	40	150
Perfluorotetradecanoic acid	376-06-7	PFTeDA	40	150
Perfluorotridecanoic acid	72629-94-68	PFTrDA	40	150
Perfluoroundecanoic acid	2058-94-8	PFUnA	40	150
Perfluorododecanesulfonic acid	79780-39-5	PFDoS	40	150
N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA	40	150
N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA	40	150
N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMeFOSE	40	150
N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtFOSE	40	150
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	40	150
4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	40	150

Table 12-3 Measurement Performance Criteria for PFAS (Solid Matrix) (Continued)

Chemical	CASRN	Acronym	Lower Control Limit (%)	Upper Control Limit (%)
Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	40	150
Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	40	150
Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	40	150
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	40	150
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	40	150
Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	40	150
3-Perfluoropropyl propanoic acid	356-02-5	3:3FTCA	40	150
2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3FTCA	40	150
3-Perfluoroheptyl propanoic acid	812-70-4	7:3FTCA	40	150

Table 12-4 Measurement Performance Criteria for PFAS (Tissue Matrix)

Chemical	CASRN	Acronym	Lower Control Limit (%)	Upper Control Limit (%)
4:2 Fluorotelomer sulfonate	75124-72-4	4:2 FTS	40	150
6:2 Fluorotelomer sulfonate	27619-97-2	6:2 FTS	40	150
8:2 Fluorotelomer sulfonate	39108-34-4	8:2 FTS	40	150
N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	40	150
N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	40	150
Perfluorobutanesulfonic acid	375-73-5	PFBS	40	150
Perfluorobutanoic acid	375-22-4	PFBA	40	150
Perfluorodecanesulfonic acid	335-77-3	PFDS	40	150
Perfluorodecanoic acid	335-76-2	PFDA	40	150
Perfluorododecanoic acid	307-55-1	PFDoA	40	150
Perfluoroheptanoic acid	375-85-9	PFHpA	40	150
Perfluoroheptanesulfonic acid	375-92-8	PFHpS	40	150
Perfluorohexanesulfonic acid	355-46-4	PFHxS	40	150
Perfluorohexanoic acid	307-24-4	PFHxA	40	150
Perfluorononanoic acid	375-95-1	PFNA	40	150
Perfluorononanesulfonic acid	68259-12-1	PFNS	40	150
Perfluorooctanesulfonamide	754-91-6	PFOSA	40	150
Perfluorooctanesulfonic acid	1763-23-1	PFOS	40	150
Perfluorooctanoic acid	335-67-1	PFOA	40	150
Perfluoropentanoic acid	2706-90-3	PFPeA	40	150
Perfluoropentanesulfonic acid	2706-91-4	PFPeS	40	150
Perfluorotetradecanoic acid	376-06-7	PFTeDA	40	150
Perfluorotridecanoic acid	72629-94-68	PFTrDA	40	150
Perfluoroundecanoic acid	2058-94-8	PFUnA	40	150
Perfluorododecanesulfonic acid	79780-39-5	PFDoS	40	150
N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA	40	150
N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA	40	150
N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMeFOSE	40	150
N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtFOSE	40	150
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	40	150
4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	40	150

Table 12-4 Measurement Performance Criteria for PFAS (Tissue Matrix) (Continued)

Chemical	CASRN	Acronym	Lower Control Limit (%)	Upper Control Limit (%)
Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	40	150
Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	40	150
Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	40	150
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	40	150
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	40	150
Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	40	150
3-Perfluoropropyl propanoic acid	356-02-5	3:3FTCA	40	150
2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3FTCA	40	150
3-Perfluoroheptyl propanoic acid	812-70-4	7:3FTCA	40	150

WORKSHEET #13
SECONDARY DATA USES AND LIMITATIONS

This worksheet will be included in the installation-specific UFP-QAPP addenda. Examples of the information that will be included is presented in the table below.

Data Type	Data Source (Originating Organization, Report Title, and Date)	Data Uses Relative to Current Project	Factors Affecting the Reliability of Data and Limitations on Data Use
Regional geology and hydrogeology	Parsons, 2022a. Final PFAS Expanded Site Investigation (ESI) Report. Former Fire House (Building 103), SEAD-25 (Fire Training and Demonstration Pad), SEAD-26 (Fire Training Pit and Area), Seneca Army Depot Activity. March 2022.	Provided geologic and hydrogeologic setting.	None known.
Meteorology	Parsons, 2021. Five-Year Review. SEAD 1, 2, 5, 12, 13, 16, 17, 23, 25, 26, 27, 39, 40, 41, 43, 44A, 44B, 46, 52, 56, 59, 62, 64A, 64B, 64C, 64D, 66, 67, 69, 71, 121C, 121I, 122B, 122E, 002-R-01, 003-R-01, 007-R-01, and the Ash Landfill Operable Unit (SEADs 3, 6, 8, 14, and 15). Seneca Army Depot Activity. August 2021	Provided climate and geographic information.	None known.

WORKSHEETS #14 AND #16
PROJECT TASKS AND SCHEDULE

The RI and SI field activities at SEDA to be conducted under this UFP-QAPP will begin in the Spring of 2023. While the schedule will be updated accordingly throughout the program, a general timeline of events is summarized below:

- Mobilization and initiation of SI and RI field activities: Late March/Early April 2023.
- Drilling, monitoring well installation and development, and soil sampling: April/May 2023.
- Laboratory analysis and data validation of SI and RI soil samples: May-June 2023.
- SI groundwater sampling: Early May-June 2023.
- Laboratory analysis and data validation of SI groundwater samples: June-July 2023.
- RI groundwater sampling (Round 1): Mid-May 2023.
- Laboratory analysis and data validation of RI groundwater samples (Round 1): May-July 2023.
- RI surface water and sediment sampling (Round 1): Late May 2023.
- Laboratory analysis and data validation of RI surface water and sediment samples (Round 1): June-July 2023.
- RI groundwater sampling (Round 2): Mid-August 2023.
- Laboratory analysis and data validation of RI groundwater samples (Round 2): August-September 2023.
- RI surface water and sediment sampling (Round 2): Late August 2023.
- Laboratory analysis and data validation of RI surface water and sediment samples: September-October 2023.
- RI deer muscle and liver tissue sampling: October-December 2023.
- Laboratory analysis of RI deer biota sampling: December 2023-February 2024.
- Site Inspection Reports: February-October 2024.
- RI and Risk Assessment Report: February-October 2024.

The proposed project schedule and milestone payment for all tasks can be seen in its entirety in the Project Management Plan.

WORKSHEET #15A
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Water
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/L)	PAL Reference[1]	ELLE		
					LOQ (ng/L)	LOD (ng/L)	DL (ng/L)
4:2 Fluorotelomer sulfonate	75124-72-4	4:2 FTS	NE[2]	NA	8.00	3.80	1.70
6:2 Fluorotelomer sulfonate	27619-97-2	6:2 FTS	NE[2]	NA	8.00	7.60	2.50
8:2 Fluorotelomer sulfonate	39108-34-4	8:2 FTS	NE[2]	NA	8.00	7.70	2.60
N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	NE[2]	NA	2.00	1.40	0.700
N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	NE[2]	NA	4.00	2.40	1.20
Perfluorobutanesulfonic acid	375-73-5	PFBS	601	EPA RSL	2.00	1.00	0.300
Perfluorobutanoic acid	375-22-4	PFBA	NE[2]	NA	8.00	4.00	2.00
Perfluorodecanesulfonic acid	335-77-3	PFDS	NE[2]	NA	2.00	1.00	0.500
Perfluorodecanoic acid	335-76-2	PFDA	NE[2]	NA	2.00	1.00	0.500
Perfluorododecanoic acid	307-55-1	PFDoA	NE[2]	NA	2.00	1.00	0.500
Perfluoroheptanoic acid	375-85-9	PFHpA	NE[2]	NA	2.00	1.00	0.520
Perfluoroheptanesulfonic acid	375-92-8	PFHpS	NE[2]	NA	2.00	1.00	0.400
Perfluorohexanesulfonic acid	355-46-4	PFHxS	39	EPA RSL	2.00	1.10	0.570
Perfluorohexanoic acid	307-24-4	PFHxA	NE[2]	NA	2.00	1.00	0.500
Perfluorononanoic acid	375-95-1	PFNA	6	EPA RSL	2.00	1.00	0.500
Perfluorononanesulfonic acid	68259-12-1	PFNS	NE[2]	NA	2.00	1.00	0.400
Perfluorooctanesulfonamide	754-91-6	PFOSA	NE[2]	NA	2.00	1.00	0.500
Perfluorooctanesulfonic acid	1763-23-1	PFOS	4	EPA RSL	2.00	1.00	0.500

WORKSHEET #15A (CONTINUED)
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Water
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/L)	PAL Reference[1]	ELLE		
					LOQ (ng/L)	LOD (ng/L)	DL (ng/L)
Perfluorooctanoic acid	335-67-1	PFOA	6	EPA RSL	2.00	1.30	0.640
Perfluoropentanoic acid	2706-90-3	PFPeA	NE[2]	NA	4.00	2.00	1.00
Perfluoropentanesulfonic acid	2706-91-4	PFPeS	NE[2]	NA	2.00	1.00	0.400
Perfluorotetradecanoic acid	376-06-7	PFTeDA	NE[2]	NA	2.00	1.00	0.500
Perfluorotridecanoic acid	72629-94-68	PFTrDA	NE[2]	NA	2.00	1.00	0.500
Perfluoroundecanoic acid	2058-94-8	PFUnA	NE[2]	NA	2.00	1.00	0.500
Perfluorododecanesulfonic acid	79780-39-5	PFDoS	NE[2]	NA	2.00	1.90	0.900
N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA	NE[2]	NA	2.00	1.00	0.500
N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA	NE[2]	NA	2.00	1.00	0.500
N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMeFOSE	NE[2]	NA	20.0	10.0	5.00
N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtFOSE	NE[2]	NA	20.0	10.0	5.00
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	6	EPA RSL	8.00	4.00	2.00
4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	NE[2]	NA	8.00	3.80	1.50
Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	NE[2]	NA	4.00	2.00	0.500
Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	NE[2]	NA	4.00	2.00	1.00
Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	NE[2]	NA	4.00	2.00	1.00
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	NE[2]	NA	8.00	3.80	1.00
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	NE[2]	NA	8.00	7.60	2.10
Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	NE[2]	NA	4.00	1.80	0.500

WORKSHEET #15A (CONTINUED)
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Water
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/L)	PAL Reference[1]	ELLE		
					LOQ (ng/L)	LOD (ng/L)	DL (ng/L)
3-Perfluoropropyl propanoic acid	356-02-5	3:3FTCA	NE[2]	NA	10.0	5.00	1.50
2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3FTCA	NE[2]	NA	50.0	25.0	10.0
3-Perfluoroheptyl propanoic acid	812-70-4	7:3FTCA	NE[2]	NA	50.0	25.0	10.0

NOTES:

[1]The PALs are the May 2022 EPA tap water RSLs based on a target cancer risk (TR) of 1E-06 and target hazard quotients (THQ) of 0.1. The RSLs are presented in a Memorandum from the Office of the Assistant Secretary of Defense, *Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program*, distributed on July 6, 2022.

[2]Health-based screening values have not been established. The compounds are being analyzed to monitor for presence in water samples.

DL = detection limit
LHA = lifetime health advisory
LOD = limit of detection
NA = not applicable
PAL = project action limit
ng/L = nanograms/liter
RSL = regional screening level
NE = not established

WORKSHEET #15B
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Solid
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/g)	PAL Reference[1]	ELLE		
					LOQ (ng/g)	LOD (ng/g)	DL (ng/g)
4:2 Fluorotelomer sulfonate	75124-72-4	4:2 FTS	NE[2]	NA	0.800	0.400	0.200
6:2 Fluorotelomer sulfonate	27619-97-2	6:2 FTS	NE[2]	NA	1.00	0.800	0.350
8:2 Fluorotelomer sulfonate	39108-34-4	8:2 FTS	NE[2]	NA	1.00	0.800	0.350
N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	NE[2]	NA	0.200	0.100	0.0500
N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	NE[2]	NA	0.200	0.100	0.0500
Perfluorobutanesulfonic acid	375-73-5	PFBS	1,900	EPA RSL	0.200	0.100	0.0500
Perfluorobutanoic acid	375-22-4	PFBA	NE[2]	NA	0.800	0.400	0.100
Perfluorodecanesulfonic acid	335-77-3	PFDS	NE[2]	NA	0.200	0.100	0.0500
Perfluorodecanoic acid	335-76-2	PFDA	NE[2]	NA	0.200	0.100	0.0500
Perfluorododecanoic acid	307-55-1	PFDoA	NE[2]	NA	0.200	0.100	0.0500
Perfluoroheptanoic acid	375-85-9	PFHpA	NE[2]	NA	0.200	0.100	0.0500
Perfluoroheptanesulfonic acid	375-92-8	PFHpS	NE[2]	NA	0.200	0.100	0.0500
Perfluorohexanesulfonic acid	355-46-4	PFHxS	130	EPA RSL	0.200	0.100	0.0500
Perfluorohexanoic acid	307-24-4	PFHxA	NE[2]	NA	0.200	0.120	0.0590
Perfluorononanoic acid	375-95-1	PFNA	19	EPA RSL	0.200	0.100	0.0500
Perfluorononanesulfonic acid	68259-12-1	PFNS	NE[2]	NA	0.200	0.100	0.0500
Perfluorooctanesulfonamide	754-91-6	PFOSA	NE[2]	NA	0.200	0.100	0.0500
Perfluorooctanesulfonic acid	1763-23-1	PFOS	13	EPA RSL	0.200	0.100	0.0510
Perfluorooctanoic acid	335-67-1	PFOA	19	EPA RSL	0.200	0.100	0.0510

WORKSHEET #15B (CONTINUED)
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Solid
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/g)	PAL Reference	ELLE		
					LOQ (ng/g)	LOD (ng/g)	DL (ng/g)
Perfluoropentanoic acid	2706-90-3	PFPeA	NE[2]	NA	0.400	0.200	0.100
Perfluoropentanesulfonic acid	2706-91-4	PFPeS	NE[2]	NA	0.200	0.100	0.0500
Perfluorotetradecanoic acid	376-06-7	PFTeDA	NE[2]	NA	0.200	0.100	0.0500
Perfluorotridecanoic acid	72629-94-68	PFTrDA	NE[2]	NA	0.200	0.100	0.0500
Perfluoroundecanoic acid	2058-94-8	PFUnA	NE[2]	NA	0.200	0.100	0.0500
Perfluorododecanesulfonic acid	79780-39-5	PFDoS	NE[2]	NA	0.200	0.100	0.0500
N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA	NE[2]	NA	0.200	0.100	0.0500
N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA	NE[2]	NA	0.200	0.100	0.0500
N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMeFOSE	NE[2]	NA	2.00	1.00	0.500
N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtFOSE	NE[2]	NA	2.00	1.00	0.500
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	23	EPA RSL	0.800	0.400	0.100
4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	NE[2]	NA	0.800	0.400	0.200
Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	NE[2]	NA	0.400	0.200	0.100
Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	NE[2]	NA	0.400	0.200	0.100
Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	NE[2]	NA	0.400	0.200	0.104
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	NE[2]	NA	0.800	0.400	0.200
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	NE[2]	NA	0.800	0.400	0.200
Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	NE[2]	NA	0.400	0.200	0.100
3-Perfluoropropyl propanoic acid	356-02-5	3:3FTCA	NE[2]	NA	1.00	0.500	0.250

WORKSHEET #15B (CONTINUED)
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Solid
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/g)	PAL Reference	ELLE		
					LOQ (ng/g)	LOD (ng/g)	DL (ng/g)
2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3FTCA	NE[2]	NA	5.00	2.50	1.00
3-Perfluoroheptyl propanoic acid	812-70-4	7:3FTCA	NE[2]	NA	5.00	2.50	1.00

NOTES:

[1]The PALs are the May 2022 EPA residential soil RSLs based on a target cancer risk (TR) of 1E-06 and target hazard quotients (THQ) of 0.1. The RSLs are presented in a Memorandum from the Office of the Assistant Secretary of Defense, *Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program*, distributed on July 6, 2022.
[2]Health-based screening values have not been established. The compounds are being analyzed to monitor for presence in soil and sediment samples.

ng/g = nanograms / gram

WORKSHEET #15C
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Tissue
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/g)	PAL Reference[1]	ELLE		
					LOQ (ng/g)	LOD (ng/g)	DL (ng/g)
4:2 Fluorotelomer sulfonate	75124-72-4	4:2 FTS	NE	NA	0.200	NA	0.0100
6:2 Fluorotelomer sulfonate	27619-97-2	6:2 FTS	NE	NA	0.200	NA	0.0100
8:2 Fluorotelomer sulfonate	39108-34-4	8:2 FTS	NE	NA	0.200	NA	0.0100
N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	NE	NA	0.200	NA	0.0100
N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	NE	NA	0.200	NA	0.0100
Perfluorobutanesulfonic acid	375-73-5	PFBS	NE	NA	0.200	NA	0.0100
Perfluorobutanoic acid	375-22-4	PFBA	NE	NA	0.200	NA	0.0100
Perfluorodecanesulfonic acid	335-77-3	PFDS	NE	NA	0.200	NA	0.0100
Perfluorodecanoic acid	335-76-2	PFDA	NE	NA	0.200	NA	0.0100
Perfluorododecanoic acid	307-55-1	PFDoA	NE	NA	0.200	NA	0.0100
Perfluoroheptanoic acid	375-85-9	PFHpA	NE	NA	0.200	NA	0.0100
Perfluoroheptanesulfonic acid	375-92-8	PFHpS	NE	NA	0.200	NA	0.0100
Perfluorohexanesulfonic acid	355-46-4	PFHxS	NE	NA	0.200	NA	0.0100
Perfluorohexanoic acid	307-24-4	PFHxA	NE	NA	0.200	NA	0.0100
Perfluorononanoic acid	375-95-1	PFNA	NE	NA	0.200	NA	0.0100
Perfluorononanesulfonic acid	68259-12-1	PFNS	NE	NA	0.200	NA	0.0100
Perfluorooctanesulfonamide	754-91-6	PFOSA	NE	NA	0.200	NA	0.0100

WORKSHEET #15C (CONTINUED)
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Tissue
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/g)	PAL Reference	ELLE		
					LOQ (ng/g)	LOD (ng/g)	DL (ng/g)
Perfluorooctanesulfonic acid	1763-23-1	PFOS	NE	NA	0.200	NA	0.0100
Perfluorooctanoic acid	335-67-1	PFOA	NE	NA	0.200	NA	0.0100
Perfluoropentanoic acid	2706-90-3	PFPeA	NE	NA	0.200	NA	0.0100
Perfluoropentanesulfonic acid	2706-91-4	PFPeS	NE	NA	0.200	NA	0.0100
Perfluorotetradecanoic acid	376-06-7	PFTeDA	NE	NA	0.200	NA	0.0100
Perfluorotridecanoic acid	72629-94-68	PFTrDA	NE	NA	0.200	NA	0.0100
Perfluoroundecanoic acid	2058-94-8	PFUnA	NE	NA	0.200	NA	0.0100
Perfluorododecanesulfonic acid	79780-39-5	PFDoS	NE	NA	0.200	NA	0.0100
N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA	NE	NA	0.200	NA	0.0100
N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA	NE	NA	0.200	NA	0.0100
N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMeFOSE	NE	NA	0.200	NA	0.0100
N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtFOSE	NE	NA	0.200	NA	0.0100
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	NE	NA	0.200	NA	0.0100
4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	NE	NA	0.200	NA	0.0100
Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	NE	NA	0.200	NA	0.0100
Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	NE	NA	0.200	NA	0.0100
Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	NE	NA	0.200	NA	0.0100
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	NE	NA	0.200	NA	0.0100

WORKSHEET #15C (CONTINUED)
PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Reference Limits and Evaluation Table for PFAS
Matrix: Tissue
Analytical Method: Draft Method 1633

Analyte	CASRN	Acronym	PAL (ng/g)	PAL Reference	ELLE		
					LOQ (ng/g)	LOD (ng/g)	DL (ng/g)
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	NE	NA	0.200	NA	0.0100
Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	NE	NA	0.200	NA	0.0100
3-Perfluoropropyl propanoic acid	356-02-5	3:3FTCA	NE	NA	0.200	NA	0.0100
2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3FTCA	NE	NA	0.200	NA	0.0100
3-Perfluoroheptyl propanoic acid	812-70-4	7:3FTCA	NE	NA	0.200	NA	0.0100

NOTES:
[1]Health-based screening values are not available for wild game/deer consumption. The compounds are being analyzed to monitor for presence in tissue samples.

ng/g = nanograms / gram

WORKSHEET #17
SAMPLING DESIGN AND RATIONALE

The sampling design and rationale recognizes the time-sensitive actions required to evaluate the presence, nature, and extent of subsurface PFAS contamination at SEDA. To achieve the objectives of the PWS, soil, groundwater, surface water, sediment, and deer tissue samples will be collected and submitted for laboratory analysis of PFAS by EPA Draft Method 1633. The samples will be collected from the 34 SI sites and 4 RI sites identified and discussed in QAPP Worksheet #10. The groundwater samples will be collected from both the newly installed monitoring wells and from selected locations within the existing monitoring well network, chosen, in part, based on the results of the PA/HRR.

The estimated number of samples and types (by media), by site, are summarized in QAPP Worksheets #18 and #20. The investigations are dynamic, and these worksheets will be further developed and refined during subsequent meetings and figures and tables will be issued as addenda.

WORKSHEET #18
DRAFT SAMPLING LOCATIONS AND METHODS

Sampling locations and methods/SOP requirements addressed by this UFP-QAPP are presented in the tables below. Addenda will be provided in the form of figures and tables based on the Systematic Project Planning Meeting November 3, 2021, the HRR Report and subsequent meetings.

Site Investigation Samples

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SEAD-002-R-01 Explosive Ordnance Disposal (EOD) Area #2 and #3, OU11							
MW002-01 thru MW002-04	002SI20001 thru 002SI20004	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD002-01 thru SWSD002-03	002SI30001 thru 002SI30003	Surface Water	NA			411.01, 404.01	
SWSD002-01 thru SWSD002-03	002SI40001 thru 002SI40003	Sediment	TBD			411.01, 404.01	
SB002-01 thru SB002-04	002SI10001 thru 002SI10004	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	
SB002-01 thru SB002-04	002SI10001 thru 002SI10004	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	
SEAD-003-R-01, SEAD- 57, EOD Area #1 (SEAD 57), OU11							
MW003-01 thru MW003-03	003SI2001 thru 003SI2003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB003-01 thru SB003-04	003SI10001 thru 003SI10004	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SB003-01 thru SB003-04	003SI10001-003SI0004	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	
SEAD-16, Building S311, Abandoned Deactivation Furnace, OU4							
TBD	16SI20001 Thru 16SI20003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
MW16-1	16SI20004		TBD				
SWSD16-01 and SWSD16-02	16SI30001 and 16SI30002	Surface Water	NA			411.01, 404.01	
SWSD16-01 and SWSD16-02	16SI40001 and 16SI40002	Sediment	TBD				
SS16-01 thru SS16-04	16SI10001 thru 16SI10004	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB16-01	16SI10001	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-17, Building 367, Active Deactivation Furnace, OU4							
MW17-?? Thru MW17-??	17SI1000? Thru 17SI1000?	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD17-01	17SI30001	Surface Water	NA			411.01, 404.01	
SWSD17-01	17SI40001	Sediment	TBD			411.01, 404.01	
SB17-1 thru SB17-3	17SI10001 thru 17SI10003	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-23, Open Burning Grounds, OU2							
MW23-8 thru MW23-10	23SI10008 thru 23SI10010	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SWSD-12 and SWSD-13	23SI30012 thru 23SI30015	Surface Water	NA			411.01, 404.01	Determine if PFAS contamination is present.
SWSD-12 and SWSD-13	23SI40012 thru 23SI40015	Sediment	TBD	PFAS	See Worksheet #20	411.01, 404.01	
SB23-1 thru SB23-4	23SI10001 thru 23SI10004	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-24, Abandoned Powder Burning Pits, OU13							
MW24-1 thru MW24-3	24SI20001 thru 24SI20003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD24-1	24SI30001	Surface Water	NA			411.01, 404.01	
SWSD24-1	24SI40001	Sediment	TBD			411.01, 404.01	
SB24-1 thru SB24-3	24SI10001 thru 24SI10003	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-45, Open Detonation Grounds, OU17							
MW45-1 Thru MW45-5	45SI20001 Thru 45SI20005	Groundwater		PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD45-1 and SWSD45-2	45SI30001 and 45SI30002	Surface Water	NA			411.01, 404.01	
SD45-1 and SD45-2	45SI40001 and 45SI40002	Sediment	NA				
SB45-1 thru SB45-5	45SI10001 thru 45SI10005	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-46, Small Arms Firing Range (aka Former 3.5” Rocket Range), OU11							
MW46-1 thru MW46-5	46SI20001 thru 46SI20005	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SWSD46-1 and SWSD46-2	46SI30001 and 46SI30002	Surface Water	NA			411.01, 404.01	Determine if PFAS contamination is present.
SWSD46-1 and SWSD46-2	46SI40001 and SI40002	Sediment	TBD				
SB46-1 thru SB46-5	46SI10001 thru 46SI10005	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-007-R-01, Grenade Range, OU11							
MW007-1 thru MW007-4	007SI20001 thru 007SI20004	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB007-1 thru SB007-4	007SI10001 thru 007SI10004	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB007-1 thru SB007-4	007SI10001 thru 007SI10004	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
Fire House Building 722							
MWFH-1 thru MWFH-3	FHSI20001 thru FHSI20003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SBFH-1 thru SBFH-3	FHSI10001 thru FHSI10003	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SBFH-1 thru SBFH-3	FHSI10001 thru FHSI10003	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-3 Incinerator Cooling Water Pond, OU1 – No samples proposed as it is very small and surrounded by other sites.							
SEAD-6, Abandoned Ash Landfill, OU1							

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
MW6-1 thru MW6-3	6SI20001 thru 6SI20003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SEAD-8, Non-Combustible Fill Area, OU1							
MW8-1 thru MW8-3	8SI20001 thru 8SI20003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB8-1 thru SB8-3	8SI20001 thru 8SI20003	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB8-1 thru SB8-3	8SI20001 thru 8SI20003	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-14, Refuse Burning Pits (2 Units), OU1							
MW14-1	14SI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD14-1 and SWSD14-2	14SI30001 and 14SI30002	Surface Water	NA			411.01, 404.01	
SWSD14-1 and SWSD14-2	14SI40001 and 14SI40002	Sediment	TBD			411.01, 404.01	
SB14-1	14SI10001	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB14-1	14SI10001	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-15, Abandoned Solid Waste Incinerator (Building 2207), OU1							
MW15-1	15SI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SEAD-5, Sewage Sludge Storage Pile, OU13							

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
MW5-1 and MW5-2	5SI20001 and 5SI20002	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB5-1 thru SB5-2	5SI10001 and 5SI10002	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB5-1 thru SB5-2	5SI10001 and 5SI10002	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-20, Sewage Treatment Plant #4, OU14							
MW20-1 thru MW20-3	5SI200001 thru 5SI200003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD20-1 and SWSD20-2	20SI30001 and 20SI30002	Surface Water	NA			411.01, 404.01	
SWSD20-1 and SWSD20-2	20SI40001 and 20SI40002	Sediment	NA				
SB20-1 thru SB20-3	20SI10001 thru 20SI10003	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB20-1 thru SB20-3	20SI10001 thru 20SI10003	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-21, Sewage Treatment Plant # 715, OU14							
MW21-1	21SI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD21-1 and SWSD21-2	21SI30001 and 21SI30002	Surface Water	NA			411.01, 404.01	
SWSD21-1 and SWSD21-2	21SI40001 and 21SI40002	Sediment	TBD				
SB21-1	21SI10001	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SB21-1	21SI10001	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-22, Sewage Treatment Plant # 314, OU14							
MW22-1	22SI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB22-1	22SI10001	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB22-1	22SI10001	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-7, Shale Pit, OU14							
MW7-1	7SI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD7-1 and SWSD7-2	7SI30001 and 7SI30002	Surface Water	NA			411.01, 404.01	
SWSD7-1 and SWSD7-2	7SI40001 and 7SI40002	Sediment	TBD				
SB7-1	7SI10001	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB7-1	7SI10001	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-9, Old Scrap Wood Site, OU14							
MW9-1	9SI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SB9-1	9SI10001	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB9-1	9SI10001	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-10, Scrap Wood Site, OU14							
MW10-1	10SI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB10-1	10SI10001	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB10-1	10SI10001	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-11, Old Construction Debris Landfill, OU8							
MW11-1 thru MW11-4	14SI20001 thru 14SI20004	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB11-1 thru SB11-4	11SI10001 thru 11SI10004	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB11-1 thru SB11-4	11SI10001 thru 11SI10004	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-58, Debris Area Near Booster Station 2131, OU14							
MW58-1 thru MW59-4	58SI20001 thru 58SI20004	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SW58-1	58SI30001	Surface Water				411.01, 404.01	Determine if PFAS contamination is present.
SD58-1	58SI40001	Sediment	NA	PFAS		411.01, 404.01	Determine if PFAS contamination is present.
SS58-1 thru SS58-4	58SI10001 thru 58SI10004	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB58-1 thru SB58-4	58SI10001 thru 58SI10004	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-59, Fill Area West of Building 315, OU6							
MW59-1 thru MW59-3	59SI20001 thru 59SI20003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD59-1	59SI30001	Surface Water	NA			411.01, 404.01	
SWSD59-1	59SI40001	Sediment	TBD				
SB59-1 thru SB59-3	59SI10001 thru 58SI10003	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB59-1 thru SB59-3	59SI10001 thru 58SI10003	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-64A, Garbage Storage Area South of Classification Area, OU14							
MW64A-1	64SI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SB64A-1	64SI10001	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB64A-1	64SI10001	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-64B, Garbage Disposal Area South of Classification Area, OU14							
MW64B-1	64BSI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD64B-1	64BSI30001	Surface Water	NA			411.01, 404.01	
SWSD64B-1	64BSI40001	Sediment	TBD				
SB64B-1	64BSI10001	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB64B-1	64BSI10001	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD 64C, Garbage Disposal Area, OU14							
MW64C-1 thru MW64C-5	64CSI20001 thru 64CSI20005	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD64C-1 and SWSD64C-2	64CSI30001 and 64CSI30002	Surface Water	NA			411.01, 404.01	
SWSD64C-1 and SWSD64C-2	64CSI40001 and 64CSI40002	Sediment	TBD			411.01, 404.01	
SB64C-1 thru SB64C-5	64CSI10001 thru 64CSI10005	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SB64C-1 thru SB64C-5	64CSI10001 thru 64CSI10005	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD 64D, Garbage Disposal Area West of Building 2203, OU14							
MW64D-1 and MW64D-2	64DSI20001 and 64D20002	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB64D-1 thru SB64D-2	64DSI10001 and 64DSI10002	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB64D-1 thru SB64D-2	64DSI10001 and 64DSI10002	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-67, Dump Site East of Sewerage Treatment Plant #4, OU14							
MW67-1 and MW67-2	67SI20001 and 67SI20002	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SWSD67-1 and SWSD67-2	67SI30001 and 67SI30002	Surface Water	NA			411.01, 404.01	
SD67-1 and SD67-2	67SI40001 and 67SI40002	Sediment	TBD				
SB67-1 and SB67-2	67SI10001 and 67SI0002	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB67-1 and SB67-2	67SI10001 and 67SI0002	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-68, Old Pest Control Shop (Building S-335), OU14							
MW68-1 and MW68-2	68SI20001 and 68SI20002	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sample Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SB68-1 and SB68-2	68SI10001 and 68SI10002	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB68-1 and SB68-2	68SI10001 and 68SI10002	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-69, Building 606 Disposal Area, OU14							
MW69-1 thru MW69-4	69SI20001 thru 69SI20004	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB69-1 thru SB69-4	69SI10001 thru 69SI10004	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB69-1 thru SB69-4	69SI10001 thru 69SI10004	Subsurface Soils	0.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SEAD-70, Former Building T-2110, Filled Area, OU11							
MW70-1 thru MW70-3	70SI20001 thru 70SI2003	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine if PFAS contamination is present.
SB70-1 thru SB70-3	70SI10001 thru 70SI10003	Surface Soils	0-0.5’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
SB70-1 thru SB70-3	70SI10001 thru 70SI10003	Subsurface Soils	1.5-2.0’	PFAS	See Worksheet #20	411.01, 403.06, 403.02	Determine if PFAS contamination is present.
	SEAD-122D, Airfield Hot Pad Spill (Site is now covered under SEAD 122E in RI)						

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Preliminary RI Samples

Sampling Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SEAD-25 Fire Training and Demonstration Pad							
MW25-1	25RI20001	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine the nature and extent of PFAS contamination and present risk imparted by it.
MW25-6	25RI20002		TBD				
MW25-8	25RI20003		TBD				
MW25-13	25RI20004		TBD				
MW25-15	25RI20005		TBD				
MW25-19	25RI20006		TBD				
MW25-20	25RI20007		TBD				
MW25-21	25RI20008		TBD				
MW25-22	25RI20009		TBD				
MW25-22D	25RI20010		TBD				
MW25-24	25RI20011		TBD				
MW25-25	25RI20012		TBD				
MW25-28	25RI20013		TBD				
MW25-31	25RI20014		TBD				
MW25-31D	25RI20015		TBD				
MW25-32	25RI20016		TBD				
MW25-33	25RI20017		TBD				
MW25-34D	25RI20018		TBD				
MW25-35	25RI20019		TBD				
MW25-36	25RI20020 and 25RI20029		TBD				
MW25-37	25RI20021 and 25RI20030		TBD				

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sampling Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
MW25-38	25RI20022 and 25RI20031		TBD				
MW25-39	25RI20022 and 25RI 20032		TBD				
MW25-40	25RI20024 and 25RI20033		TBD				
TBD	25RI20025 thru 25RI20027	GW Dups	TBD				
SB25-17 thru SB25-46	25RI10001-0.0-0.5 thru 25RI10033-0.0-0.5	Surface Soil	0-0.5’			411.01, 403.06, 403.02	
TBD	25RI10034-1.5-2.0 thru 25RI10044-1.5-2.0	Subsurface Soils	1.5-2.0’			411.01, 403.06, 403.02	
SWSD25-1 thru SWSD-12	25RI30001 thru 25RI30030	Surface Water	NA	PFAS	See Worksheet #20	411.01, 404.01	
SWSD25-1 thru SWSD25-12	25RI40001 thru 25RI40032	Sediment	TBD			411.01, 404.01	
SB25-1 thru SB25-30		Surface Soil	0-0.5’			411.01, 403.06, 403.02	
SB25-1 thru SB25-7		Subsurface Soil	1.5-2.0’				
SEAD-26 Fire Training Pit							
MW26-12	26RI20001 THRU 26RI20025	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 404.01	411.01, 404.01
MW26-13							
MW26-15							
MW26-16							

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sampling Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
MW26-18							
MW26-19							
MW26-20							
MW26-23							
MW26-23D							
MW26-24							
MW26-25							
MW26-26							
MW26-27							
MW26-28							
MW26-28D							
MW26-29							
MW26-30							
MW26-31							
MW26-32D							
MW26-33							
MW26-34			TBD				
SWSD26-1 thru SWSD26-10	26RI30001 thru 26RI30026	Surface Water	NA			411.01, 404.01	
SWSD26-1 thru SWSD26-10	26RI4001 thru 26RI40022	Sediment	TBD				
SB26-13 thru SB26-42	26RI10001-0.0-0.5 thru 26RI10032-0.0-0.5	Subsurface Soil	0.5-2.0’			411.01, 403.06, 403.02	

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sampling Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
TBD	26RI10034-1.5-2.0 thru 26RI10046-1.5-2.0	Surface Soil	0-0.5’			411.01, 403.06, 403.02	
SEAD-122D/122E Airfield							
MW122E-04	122ERI20001 thru 122ERI20018	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine the nature and extent of PFAS contamination and present risk imparted by it.
MW122E-05							
MW122E-06							
MW122E-07							
MW122E-08							
MW122E-09							
MW122E-10							
MW122E-11							
MW122D-01	122DRI20001 thru 122DRI20010						
MW122D-02							
MW122D-03							
MW122D-04							
SWSD122D-01 thru SWDS122D-02	122DRI30001 thru 122DRI30008	Surface Water	NA			411.01, 404.01	
SWSD122D-01 thru SWSD122D-02	122DRI40001 thru 122DRI40008	Sediment	TBD				
SWSD122E-01 thru SWSD122E-06	122ERI30001 thru 122ERI30016	Surface Water	NA				

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sampling Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
SWSD122E-01 thru SWSD122E-06	122DRI40001 thru 122DRI40016	Sediment	TBD				
SB122D-01 thru SB1225-05	122DRI10001-0.0-0.5 thru 122DRI10009-0.0-0.5	Surface Soil	0-0.5’			411.01, 403.06, 403.02	
SB122E-01 thru SB122E-25	122ERI10001-0.0-0.5 thru 122ERI10033-0.0-0.5	Surface Soil	0-0.5’				
TBD	122DRI10001-1.5-2.5 thru 122DRI10005-1.5-2.5	Subsurface Soil	1.5-2.0’				
TBD	122ERI10001-1.5-2.0 thru 122ERI10033-1.5-2.0	Subsurface Soil	1.5-2.0’				
Firehouse							
MWFH-02	FHRI20001 thru FHRI20025	Groundwater	TBD	PFAS	See Worksheet #20	411.01, 402.01	Determine the nature and extent of PFAS contamination and present risk imparted by it.
MWFH-03							
MWFH-04							
MWFH-05							
MWFH-06							
MWFH-09							
MWFH-10D							
MWFH-12							
MWFH-13							

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sampling Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
MWFH-14							
MWFH-15							
MWFH-16							
MWFH-16D							
MWFH-18							
SBFH-1 thru SSFH--20	FHRI10001-0.0-0.5 thru FHRI10022-0.0-0.5	Surface Soil	0-0.5’			411.01, 403.06, 403.02	
TBD	FHRI10023-0.0-0.5 thru FHRI10031-0.0-0.5	Subsurface Soils	1.5-2.0’				
SBFH-21/MHFH-12 thru SBFH-25/MHFH-16	FHRI10021-0.0-0.5 thru FHRI10025-0.0-0.5	Surface Soil	0-0.5’				
SBFH-21/MHFH-12 thru SBFH-25/MHFH-16	FHRI10023-1.5-2.0 thru FHRI10031-1.5-2.0	Subsurface Soils	1.5-2.0’				
Biota							
Deer Muscle Tissue				PFAS	See Worksheet #20		Determine the nature and extent of PFAS contamination and
DEERMUSC-01 thru DEERMUSC-17	SARI50001 thru SARI50017	MUSC	NA				
Deer Liver Tissue							

WORKSHEET #18 (CONTINUED)
DRAFT SAMPLING LOCATIONS AND METHODS

Sampling Location	Field Sample ID Number	Matrix	Depth (ft bgs)	Analytical Methods	Number of Samples (identify field QC)	Sampling SOP References	Rationale for Sampling Location
DEERLIV-01 thru DEERLIV 17	FHRI10023-0.0-0.5 thru FHRI10031-0.0-0.5	LIV	NA				present risk imparted by it.

WORKSHEETS #19 AND #30
SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

Laboratory Name: ELLE, 2425 New Holland Pike, Lancaster, PA 17601
Accreditations[1]: DoD Environmental Laboratory Accreditation Program (ELAP), expires 11/30/2024
Sample Delivery Method: FedEx

Analyte / Analytical Group	Matrix	Method / SOP[2]	Containers (Number, Size, and Type)	Preservation	Holding Time (Preparation / Analysis)	Data Package Turnaround
PFAS	Water	Draft Method 1633 / WI46412	2 x 500 mL HDPE with no Teflon liner in cap	≤6 degrees Celsius (°C) for up to 48 hours after sampling then ≤ -20°C; keep protected from light	28 days to extraction/28 days from extraction to analysis	10-15 business days
PFAS	Solid	Draft Method 1633 / WI48593	1 x 4.5 oz wide mouth HDPE with no Teflon liner in lid	≤6°C for up to 48 hours after sampling then ≤ -20°C; keep protected from light	28 days to extraction/28 days from extraction to analysis	10-15 business days
PFAS	Tissue	Draft Method 1633 / WI48593	1 x 4.5 oz wide mouth HDPE with no Teflon liner in lid	≤6°C for up to 24 hours after sampling then ≤ -20°C; keep protected from light. If a longer transport time is necessary, freeze the sample before shipping. Ideally, deer tissue should be frozen upon collection and shipped to the laboratory on ice.	28 days to extraction/28 days from extraction to analysis	10-15 business days

[1] Laboratory accreditation is included in Attachment 3.

[2] Laboratory standard operating procedures (SOP) are included in Attachment 1.

HDPE = high-density polyethylene
mL = milliliter
oz = ounce

WORKSHEET #20
FIELD QUALITY CONTROL SUMMARY

Matrix	Analytical Group	Field Sample	Field Duplicate[1]	MS/MSD[2]	Field Blanks[3]	Equipment Blanks[4]	Trip Blanks	Total No. of Analyses
SI Samples								
Groundwater	PFAS	82	8	4	10	NA	NA	104
Surface Water	PFAS	23	3	2	2	NA	NA	30
Sediment	PFAS	23	3	2	2	1	NA	31
Soil	PFAS	159	16	8	10	1	NA	194
RI Samples								
Groundwater	PFAS	98	13	9	10	NA	NA	130
Surface Water	PFAS	73	12	NA	6	NA	NA	91
Sediment	PFAS	57	8	NA	1	1	NA	67
Soil	PFAS	168	19	12	4	4	NA	207
Biota	PFAS	30	4	2	2	NA	NA	38

Notes:
1 - One field duplicate will be collected for every 10 field samples (1/10 samples).
2 - One set of MS/MSD samples will be collected for every 20 field samples (1/20 samples).
3 - One field blank sample will be collected for each day of the field event.
4 - Disposable equipment (HDPE tubing with peristaltic pumps and/or disposable HDPE bailers) will be used to collect groundwater samples; therefore, no equipment blank will be collected for aqueous matrices. Surface water samples will be collected directly into the applicable sample containers.
For soil and sediment, equipment blanks will be collected at a rate of 1 per day per matrix. All disposable sample collection equipment will be confirmed to be PFAS-free.
MS = matrix spike
MSD = matrix spike duplicate

WORKSHEET #21
FIELD SOPS

SOP# or Reference Number[1,2]	Title, Revision Date and URL (if available)	Originating Organization	SOP Option or Equipment Type (if SOP provides different option)	Modified for Project? Y/N	Comments
411.01	Per- and Polyfluoroalkyl Substances Sampling Procedures, February 2020	HGL	NA	N	-
411.02	Sampling Equipment Cleaning and Decontamination	HGL	NA	N	-
402.01	Low-Flow (Minimal Drawdown) Groundwater Sampling Procedures, Revision 4; December 2019	HGL	NA	N	-
403.02	Hand-Operated Auger Soil Sampling, Revision 2; August 2019	HGL	NA	N	-
403.06	Surface and Shallow Depth Soil Sampling, Revision 3; June 2020	HGL	NA	N	-
403.08	Sediment Sampling, Revision 2; March 2020	HGL	NA	N	-
404.01	Surface Water Sampling, Revision 2; March 2020	HGL	NA	N	-
406.01	Monitoring Well Development, Revision 2; November 2019	HGL	NA	N	-
406.02	Monitoring Well Installation, Revision 3; November 2020	HGL	NA	N	-
300.04	Field Logbook Use and Maintenance, November 2019	HGL	NA	N	-

Notes:

[1] SOPs are listed in the order of hierarchy for SEAD Project. In cases where duplicate procedures are presented in multiple SOPs, the SOP with the highest hierarchy shall be followed.
[2] The SOPs listed above are presented in Attachments 2 and 4.

WORKSHEET #22
FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA[2]	Responsible Person	SOP Reference
Oxidation reduction potential (ORP) meter[1]	NA	NA	Single standard calibration check	NA	Daily, before sampling	Two successive readings within ±10 mV	Recalibrate instrument	Field sampling team	2.22
	Sensitivity verification	NA	NA	NA	Daily, before sampling	ORP should decrease as pH is increased	If ORP increases, correct the polarity of electrodes. If ORP still does not decrease, clean electrodes and repeat procedure	Field sampling team	2.22
Turbidity meter[1]	Single standard calibration with formazan standard per instrument range used	NA	NA	NA	Daily, before sampling	±5 units,0–100 range; ±0.5 units,0–20 range; ±0.2 units,0–1 range	Recalibrate instrument	Field sampling team	2.22
Dissolved Oxygen meter[1]	NA	NA	Function check	NA	Daily, before sampling	Meter reads 8% ±2%	Replace instrument	Field sampling team	2.22
Aqueous pH meter[1]	2-point calibration with pH buffers	NA	NA	NA	Daily, before sampling	±0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace, if necessary. Repeat calibration.	Field sampling team	2.22

WORKSHEET #22 (CONTINUED)
FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA[2]	Responsible Person	SOP Reference
Conductance meter[1]	Calibration with potassium chloride standard	NA	NA	NA	Daily, before sampling	±5%	If calibration is not achieved, check meter, standards, and probe; recalibrate.	Field sampling team	2.22
GPS	Establish control points using the Global Navigation Satellite System (GNSS).	NA	NA	NA	Daily, before use	±6 meters	Recalibrate the instrument using the GNSS.	Field sampling team	2.22

1 Direct reading from real-time probe associated with a flow-through cell.
2 If CA does not solve the problem, the equipment will be removed from service and replaced until it has been repaired.
mV = millivolt
GPS = Global positioning system
ORP = oxidation reduction potential

WORKSHEET #23
ANALYTICAL SOP REFERENCES TABLE

Lab SOP Number	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix and Analytical Group	SOP Options or Equipment Type	Modified for Project Work? (Y/N)
WI48593	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24, Version 1, effective 05/26/2022.	Definitive	Solids	Liquid chromatography/tandem mass spectrometry (LC/MS/MS)	N
WI46412	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24, Version 1, effective 06/03/2022.	Definitive	Water	LC/MS/MS	N
WI23588	Preventative and Corrective Maintenance for the API 4000 and AB Sciex 4500, 5500, 5500+ Liquid Chromatograph Mass Spectrometers (LC/MS/MS), Rev 3, effective 02/2022.	Instrument maintenance	Not applicable	LC/MS/MS	N

WORKSHEET #24
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible of CA	SOP Reference
LC/MS/MS	Mass Calibration	Instrument must have a valid mass calibration prior to any sample analysis. Thereafter, performed annually and after any major maintenance. Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL).	Calibrate the mass scale of the mass spectrometer (MS) with calibration compounds and procedures described by the manufacturer. Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ±0.5 atomic mass units of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard.	If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance.	Analyst	WI48593; WI46412
LC/MS/MS	Mass Spectral Acquisition Rate	Each analyte, extracted internal standard analyte, and injection internal standard analyte.	A minimum of 10 spectra scans are acquired across each chromatographic peak.	None	Analyst	WI48593; WI46412
LC/MS/MS	Tuning	When masses fall outside ± 0.5 amu of true masses	Within 0.5 amu of true value.	Retune and verify. If tuning fails acceptance criteria, perform a mass calibration and repeat the tune check.	Analyst	WI48593; WI46412

WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible of CA	SOP Reference
LC/MS/MS	Calibration, Calibration Verification, and Spiking Standards	All analytes	Standards containing both branched and linear isomers must be used when commercially available. For PFAS compounds that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions, and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the ICAL that uses the linear isomer quantitative standard.	None	Analyst	WI48593; WI46412
LC/MS/MS	Sample PFAS Identification	All analytes detected in a sample	The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (e.g., PFBA and PFPeA). Documentation of the primary and confirmation transitions and the ion ratio is required. In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50-150%. Signal-to-noise ratio must be ≥ 10:1 for all ions used for quantification and must be ≥ 3:1 for all ions used for confirmation. Quant ion and confirmation ion must be present and must maximize simultaneously (±2 seconds)	None	Analyst	WI48593; WI46412

WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible of CA	SOP Reference
LC/MS/MS	Ion Transitions (Precursor -> Product)	Every field sample, standard, blank, and QC sample	1) If a qualitative or quantitative standard containing an isomeric mixture (branched and linear isomers) of an analyte is commercially available for an analyte, the quantification ion used must be the quantification ion identified in Table 2 of EPA Draft Method 1633 unless interferences render the product ion unusable as the quantification ion. 2) In cases where interferences render the product ion unusable as the quantification ion, project approval is required before using the alternative product ion.	Provide technical justification in the Case Narrative.	Analyst	WI48593; WI46412
LC/MS/MS	Ion Ratio	All analytes detected in a sample.	Must meet all the requirements of EPA Draft Method 1633.	Document and discuss the failure in the Case Narrative. Apply I-flag to the result associated with the failure.	Analyst	WI48593; WI46412
LC/MS/MS	Extracted Internal Standard (EIS) Compounds	Every field sample, standard, blank, and QC sample.	1) Isotopically labeled analogs of analytes must be used when they are commercially available. 2) QC samples and field samples must recover within in-house limits if project limits are not provided; otherwise, project limits must be met. Preliminary inhouse acceptance criteria of 20-150% must be used until inhouse limits are generated in accordance with Sections 9.4.1 and 9.4.2 of EPA Draft Method 1633. 3) The lower limit of inhouse acceptance criteria cannot be < 20%.	Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, follow the requirements listed in EPA Draft Method 1633, Section 15.3.2. If EIS recoveries still fall outside of the acceptance range, the client must be contacted for additional measures to be taken.	Analyst	WI48593; WI46412

WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible of CA	SOP Reference
LC/MS/MS	Non-extracted Internal Standard (NIS) Compounds	Every field sample, standard, blank, and QC sample.	1) NIS areas must be greater than 30% of the average area of the calibration standards in undiluted sample extracts and sample extracts that required additional NIS to be added. 2) NIS areas corrected for the dilution factor must be greater than 30% of the average area of the calibration standards in diluted samples when additional NIS was not added post dilution of the extract.	Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst	WI48593; WI46412
LC/MS/MS	ICAL (minimum of 6 points for a quadratic curve)	At instrument set-up and initial calibration verification (ICV) or calibration verification (CV) failure, prior to sample analysis	The isotopically labeled analog of an analyte (EIS) must be used for quantitation if commercially available (isotope dilution quantitation). Commercial PFAS standards available as salts are acceptable providing the measured mass is corrected to the neutral acid concentration. Results shall be reported as the neutral acid with appropriate CAS number. If a labeled analog is not commercially available, the EIS analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (Internal Standard Quantitation) Analytes must be within 70-130% of their true value for each calibration standard. Signal-to-noise ratio must be ≥ 10:1 for all ions used for quantification. ICAL must meet one of the two options below: Option 1: The relative standard deviation of the response factors for all analytes must be ≤ 20%. Option 2: Linear or non-linear calibrations must have $r^2 \geq 0.99$ for each analyte.	Correct problem, then repeat ICAL.	Analyst	WI48593; WI46412

WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible of CA	SOP Reference
LC/MS/MS	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 CV is used.	None	Analyst	WI48593; WI46412
LC/MS/MS	Retention time window width	Every field sample, standard, blank and QC sample	Retention time of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the daily CV or, on days when ICAL is performed, from the midpoint standard of the ICAL. Analytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to analyte and labeled analog pairs.	Correct problem and reanalyze all affected samples.	Analyst	WI48593; WI46412
LC/MS/MS	Instrument sensitivity check (ISC)	Daily. At the beginning of each analytical sequence, prior to sample analysis.	Analyte concentrations must be at LOQ; concentrations must be within ±30% of their true values. Signal-to-noise ratio must be ≥ 3:1 for all ions used for confirmation.	Correct problem, rerun ISC. If problem persists, repeat ICAL.	Analyst	WI48593; WI46412
LC/MS/MS	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis	All analyte concentrations must be within ±30% of their true value.	Correct problem, rerun ICV. If problem persists, repeat ICAL.	Analyst	WI48593; WI46412

WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible of CA	SOP Reference
LC/MS/MS	CV	Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence	Concentration of analytes must range from the LOQ to the mid-level calibration concentration. Analyte concentrations must be within ±30% of their true value.	Immediately analyze two additional consecutive CVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CVs cannot be run, perform CA(s) and repeat CV and all associated samples since last successful CV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CV.	Analyst	WI48593; WI46412

WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible of CA	SOP Reference
LC/MS/MS	Instrument Blanks	Immediately following the highest standard analyzed in the calibration, daily prior to analyzing standards, after each CV, and immediately following samples with PFAS concentrations exceeding the quantification range.	Concentration of each analyte must be ≤ 1/2 the LOQ. Instrument Blank must contain EIS and NIS to enable quantitation of contamination.	If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met. If sample concentrations exceed the highest calibration standard and the sample(s) following exceed this acceptance criteria (>1/2 LOQ), they must be reanalyzed using a fresh aliquot of the sample extract.	Analyst	WI48593; WI46412

WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION FOR PFAS ANALYSIS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible of CA	SOP Reference
LC/MS/MS	Bile Salt Standards	Daily, prior to analysis of all matrix types (aqueous, solid, tissue, and AFFF).	All EPA Draft Method 1633 requirements for evaluation of the relationship of the retention time of the bile salt peak(s) to the retention time window of PFOS must be met for all matrix types. The retention time window of PFOS applies to the retention time of all isomers of PFOS. The retention time of the bile salt(s) peak must fall out of the retention time window of PFOS by at least 1 minute.	None	Analyst	WI48593; WI46412

WORKSHEET #25
ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference
LC/MS/MS	Backflush of column, injection port and pre-columns, cleaning of ion spray cone, adjustment of collision energies, others as needed.	Calibration Check	Visual inspection	As needed.	Initial calibration or calibration verification passes method specifications.	Perform additional maintenance prior to instrument calibration or calibration verification	Analyst	WI23588; WI48593; WI46412

WORKSHEETS #26 AND #27
SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Sample shipment procedures will include overnight shipment by commercial courier or direct transport by commercial courier, laboratory courier, or field team. When samples are collected on a Friday, HGL will coordinate with the laboratory to ensure the samples can be received at the laboratory on Saturday.

Sample Numbering

This numbering system will ensure 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
FH = Firehouse	RI = Remedial Investigation	000## = Field QC items (e.g., Rinsate Blanks)
45 = SEAD 45 (OD Grounds)	SI = Site Inspection	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
	--	5#### = Deer Tissue Samples

Every sample number will be preceded by the site name/number designation to identify the site (SEAD) from which the sample was collected. The numerical component for each sample will build upon past sample events, if any. 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 event, if any. Sample name/numbering examples are presented in **Table 26.1**, and the complete sample list for the next round of sampling for each site is detailed on **Worksheet #18** and in the SI and RI work plans.

WORKSHEETS #26 AND #27 (CONTINUED)
SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Sample Collection, Packaging, and Shipment (Reference subsequent pages of this worksheet and field SOP[1])
Sample Collection (Personnel/Organization): Site Staff/USACE contractor
Sample Packaging (Personnel/Organization): Site Staff/USACE contractor
Coordination of Shipment (Personnel/Organization): Site Staff/USACE contractor, Sample Receipt Manager
Type of Shipment/Carrier: See introductory text.
Field Sample Storage (number of days from sample collection): Samples will be held in the field no longer than overnight unless prior arrangements have been made with the laboratory. Holding times must not be compromised by holding samples in the field.
Special Sample Shipment Considerations: See introductory text.
Sample Receipt and Analysis (Reference Laboratory SOP[2])
Sample Receipt (Personnel/Organization): Sample Management Staff
Sample Custody and Storage (Personnel/Organization): Sample Management Staff
Sample Preparation (Personnel/Organization): Organic Preparation Staff, Inorganic Preparation Staff, and Bench Chemists
Sample Determinative Analysis (Personnel/Organization): Bench Chemists
Sample Archiving (Reference Laboratory SOP[2])
Sample Extract/Digestate Storage (number of days from extraction/digestion): For 60 days from data report release or as required on a site-specific basis
Biological Sample Storage (number of days from sample collection): As required on a site-specific basis
Sample Disposal (Reference Laboratory SOP[2])
Personnel/Organization: Sample Management Staff
Number of Days from Analysis: 60 from data report release; up to 6 months on sample-specific request from USACE contractor

[1] Worksheet #21, Field SOP References Table.

[2] Worksheet #23, Analytical SOP References Table.

WORKSHEETS #26 AND #27 (CONTINUED)
SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to the laboratory):

USACE contractor will maintain chain of custody (CoC) records for all field and field QC samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in his or her possession; (2) it is in his or her view after being in the individual's possession; (3) it was in his or her possession and is locked up; or (4) it is in a designated secure area after being in his or her possession.

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analyses, storage, data generation, reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in the field and laboratory records. All sample containers will be sealed in a manner that will prevent tampering or indicate tampering, should it occur. In no instance will sample containers be sealed with tape.

Sample Labeling: Each sample will have a unique sample identification (ID) assigned in accordance with the site-specific sample IDs presented in Worksheet #18. Sample IDs will include the site number, sample media (SW, SED, SS, MW etc.) and sample number at a minimum. Field QC samples will be identified in accordance with the ID protocols presented in Worksheet #20. The following information will be included on the label:

- Project ID,
- Sample ID,
- Type of sample matrix, MW, SED, SW, SS or SB
- Preservative added,
- Date and time of collection,
- Required analytical methods, and
- Sampler's initials.

The samples labels will be placed on the sample containers so as not to obscure any QA/QC data on the bottles. Sample information will be printed in a legible manner using an ink pen or will be preprinted. Field ID must be sufficient to enable cross-reference with the appropriate sample documentation forms. CoC forms will be completed at the time of collection, including all required information, and ensuring that the CoC information matches the information on the sample labels.

Sample Packaging: Preservation reagents will be added to sample containers before or immediately after collection of the sample, as indicated in Worksheets #19 and #30. The samples will immediately be placed on ice and will be kept chilled during the workday until packaged for shipment to the laboratory.
Sample coolers will be supplied by the laboratory. When packaging samples for shipment, the cooler drainage plug will be closed and the cap will be sealed in place with duct tape. Sample containers will be placed inside sealed PFAS-free plastic bags as a precaution against cross-contamination caused by leakage or breakage. Bagged sample containers will be placed in the coolers in such a manner as to eliminate the chance of breakage during shipment. Ice in PFAS-free plastic bags will be placed in the coolers to keep the samples at 6 ºC or less throughout shipment. Prior to sealing the cooler, the sampler's copy of the CoC forms will be detached and provided to the SS for the project file. The remaining portion of the completed CoC forms will be attached to the underside of the cooler lid in a sealed PFAS-free plastic bag. The cooler will then be taped shut and at least two completed custody seals will be affixed across the gap between the lid and body of the cooler.

WORKSHEETS #26 AND #27 (CONTINUED)
SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Sample Custody Requirements (continued)

Sample Shipment: Samples collected in the field will be shipped to the laboratory as expeditiously as possible. Sample shipment will be performed in accordance with all applicable Department of Transportation regulations. The samples will be shipped to the laboratory by the procedures identified in this worksheet. Arrangements will be made between the USACE contractor and the contract laboratory point-of-contact for samples that are to be delivered to a laboratory on a weekend so that sample condition and holding times are not compromised.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal):

The designated sample custodian(s) and staff are responsible for samples received at the laboratory. In addition to receiving samples, the sample receipt staff also is responsible for documentation of sample receipt and storage before and after sample analysis. Summaries of the minimal laboratory receipt procedures are as follows:

- Upon receipt, sign, date, and document the time of sample receipt on the air bills or other shipping manifests received from the couriers.
- Sign the CoC form assuming custody of the samples. If a CoC form is not received with a set of samples, the laboratory will immediately notify the USACE contractor Project Chemist.
- Inspect the sample cooler for integrity and then document the following information:
  - Type of courier and whether the samples were shipped, or hand delivered (copies of the air bills are maintained).
  - Availability and condition of custody information.
  - Sample temperature.
  - If the temperature of the samples upon receipt at the laboratory exceeds the temperature requirements, individual sample containers will be measured. All exceedances will be documented in laboratory records, and the laboratory must contact the USACE contractor Project Chemist immediately and document any decision regarding the potentially affected samples.
  - Presence of leaking or broken containers and indication of sample preservation.
- Verify that the holding time has not been exceeded. If a sample has exceeded holding time, the USACE contractor Project Chemist must be notified.
- Match the sample container information (e.g., sample tag/label), CoC records, and all pertinent information associated with the sample. The sample custodian then verifies sample identity to ensure all information is correct. Any inconsistencies are resolved with the USACE contractor through the Laboratory PM. CA measures are documented before sample analysis proceeds.

WORKSHEET #28
ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION FOR PFAS

Matrix	**Water / Solid / Tissue**
Analytical Group	**PFAS**
Analytical Method/ SOP Reference	**EPA LC/MS/MS in compliance with Table B-24 of DoD QSM 5.4 or more recent version/ WI48593 and WI46412**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Flagging Criteria
Method Blank	One per batch of 20 or fewer samples per matrix	No analytes detected >½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit (whichever is greater).	Correct the problem; if required, re-prepare and/or reanalyze method blank and all QC samples and samples processed with the contaminated blank.	Analyst	Overall accuracy/ bias (contamination)	See Method/ SOP QC Acceptance Limit Column
LCS/OPR and LLLCS/LLOPR (includes LCS duplicate [LCSD] if MS/MSD not performed)	One per batch of 20 or fewer samples per matrix	In-house limits unless project-specific limits are established. If analytes are not listed use in-house limits; preliminary in-house acceptance criteria of 40-150% from Table B-24 of QSM 5.4 must be used until in-house limits are generated. RPD ≤30 (if LCSD is performed).	Correct the problem; then, if sufficient sample material is available, reprepare and reanalyze the LCS/OPR/LLLCS/LLOPR and all samples in the associated preparatory batch for failed analytes.	Analyst	Accuracy Bias, Precision	See Method/ SOP QC Acceptance Limit Column
MS (Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration.)	One per batch of 20 or fewer samples per matrix. MS is not required for aqueous samples prepared by serial dilution instead of SPE.	In-house limits unless project-specific limits are established. If analytes are not listed use in-house limits; preliminary in-house acceptance criteria of 40-150% from Table B-24 of QSM 5.4 must be used until in-house limits are generated.	Contact the client to determine if additional measures are required. For specific analyte(s) in parent sample, apply J flag if acceptance criteria not met; explain in Case Narrative.	Analyst	Accuracy Bias	See Method/ SOP QC Acceptance Limit Column

WORKSHEET #28 (CONTINUED)
ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION FOR PFAS

Matrix	**Water / Solid / Tissue**
Analytical Group	**PFAS**
Analytical Method/ SOP Reference	**EPA LC/MS/MS in compliance with Table B-24 of DoD QSM 5.4 or more recent version/ WI48593 and WI46412**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Flagging Criteria
MSD or Matrix duplicate (MD)	For MSD: One per preparatory batch. For MD: Each aqueous sample prepared by serial dilution instead of SPE.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. For matrix evaluation, use MS recovery criteria; RPD ≤30% (between MS and MSD or sample and MD).	Contact the client to determine if additional measures are required. For specific analyte(s) in parent sample, apply J flag if acceptance criteria not met; explain in Case Narrative.	Analyst	Accuracy Bias, Precision	See Method/ SOP QC Acceptance Limit Column
Post Spike Sample	Only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of <LOQ for analyte(s).	Spike aliquot(s) of sample at the final dilution(s) reported for sample with all analytes that have reported value of <LOQ in the final dilution. The spike must be at the LOQ concentration to be reported with the sample (the <LOQ value). When analyte concentrations are calculated as <LOQ, the spike must recover within 70-130% of its true value.	When analyte concentrations are calculated as <LOQ, and the spike recovery does not meet the 70-130% acceptance criteria, the sample, sample duplicate, and post-spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.	Analyst	Accuracy Bias, Precision	See Method/ SOP QC Acceptance Limit Column

WORKSHEET #29
PROJECT DOCUMENTS AND RECORDS

Record	Generation	Verification	Storage Location/ Archival
Sample Collection and Field Records			
Field Logbook or data collection sheets	Field Sampler	Field Team Leader	Project file
CoC form	Field Sampler	Field Team Leader	Project file
Air bills	Field Sampler	Field Team Leader	Project file
Contractor Daily QC Reports	Field Team Leader	CQCSM	Project file
Investigation Derived Waste	Field Team Leader	CQCSM	Project file
Deviations	Field Team Leader	CQCSM	Project file
Project Assessments			
Data Verification Checklist	Project Chemist	CQCSM	Project File
Data Validation Report	Third-party Data Validator	CQCSM	Project File
Data Usability Assessment Report	Project Chemist	Project Team/CQCSM/PM	Project File
Laboratory Records			
Sample Receipt, Custody, and Checklist	Laboratory Sample Receiving	Laboratory PM, PM	Laboratory and Project File
Equipment Maintenance, Testing, and Inspection logs	Laboratory Analyst	Laboratory QA Manager	Laboratory File
Standard Traceability Logs	Laboratory Analyst	Laboratory QA Manager	Laboratory File
Sample Preparation Logs	Laboratory Analyst	Laboratory QA Manager, PM	Laboratory and Project File
Run Logs	Laboratory Analyst	Laboratory QA Manager, PM	Laboratory and Project File
Analytical Results	Laboratory Analyst	Laboratory QA Manager, PM	Laboratory and Project File
QC Samples and Standards	Laboratory Analyst	Laboratory QA Manager, PM	Laboratory and Project File
Instrument Results (raw data)	Laboratory Analyst	Laboratory QA Manager	Laboratory and Project File
Sample Disposal Records	Laboratory Technician	Laboratory QA Manager	Laboratory File

WORKSHEET #29 (CONTINUED)
PROJECT DOCUMENTS AND RECORDS

Record	PFAS
Case narrative	X
CoC	X
Sample receipt records	X
Communication records	X
Lab chronicle	X
Sample results	X
QC summaries	X
QC data	X
Calibration (Initial, continuing and etc.)	X
Instrument and preparation logs	X
Instrument quantitation forms (raw data)	X
Instrument chromatograms and spectra	X
Standards traceability	X
Electronic Data Deliverables (in Environmental Restoration Program Information Management System [ERPIMS] format)	X

WORKSHEETS #31, #32, AND #33
ASSESSMENTS AND CORRECTIVE ACTION

Assessments:

Assessment Type	Responsible Party and Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Review of programmatic UFP--QAPP, installation-specific UFP-QAPP addenda, SOPs, Accident Prevention Plan, and Site Safety and Health Plan with Field Staff	PM	Prior to sampling startup and with all new field staff before assignment	TBD	Completed acknowledgment signature pages	48 hours following assessment
Work performed in accordance with the programmatic UFP-QAPP and installation-specific UFP-QAPP addenda	Field Team Leader	Ongoing during field activities. Internal QC field audits are conducted as needed during field sampling events and any non-conformance corrective action taken.	TBD	Daily QC report	24 hours following conclusion of business day
Logbook and Field Form Review	Field Team Leader	Daily	Ongoing	NA; corrections will be made directly to reviewed document	24 hours following assessment
Field Sampling and CoC Form Review against programmatic UFP-QAPP and installation-specific UFP-QAPP addenda requirements	Project Chemist	Daily	Ongoing	Correction will be made directly to reviewed documents; communication may be in the form of email	24 hours following assessment
Laboratory Audit	Laboratory PM	At the discretion of the DoD.	TBD	Audit report	TBD
Project-Specific Performance Evaluation Samples[2]	NA	NA	NA	NA	NA

WORKSHEETS #31, #32, AND #33 (CONTINUED)
ASSESSMENTS AND CORRECTIVE ACTION

Assessment Response and CAs:

Assessment Type	Responsibility for Responding to Assessment Findings	Assessment Response Documentation	Time Frame for Response	Responsibility for Implementing CA	Responsibility for Monitoring CA Implementation
Review of programmatic UFP-QAPP, installation-specific UFP-QAPP addenda, and SOPs with Field Staff	PM	Completed acknowledgment signature pages	48 hours following assessment	PM	CQCSM
Work performed in accordance with the programmatic UFP-QAPP and installation-specific UFP-QAPP addenda	PM	Interim CAs documented pending final approval	By close of same business day	PM	CQCSM
Logbook and Field Form Review	Field Team Leader	Correction will be made directly to reviewed documents	NA	PM	CQCSM
Field Sampling and CoC Form Review against programmatic UFP-QAPP and installation-specific UFP-QAPP addenda	Field Team Leader Project Chemist	Response to email	48 hours after notification	PM	CQCSM
Laboratory Audit	Laboratory PM	Memorandum on CAs performed in response to audit report.	NA	Laboratory PM	Laboratory QC Director
Project-Specific Performance Evaluation Samples[1]	NA	NA	NA	NA	NA

1 Laboratory routinely verifies its performance with performance evaluation sample analysis; therefore, project-specific performance evaluation samples will not be performed.

WORKSHEET #34
DATA VERIFICATION AND VALIDATION INPUTS

Item	Description	Verification (Completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	**X**	
2	Contract	**X**	
3	Field SOPs	**X**	
4	Laboratory SOPs	**X**	
Field Records			
5	Field Logbooks	**X**	**X**
6	Equipment Calibration Records	**X**	**X**
7	CoC Forms	**X**	**X**
8	Sampling Diagrams/Surveys	**X**	**X**
9	Relevant Correspondence	**X**	**X**
10	Change Orders/Deviations	**X**	**X**
11	Field CA Reports	**X**	**X**
Laboratory Data Deliverable			
12	Cover Sheet (laboratory identifying information)	**X**	**X**
	Table of Content		
13	Case Narrative	**X**	**X**
14	CoC	**X**	**X**
15	Shipping Documents	**X**	**X**
16	Sample Receipt Records	**X**	**X**
17	Sample Chronology	**X**	**X**
18	Communication Records	**X**	**X**
19	Instrument Calibration Records	**X**	**X**
20	Definition of Laboratory Qualifiers	**X**	**X**
21	Results Reporting Forms	**X**	**X**
22	Method QC Forms	**X**	**X**
23	Instrument QC Forms	**X**	**X**
24	CA Reports	**X**	**X**
25	Instrument and Preparation Logs	**X**	**X**
26	Raw Data (Instrument Quantitation Forms)	**X**	**X**
27	Instrument Chromatograms and Spectra	**X**	**X**
28	Standard Traceability	**X**	
29	Electronic Data Deliverable	**X**	**X**

WORKSHEET #35
DATA VERIFICATION PROCEDURES

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field Logbook	QAPP	Field logbooks, forms, and/or data collection sheets will be reviewed for verification that the information is complete.	Field Team Leader
CoC Forms	QAPP	CoC forms will be reviewed upon completion and verified for completeness.	Field Team Leader
Sample Receipt	QAPP	Sample receipt by the laboratory will be verified when login confirmation is received from the laboratory.	Laboratory PM; Project Chemist
Sample Login	QAPP	Sample login information will be reviewed and verified for completeness according to the CoC.	Laboratory PM; Project Chemist
Laboratory Data Deliverable	QAPP	Verify that the laboratory deliverable contains all records specified in Worksheet #34 of the QAPP. Compare the data package with the CoCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Verify that necessary signatures and dates were present.	Before release: Laboratory PM After release: Project Chemist
Audit Reports, CA Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that CA was implemented according to the plan.	CQCSM

WORKSHEET #36
DATA VALIDATION PROCEDURES FOR PFAS

Analytical Methods	PFAS
Data deliverables requirements	Stage 4 data package, to include summary sheets, calibration, and bench worksheets in accordance with Appendix A of the QSM 5.4 or more recent version in pdf format.
Analytical specifications	Worksheet #28
Measurement performance criteria:	Worksheet #12
Percent of data packages to be validated	Stage 2B and Stage 4 data validation will be performed by LDC within the contract-specified turnaround time.
Percent of raw data reviewed	10% - Stage 4 data validation will be performed on samples with the highest reported levels of PFAS per field event as specified by the Project Chemist.
Percent of results to be recalculated	10%
Validation procedure	• U.S. Army Corps of Engineers Guidance for Evaluating Performance-based Chemical Data, Engineer Manual 200-1-10 (30 June 2005) • DoD QSM, Version 5.4, Table B-24 (October 2021) • Environmental Data Quality Workgroup, Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15 (01 May 2020) • Environmental Data Quality Workgroup, Data Validation Guidelines Module 6: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-24 (18 October 2022) • Environmental Data Quality Workgroup, General Data Validation Guidelines (04 November 2019) • HGL SOP 412.501, Data Validation, US EPA/DoD Stage 2A and Stage 2B, Revision 3 (June 2021) • LDC SOP 4.96DOD.0, Data Qualification for Perfluoroalkyl and Polyfluoroalkyl Substances Using DoD QSM 5.3 Table B-15
Validation code:	90% - Stage 2B Validation Manual (S2BVM) 10% - Stage 4 Validation Manual (S4VM)
Electronic validation program/version:	ERPToolsX

WORKSHEET #37
DATA USABILITY ASSESSMENT

This worksheet provides the procedures, methods, and activities to determine whether data are of the right type, quality, and quantity to support environmental decision-making for the project.

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

It is the responsibility of the Project Chemist and the laboratory to ensure the data meet the method detection limits, reporting limits/minimum detected activities, and laboratory QC limits listed in this programmatic UFP-QAPP. During the data validation assessment, non-conformances are documented, and data are qualified for use in the decision making. Data gaps will be present if a sample is not collected, a sample is not analyzed for the requested parameters, or the data are determined to be unusable. The need for further investigation will be determined on a case-by-case basis, depending on whether data can be extrapolated from adjacent sampling locations, and whether or not the results are unnecessary based on the results from adjacent locations. All data are usable as qualified by the data validator, except for data qualified for possible exclusion. Estimated and/or biased results are usable. Outliers, if present, can be addressed on a case-by-case basis.

Usability assessment process and procedures:

Compliance with measurement quality objectives for the data quality indicators (e.g., precision, accuracy, representativeness, completeness, comparability, and sensitivity [PARCCS]) will be evaluated to support assessment of data usability. Data qualified with the "X" qualifier as excluded are evaluated by the project team to consider rejection. The formulated project team will consist of, at a minimum, the USACE Project Chemist and HGL Project Chemist. Data qualified with the "J" qualifier are considered estimated. The project team will determine if any bias that might be present in the qualified results affects the usability of the data for the intended purpose. Several different types of laboratory QC information (field and lab duplicates, MS/MSD, LCS, and etc.) will be used as multiple lines of evidence to understand the possible bias before concluding that data is usable for decision making purposes.

Evaluative procedures used to assess overall measurement error associated with the project:

After all data evaluations are completed, any limitations on the use of data will be known and the limitations will be considered during decision making. After data validation and an overall review of data quality indicators, the data will be reconciled with the DQOs to determine whether sufficient data of acceptable quality are available for decision making.

The following is a summary of the usability assessment process and all procedures including interim steps and any statistics, equations, and computer algorithms that will be used:

The QC program to be used is designed to obtain data quality indicators for each field procedure and analytical method. The PARCCS criteria are the qualitative and quantitative indicators of data quality. An objective of this programmatic UFP-QAPP is to assure that collected data are precise, accurate, representative, complete, and comparable to actual site conditions. QC results will be used to assess all project data. All affected project samples will be qualified if low/high-biased QC results are consistently reported. PARCCS criteria are defined as follows:

Precision

Precision measures the reproducibility of measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Precision is evaluated by comparing the RPDs of field versus field duplicate, MS versus MSD, LCS versus LCSD – also known as blank spike – and blank spike duplicate against the limits established by the laboratory. The formula for the calculation of precision is:

$$\text{RPD \%} = \left(\frac{(X_1 - X_2)}{(X_1 + X_2)/2} \right) \times 100$$

Where:

X_1 = Concentration of analyte in sample, and

X_2 = Concentration of analyte in corresponding replicate/duplicate sample.

Precision is determined for analytical results using field and laboratory duplicates or duplicate MS samples. Precision measures the reproducibility of measurements under a given set of conditions.

Accuracy

Accuracy is the statistical measurement of correctness and includes components of random error (variability due to imprecision) and systematic error. A measurement is accurate when the reported value does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the percent recovery (organic and inorganic) of analytes spiked into LCSs and MS and/or MSD samples to the DoD QSM control limits. Recoveries outside the control limits indicate a cause other than normal measurement error. CA may include instrument recalibration, reanalysis of the QC sample, or reanalysis of the samples in the batch. For organic analyses, surrogate compound recoveries and tracer yields, respectively, also are used to assess accuracy and method performance for each sample analyzed. The calculation used for percent recovery/yield is expressed as:

$$\text{Percent Recovery} = \frac{X - D}{D} \times 100$$

Where:

X represents the value/activity of the spike sample.

D represents the spike concentration added.

Accuracy of analytical results reported in environmental samples also are measured against any contamination present in laboratory method blanks and instrument blanks, as well as field blanks, such as trip and equipment rinsate blank samples. Frequency of sampling and analysis of laboratory and field blanks is specified on Worksheet #20.

The temperature receipt of the cooler is measured from a representative sample in the cooler (the temperature of the outside of a container measured with an infrared gun). The cooler temperature is recorded and reported by the laboratory for evaluation during data validation. CA for coolers received at temperatures outside the acceptance limits (< 6°C) may result in the qualification of results or resampling of affected samples.

Sensitivity

Sensitivity is the ability of the method or instrument to detect the contaminant of concern and other target compounds at the level of interest. Sensitivity is achieved for a majority of the methods by the use of a low-level calibration standard. For those methods that require a multi-level ICAL, the low-level calibration standard is spiked at or below the quantitation level specified on Worksheet #15. The criterion used to measure the performance of this QC sample is the ICAL acceptance criteria specified in the DoD QSM and summarized on Worksheet #24. The CA performed if the acceptance criterion is not achieved is summarized on Worksheet #24.

Comparability

Comparability is a qualitative parameter that expresses the confidence with which one data set may be compared to another. This is prime concern when current data is being integrated with historical data. Comparability of data is maximized through the use of standard operating procedures in the field and laboratory, standardized analytical methods, and consistent units of measure. The laboratory shall make the necessary provisions to ensure the comparability of all data. These procedures include, but are not limited to, the use of standard units and reporting formats, the use of calculations as referenced in the methodology for quantitation, and the use of standard measures of accuracy and precision for quality control samples.

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under correct, normal conditions. Completeness will be evaluated qualitatively and quantitatively. The qualitative evaluation of completeness will be determined as a function of all events contributing to the sampling event. This includes items such as samples arriving at the laboratory intact, properly preserved, and in sufficient quantity to perform the requested analyses. The quantitative description of completeness will be defined as the percentage of QC parameters that are acceptable. Data validation and data quality assessment will determine which data are valid and which data are rejected or missing. The quantitative assessment of completeness must meet a criterion of 90% and will be calculated for each analytical method as:

$$\text{Completeness} = (S/R) \times 100$$

Where:

S = Number of acceptable sample results, and

R = Number of requested sample results.

REFERENCES

EPA, 2012, *Uniform Federal Policy for Quality Assurance Project Plans (Intergovernmental Data Quality Task Force, 2005).* March.

A list of additional references can be found on pages 14 & 15 of this document.

ATTACHMENT 1

LABORATORY STANDARD OPERATING PROCEDURES

ELLE: Polyfluorinated Alkyl Substances (PFASs) in Solids by LC/MS/MS Using Draft Method 1633/ QSM 5.4 Table B-24

ELLE: Polyfluorinated Alkyl Substances (PFASs) in Aqueous Samples by LC/MS/MS Using Draft Method 1633/ QSM 5.4 Table B-24

ELLE: Preventative and Corrective Maintenance for the API 4000 and AB Sciex 4500, 5500, 5500+ Liquid Chromatograph Mass Spectrometers (LC/MS/MS), Revision 3

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eurofins	Always check on-line for validity.	Level:
Document number: **T-PFAS-WI46412**	**Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24**	**Work Instruction**
Old Reference:		
Version: **1**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date **03-JUN-2022**	Document users: **5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep**	Responsible: **5_EUUSLA_PFAS_Manager**

Revision Log

Revision:	1	Effective date: This version
Section	**Justification**	**Changes**
Revision Log	NEW	NEW

Reference

1. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft method 1633), Department of Defense Quality System Manual Version 5.4, Table B-24.

2. US EPA Method 1633, *Analysis of Per and Polyfluoroalkyl Substances(PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS*, Version DRAFT, August 2021.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-PFAS-WI21568	Manifold and N-EVAP Cleaning for PFAS Extractions
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
QA-SOP11178	Demonstrations of Capability
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5

Analyte	Acronym	CAS#
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonic acid	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6

Analyte	Acronym	CAS#
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

A 500-mL aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. Carbon cleanup is performed on each sample extract. The extract is filtered and fortified with Isotopically-labeled injection internal standards. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.

3. All part of the SPE manifold must be cleaned as per *T-PFAS-WI21568*.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste

disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level.

See *QA-SOP11178* for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 500-mL HDPE containers. The second aliquot may be collected in a smaller sample container (e.g. 250 mL or 125 mL). All sample containers must have linerless HDPE or polypropylene caps. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.

2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, and protected from light until extraction. Alternatively, to meet project requirements, samples may be stored at $\leq$ -20°C and protected from light until extraction.

3. Water samples must be extracted within 28 days when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Water samples must be extracted within 90 days when stored at a temperature ≤ -20°C and protected from light. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C.

Apparatus and Equipment

A. Apparatus

1. 500 mL HDPE bottles: Scientific Specialties; #334008-blk-1, or equivalent.
2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 05-539-5 or equivalent
3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288 or equivalent.
4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC.
5. Analytical Balance – Capable of weighing to 0.0001 g
5. Top-Loading Balance – Capable of weighing to 0.01 g
7. Solid phase extraction (SPE) Weak Anion Exchange ("WAX") cartridge – Agilent; Sampli-Q WAX Polymer; 150mg/6mL; Cat. # 5982-3667.
3. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product # 54258-U.
9. SPE Tube Adapter - Millipore-Sigma; Product # 57020-U.
10. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.
11. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.
12. Centrifuge – "Q-Sep 3000"; Restek Corp. Cat. No. 26230, or equivalent, capable of a minimum rotational speed of 3000 rpm.
13. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent.
14. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. FisherScientific cat # 14-287-150, or equivalent.
15. Polypropylene pipette tips: 0-200µl. Fisher; Cat. No. 02-681-135
16. Polypropylene pipette tips: 101-1000µl. Fisher, Cat. No. 02-707-508
17. Pipettes – Disposable transfer. FisherScientific, Cat. No. 13-711-7M
18. Vortex mixer, variable speed, Fisher Scientific or equivalent.

19. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control. Organomation, Inc. Cat. #11250, or equivalent.

20. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTPF08831.

21. Thermo Target PP Polyspring inserts, catalog number C4010-630P

22. Agilent 9mm vial kit pack, catalog number 5190-2278, or equivalent

23. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent

24. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125

25. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product # 11-865-130.

26. pH paper, range 0-14, Whatman Panpeha or equivalent, 0.5 unit readability

27. Syringe filter - Acrodisc, Syringe Filter, GHP,13 mm, 0.2 µm, Aqueous, 100/pkg, Part # WAT097962.

28. Silanized glass wool (Sigma-Alrich, Cat #20411 or equivalent

29. Disposable syringe filter, 25-mm, 0.2um Nylon membrane, PALL/Acrodisc or equivalent

30. Glass fiber filter, 47 mm, 1 um, PALL A/E or equivalent

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

   ExionLC Controller
   ExionLC AC Pump
   ExionLC AC Autosampler
   Exion AC Column Oven
   Data system –Analyst 1.6.3

2. HPLC columns

   a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-YO or equivalent

   b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

1. Methanol (MeOH) – Honeywell Burdick and Jackson "Chromasolv LC-MS" grade Cat. No. BJ34966-4L or equivalent
2. Acetonitrile (ACN) – Fisher Scientific, Optima Cat. No. A955-4 or equivalent
3. Ammonium acetate – Fisher Scientific, Cat. No. A637-500 or equivalent
4. Ammonium hydroxide (NH_4OH), 5.0 M; Ricca, Cat. No. 644-32 or equivalent
5. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
6. Aqueous ammonium hydroxide (3%) – add ammonium hydroxide (10 mL, 30%) to reagent water (90 mL), store at room temperature, replace after 3 months
7. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
8. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
9. Acetic Acid – ACS grade or equivalent, store at room temperature
10. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
11. Formic acid
    a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years
    b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years
    c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years
    d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years

e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years

12. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.

13. 20 mM ammonium acetate solution in 95:5 Milli-Q water/acetonitrile – Weigh 1.54 ± 0.01g ammonium acetate into a 1-L glass mobile phase bottle. Add 950mL of Milli-Q water and mix well to dissolve the ammonium acetate. Add 50 mL acetonitrile and mix well. Store at room temperature for up to one 2 months.

B. Standards:

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10 ml Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent. After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in glass or HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 4*.
If the compound purity is assayed to be 96% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent, i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.

Attachment 5 - Native PFAS Intermediate A
Attachment 6 – Native PFAS Intermediate B
Attachment 7 – Working Labeled Extraction Standard Spike
Attachment 8 – Working Internal Standard Spike
Attachment 9 – Native 1633 Mid-Level Spike
Attachment 10 – Native 1633 Low-Level Spike
Attachment 11 – 1633 Initial Calibration Standards Preparation
Attachment 12 – 1633 Initial calibration Standards Concentrations
Attachment 13 – TDCA Stock Solution
Attachment 14 – TDCA Working Solution A
Attachment 15 – TDCA Working Solution B
Attachment 16 – 1633 Linear/Branched TDCA Intermediate
Attachment 17 – 1633 Linear/Branched TDCA Solution
Attachment 18 – PFAS 1633 ICV Working Standard
Attachment 19 – 1633 Labeled Ampulated Standards
Attachment 20 – 1633 Native Ampulated Standards

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.

2. Analyze a Cal4 level standard that contains TDCA retention time marker and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

   Example Initial Calibration Sequence:
   1. Instrument Blank
   2. Instrument Blank
   3. Instrument Blank
   4. CAL 1
   5. CAL 2
   6. CAL 3
   7. CAL 4
   8. CAL 5
   9. CAL 6
   10. CAL 7
   11. ICB (Instrument Blank)
   12. ICV
   13. MDL
   14. WDM (Linear Branched/TDCA standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 2* for additional information about compound relationships.

4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

   NOTE: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA.

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA

NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA

NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

5. Fit the curve

   a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

   b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for the compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.

   c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

   d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

   NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See *Attachment 4*.

6. Initial Calibration Verification (ICV)

   A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must be within ±30% of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

2. Acceptance criteria

   a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

   b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

A. Sample Preparation

1. Weigh sample container with contents on a calibrated top loading balance, and record the first reading in the automated prep entry system.

   a. For all samples, the full bottle must be extracted. The sample must remain in the original sample container until after spiking solutions have been added.

   b. If limited sample is submitted, spike sample in original container, then add Milli-Q water to bring to final volume of 500 mL prior to SPE extraction (see B.6 for spiking details).

2. Use a 500 mL HDPE bottle for the method blank, the laboratory control sample (LCS), and the low level laboratory control sample (LLCS). Fill each bottle with 500 mL of Milli-Q water. Record 500 mL as the volume for the batch QC samples on the batchlog.

3. Check that the pH is 6.5±0.5. If necessary, adjust the pH with 50% formic acid or ammonium hydroxide (or with 5% formic acid and 3% aqueous ammonium hydroxide).

B. Solid Phase Extraction (SPE)

1. Pack clean silanized glass wool to half the height of the WAX SPE cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

3. Condition each SPE cartridge with the following reagents in the following order without allowing the cartridges to go dry:

   a. 15 mL 1% methanolic ammonium hydroxide

   b. 5 mL 0.3M formic acid

c. Discard conditioning eluent(s)

4. Label each sample bottle, cap and reservoir with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Vortex all spike solutions prior to use.

6. Spike QC and all samples with 25 µl of Mass Labeled PFAS Extraction Standard Solution (PFC_ST_XXXXX). Spike LCS/MS/MSD with 200 µl of mid-level native spike (PFC_1633_MID_XXXXX). Spike LLCS with 400 µl of native spike (PFC_1633_LOW_XXXXX). Vortex/Shake containers to mix thoroughly.

7. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges. Adjust the vacuum to pass the samples through the cartridge at 5 mL/min.

8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 1:1 0.1M formic acid/methanol and pass the rinses through the cartridge using vacuum. Dry the cartridge by pulling air through for 15 seconds. Discard the rinse solution.

9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge ensuring the delivery needles to do not touch the sides of the tubes.

10. Rinse the inside of each empty sample/QC bottle with 5mL of 1% methanolic ammonium hydroxide.

11. Using a glass pipette, transfer the rinse from the bottles to the SPE reservoirs, washing the walls of the reservoirs. Set empty bottles aside to air dry.

12. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

13. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

14. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix.

15. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

Note: The instrument lab chemist performs the next steps.

16. Add 10 mg of Superclean Envi-Carb to each sample and batch QC extracts using a 10 mg scoop.

17. Handshake occasionally for no more than 5 minutes. Immediately vortex and centrifuge for 10 minutes.

18. Add 25 uL of Mass Labeled PFAS Injection Standard Solution (PFC_ST_XXXXX) to a clean 15-mL polypropylene centrifuge collection tube.

19. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 5 mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the plunger and filter the entire extract into the new collection tube containing the

internal standard.

20. QS each sample extract using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

21. Cap and vortex to mix.

22. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

23. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for dilution or reinjection if needed.

C. LC/MS/MS Analysis

1. Mass Calibration and Tuning

   a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

   b. When masses fall outside of the ±0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Acquisition method: See *Attachment 3*. Mass Transitions: See *Attachment 1*.

4. Instrument Sensitivity Check (ISC) and Instrument Blanks
   a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within ±30% of their true values for 90% of the native and isotopically labeled compounds, with the other recoveries achieving 50-150%. The signal-to-noise ratio must be greater than or equal to 3:1. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
   b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes TDCA RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

   Example Sample Sequence:
   1. Instrument blank
   2. Instrument blank
   3. Instrument blank
   4. Instrument Sensitivity Check (CCVIS _CAL1)
   5. CCV 1_CAL4
   6. Linear Branched/TDCA marker (WDM)
   7. Instrument Blank (ICB)
   8. Method Blank (MB)
   9. Low Level LCS (LLCS)
   10. LCS
   11. Sample (10 or less)
   12. CCV 2_CAL4
   13. Instrument Blank

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

   a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.

   b. If LLCS and LCS recoveries are acceptable, proceed to sample quantitation.

   c. If the LCS and LLCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS must be reextracted.

   d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) is(are) again outside the QC limit(s), consult a supervisor to determine the appropriate course of action based on batch and sample results.

11. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

12. Compare the retention times of all of the analytes, surrogates, and internals standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than

    a. 0.4 minutes for isotopically-labeled compounds

    b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound

    c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

    If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

13. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of 13C4-PFBA, 13C5-PFPeA, 13C4-PFHpA, 13C8-PFOA, 13C9-PFNA, 13C6-PFDA, 13C7-PFUnA, 13C2-PFDA,13C2-PFDoDA 13C2-PFTeDA, 13C8-PFOSA, D3-NMePFOSA, D5-NEtFOSAA, D3-NMeFOSAA, D5-NEtPFOSA, D7-NMePFOSAE, D9-NEtPFOSAE, 13C3-PFBA, 13C4-PFOA, 13C5-PFNA, 13C2-PFOA, 18O2-PFHxS, PFBA, PFECA F(PFMPA), PFECA A(PFMBA), NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.

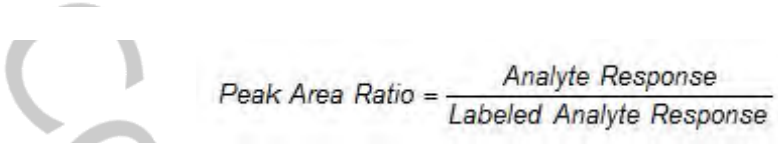
14. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.

15. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract with Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of labeled internal injection standard in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring dilutions greater than 10X should be reextracted using a reduced aliquot.

    Dilution Example 1/10: Mix 895 µl of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 µl of sample extract and 5 uL of injection standard. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio



$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

   On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

   On-column Concentration = (peak area ratio - intercept) ÷ slope

4. Sample Concentration

   Sample concentration (ng/l) = (On-column concentration x Final Sample Volume x DF) ÷ Initial Sample Volume

5. Ion Ratio

   ion ration = (peak area or height of quantifier)/(peak area or height of qualifier)

6. See *T-PEST-WI9847* for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are statistically derived when sufficient data points are available. If sufficient data points are not available to generate statistical windows advisory limits will be used.

QC parameter	Lower acceptance limit	High acceptance limit
Extracted Internal standard (EIS)	20%	150%
Non-extracted Internal Standard (NIS)	>30% of the average NIS from the initial calibration	200%

Analyte recoveries LCS/LLCS/MS/MSD	40%	150%

Note: lower acceptance limit for EIS cannot not be <20%, lower acceptance limit for analyte recovery cannot be <40%.

Historical data for MS/Ds, LCSs, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to *QA-SOP11892* for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank and an LCS/LLCS (Milli Q water spiked with all compounds to be determined carried through the entire procedure) must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

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QA-SOP11178 Demonstrations of Capability
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation

T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PFAS-WI21568 Manifold and N-EVAP Cleaning for PFAS Extractions
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End of document

Version history

Version	Approval	Revision information	
1	20.MAY.2022		

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.8
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

Attachment 1

Compound	Parent Ion	Daughter Ion
13C8-PFOA	421.1	376.0
PFOA	413.0	369.0
PFOA (2)	413.0	169.0
13C4-PFOS	502.8	79.9
13C4-PFOS (2)	502.8	98.9
13C8-PFOS	507.1	79.9
13C8-PFOS (2)	507.1	98.9
PFOS	498.9	79.9
PFOS (2)	498.9	98.8
13C5-PFNA	468.0	423.0
13C9-PFNA	472.1	427.0
PFNA	463.0	419.0
PFNA (2)	463.0	219.0
13C8-PFOSA	506.1	77.8
PFOSA	498.1	77.9
PFOSA (2)	498.1	478.0
PFNS	548.8	79.9
PFNS (2)	548.8	98.8
13C2-PFDA	515.1	470.1
13C6-PFDA	519.1	474.1
PFDA	512.9	469.0
PFDA (2)	512.9	219.0
13C2-8:2-FTS	529.1	80.9
13C2-8:2-FTS (2)	529.1	509.0
8:2-FTS	527.1	507.0
8:2-FTS (2)	527.1	80.8
d7-NMePFOSAE	623.2	58.9
NMePFOSAE	616.1	58.9
d3-NMePFOSA	515.0	219.0
NMEPFOSA	511.9	219.0
NMEPFOSA (2)	511.9	169.0
d3-NMeFOSAA	573.2	419.0
NMeFOSAA	570.1	419.0
NMeFOSAA (2)	570.1	483.0
d9-NEtPFOSAE	639.2	58.9
NEtPFOSAE	630.0	58.9
d5-NETPFOSA	531.1	219.0
NEtPFOSA	526.0	219.0
NEtPFOSA (2)	526.0	169.0
PFDS	599.0	79.9

Attachment 1

Compound	Parent Ion	Daughter Ion
PFDS (2)	599.0	98.8
13C7-PFUnDA	570.0	525.1
PFUnDA	563.1	519.0
PFUnDA (2)	563.1	269.1
d5-NEtFOSAA	589.2	419.0
NEtFOSAA	584.2	419.1
NEtFOSAA (2)	584.2	526.0
13C2-PFDoDA	615.1	570.0
PFDoDA	613.1	569.0
PFDoDA (2)	613.1	319.0
PFDoS	699.1	79.9
PFDoS (2)	699.1	98.8
PFTrDA	663.0	619.0
PFTrDA (2)	663.0	168.9
13C2-PFTeDA	715.2	670.0
PFTeDA	713.1	669.0
PFTeDA (2)	713.1	168.9
13C3-HFPODA	286.9	168.9
13C3-HFPODA (2)	286.9	184.9
HFPODA	284.9	168.9
HFPODA (2)	284.9	184.9
DONA	376.9	250.9
DONA (2)	376.9	84.8
9Cl-PF3ONS	530.8	351.0
9Cl-PF3ONS (2)	532.8	353.0
11Cl-PF3OUdS	630.9	450.9
11Cl-PF3OUdS (2)	632.9	452.9
PFECA B (NFDHA)	295.0	201.0
PFECA B(NFDHA) (2)	295.0	84.9
PFECA F (PFMPA)	229.0	84.9
3:3 FTCA	241.0	177.0
3:3 FTCA (2)	241.0	117.0
PFECA A (PFMBA)	279.0	85.1
PFEESA (PES)	314.8	134.9
PFEESA (PES) (2)	314.8	82.9
5:3 FTCA	341.0	237.1
5:3 FTCA (2)	341.0	217.0
7:3 FTCA	441.0	316.9
7:3 FTCA (2)	441.0	336.9

Native 1633 Low-Level Spike								
Solution Name	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Native 1633 Low-Level Spike (ppb)**
Wellington	PFAC-MXF	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1890	0.05	5mL	236.250
		9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1870			233.750
		4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1890			236.250
		Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000			250.000
Wellington	PFAC-MXH	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	3840	0.03		480.000
		1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	3750			468.750
		1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	3800			475.000
		N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	1000			125.000
		N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	1000			125.000
		Perfluorobutanesulfonic acid	375-73-5	PFBS	887			110.875
		Perfluorobutanoic acid	375-22-4	PFBA	4000			500.000
		Perfluorodecanesulfonic acid	335-77-3	PFDS	965			120.625
		Perfluorodecanoic acid	335-76-2	PFDA	1000			125.000
		Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	970			121.250
		Perfluorododecanoic acid	307-55-1	PFDoDA	1000			125.000
		Perfluoroheptanesulfonic acid	375-92-8	PFHpS	953			119.125
		Perfluoroheptanoic acid	375-85-9	PFHpA	1000			125.000
		Perfluorohexanesulfonic acid	355-46-4	PFHxS	914			114.250
		Perfluorohexanoic acid	307-24-4	PFHxA	1000			125.000
		Perfluorononanesulfonic acid	68259-12-1	PFNS	962			120.250
		Perfluorononanoic acid	375-95-1	PFNA	1000			125.000
		Perfluorooctanesulfonamide	754-91-6	PFOSA	1000			125.000
		Perfluorooctanesulfonic acid	1763-23-1	PFOS	928			116.000
		Perfluorooctanoic acid	335-67-1	PFOA	1000			125.000
		Perfluoropentanesulfonic acid	2706-91-4	PFPeS	941			117.625
		Perfluoropentanoic acid	2706-90-3	PFPeA	2000			250.000
		Perfluorotetradecanoic acid	376-06-7	PFTeDA	1000			125.000
		Perfluorotridecanoic acid	72629-94-8	PFTrDA	1000			125.000
		Perfluoroundecanoic acid	2058-94-8	PFUnDA	1000			125.000
Wellington	PFAC-MXG	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	2000	0.03		250.000
		Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	2000			250.000
		Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	2000			250.000
		Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	1780			222.500
Wellington	PFAC-MXI	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	10000	0.03		1250.000
		N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	1000			125.000
		2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	10000			1250.000
		N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	1000			125.000
Wellington	PFAC-MXJ	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	4000	0.03		25.000
		3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	20000			125.000
		3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	20000			125.000

1633 Initial Calibration Standards Preparation

Solution Name	MDL	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Native Replacement PFAS Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.020	0.050	0.250
Native Perfluoroalkyl Ether Carboxylic Acids and Sulfonate Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.010	0.025	0.125
Native PFAS Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.010	0.025	0.125
Native N-NMe/EtFOSA & N-Nme/EtFOSE Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.010	0.025	0.125
Native X:3 Flourotelomer Caroxylic Acid Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.0125	0.0312	0.1560
Native PFAS Intermediate A Aliquot (mL)	0.008	0.016	0.040	0.100	0.200	NA	NA	NA
Native PFAS Intermediate B Aliquot (mL)	0.010	0.020	0.050	0.125	0.250	NA	NA	NA
Mass-Labelled PFAS Injection Standard Solution/Mixture - IS Aliquot (mL)	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010
Mass-Labelled PFAS Extraction Standard Solution/Mixture - ES Aliquot (mL)	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010
Final Volume (mL)	**2**	2	2	2	2	2	2	2

1633 Initial Calibration Standards Concentrations							
	1	2	3	4	5	6	7
Compound Name	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)
PFBA	0.8	2	5	10	20	50	250
PFPeA	0.4	1	2.5	5	10	25	125
PFHxA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFHpA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFOA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFNA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFUnA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDoA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFTrDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFTeDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFBS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFPeS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFHxS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFHpS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFOS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFNS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDoS	0.2	0.5	1.25	2.5	5	12.5	62.5
4:2FTS	0.8	2	5	10	20	50	NA
6:2FTS	0.8	2	5	10	20	50	NA
8:2FTS	0.8	2	5	10	20	50	NA
PFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSAA	0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSAA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSE	2	5	12.5	25	50	125	625
NEtFOSE	2	5	12.5	25	50	125	625
HFPO-DA	0.8	2	5	10	20	50	250
ADONA	0.8	2	5	10	20	50	250
PFMPA	0.4	1	2.5	5	10	25	125
PFMBA	0.4	1	2.5	5	10	25	125
NFDHA	0.4	1	2.5	5	10	25	125
9Cl-PF3ONS	0.8	2	5	10	20	50	250
11Cl-PF3OUdS	0.8	2	5	10	20	50	250
PFEESA	0.4	1	2.5	5	10	25	125
3:3FTCA	1	2.5	6.26	12.5	25	62.4	312
5:3FTCA	5	12.5	31.3	62.5	125	312	1560
7:3FTCA	5	12.5	31.3	62.5	125	312	1560
^{13}C4-PFBA	10	10	10	10	10	10	10
^{13}C5-PFPeA	5	5	5	5	5	5	5
^{13}C5-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C4-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C8-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C9-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C6-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C7-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C2-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C2-PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C3-PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C3-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C8-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C2-4:2 FTS	5	5	5	5	5	5	5
^{13}C2-6:2 FTS	5	5	5	5	5	5	5
^{13}C2-8:2 FTS	5	5	5	5	5	5	5
^{13}C8-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSAA	5	5	5	5	5	5	5
D5-NEtFOSAA	5	5	5	5	5	5	5
D7-NMeFOSE	25	25	25	25	25	25	25
D9-NEtFOSE	25	25	25	25	25	25	25
^{13}C3-HFPO-DA	10	10	10	10	10	10	10
^{13}C3-PFBA	5	5	5	5	5	5	5
^{13}C2-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C4-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C5-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C2-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{18}O2-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C4-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5

TDCA Stock Solution								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (mg)**	**Aliquot (g)**	**Final Volume**	**Final Conc. TDCA Stock Solution (ppb)**
Sigma Alrich	T0557-500MG	Sodium Taurodeoxycholate hydrate	207737-97-1	TDCA	1000000	0.05	50mL	2000000

TDCA Working Solution A							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mg)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. TDCA Working Solution A (ppb)**
TDCA Stock Intermediate	Sodium Taurodeoxycholate hydrate	207737-97-1	TDCA	2000000	1.25	4mL	625000

TDCA Working Solution B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mg)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. TDCA Working Solution B (ppb)**
TDCA Working Solution A	Sodium Taurodeoxycholate hydrate	207737-97-1	TDCA	625000	0.16	5mL	20000

1633 Linear/Branched TDCA Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. 1633 Linear/Branched TDCA Intermediate (ppb)**
Wellington	T-PFOA	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7TG	T-PFOA	500	0.02	2mL	500
Camridge Isotope Laboratories, Inc.	ULM-11036-S	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500	0.02		500
Camridge Isotope Laboratories, Inc.	ULM-11034-S	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500	0.02		500
Camridge Isotope Laboratories, Inc.	ULM-10780-S	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500	0.01		500
Camridge Isotope Laboratories, Inc.	ULM-10779-S	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500	0.01		500
Camridge Isotope Laboratories, Inc.	ULM-10977-S	Perfluorooctanesulfonamide	754-91-6	PFOSA	500	0.02		500
Wellington	ipPFNA0516	Perfluoro-7-methyloctanoic acid	15899-31-7	PF7MOA	500	0.02		500

1633 Linear/Branched TDCA Solution							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. 1633 Linear/Branched TDCA Solution (ppb)**
TDCA Working Solution B	Sodium Taurodeoxycholate hydrate	207737-97-1	TDCA	5000	0.01	2mL	25
1633 Linear/Branched TDCA Intermediate	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500	0.02		5
	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500			5
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500			5
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			5
	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			5
	Perfluoro-7-methyloctanoic acid	15899-31-7	PF7MOA	500			5

PFAS 1633 ICV Working Standard							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ug/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. PFAS 1633 ICV Working Standard (ppb)**
Native PFAS Intermediate A	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	94.500	0.20	2mL	9.450
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	93.500			9.350
	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	94.500			9.450
	Perfluoro(2-propoxypropanoic) acid	13252-13-6	HFPODA	100.000			10.000
	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	93.750			4.690
	1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	95.000			4.755
	1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	96.000			4.800
	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	25.000			2.500
	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	25.000			2.500
	Perfluorobutanesulfonic acid	375-73-5	PFBS	22.175			2.218
	Perfluorobutanoic acid	375-22-4	PFBA	100.000			10.000
	Perfluorodecanesulfonic acid	335-77-3	PFDS	24.125			2.413
	Perfluorodecanoic acid	335-76-2	PFDA	25.000			2.500
	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	24.250			2.425
	Perfluorododecanoic acid	307-55-1	PFDoDA	25.000			2.500
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	23.825			2.383
	Perfluoroheptanoic acid	375-85-9	PFHpA	25.000			2.500
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	22.850			2.285
	Perfluorohexanoic acid	307-24-4	PFHxA	25.000			2.500
	Perfluorononanesulfonic acid	68259-12-1	PFNS	24.050			2.405
	Perfluorononanoic acid	375-95-1	PFNA	25.000			2.500
	Perfluorooctanesulfonamide	754-91-6	PFOSA	25.000			2.500
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	23.200			2.320
	Perfluorooctanoic acid	335-67-1	PFOA	25.000			2.500
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	23.525			2.353
	Perfluoropentanoic acid	2706-90-3	PFPeA	50.000			5.000
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	25.000			2.500
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	25.000			2.500
	Perfluoroundecanoic acid	2058-94-8	PFUnDA	25.000			2.500
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50.000			5.000
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50.000			5.000
	Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50.000			5.000
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44.500			4.450
	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	250.000			25.000
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	25.000			2.500
	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	250.000			25.000
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	25.000			2.500
Native PFAS Intermediate B	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	100.000	0.25		12.500
	3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	500.000			62.500
	3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	500.000			62.500
MPFACHIFES	Perfluoro-n-[^{13}C4]butanoic acid	STL00992	^{13}C4-PFBA	2000	0.01		10.000
	Perfluoro-n-[^{13}C5]pentanoic acid	STL01893	^{13}C5-PFPeA	1000			5.000
	Perfluoro-n-[1,2,3,4,6-^{13}C5]hexanoic acid	STL02577	^{13}C5 -PFHxA	500			2.500
	Perfluoro-n-[1,2,3,4-^{13}C4]heptanoic acid	STL01892	^{13}C4-PFHpA	500			2.500
	Perfluoro-n-[^{13}C8]octanoic acid	STL01052	^{13}C8-PFOA	5050			2.500
	Perfluoro-n-[^{13}C9]nonanoic acid	STL02578	^{13}C9-PFNA	250			1.250
	Perfluoro-n-[1,2,3,4,5,6-^{13}C6]decanoic acid	STL02579	^{13}C6-PFDA	250			1.250
	Perfluoro-n-[1,2,3,4,5,6,7-^{13}C7]undecanoic acid	STL02580	^{13}C7-PFUnA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]dodecanoic acid	STL02703	^{13}C2-PFDoA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]tetradecanoic acid	STL02116	^{13}C2-PFTeDA	250			1.250
	Perfluoro-1-[2,3,4-^{13}C3]butanesulfonic acid	STL02337	^{13}C3-PFBS	466			2.330
	Perfluoro-1-[1,2,3-^{13}C3]hexanesulfonic acid	STL02581	^{13}C3-PFHxS	474			2.370
	Perfluoro-1-[^{13}C8]octanesulfonic acid	STL01054	^{13}C8-PFOS	479			2.395
	Perfluoro-1-[^{13}C8]octanesulfonamide	STL01056	^{13}C8 -PFOSA	500			2.500
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	1000			5.000
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	1000			5.000
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]hexan sulfonic acid	STL02395	^{13}C2-4:2FTS	938			4.690
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]octanesulfonic acid	STL02279	^{13}C2-6:2FTS	951			4.755
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]decanesulfonic acid	STL02280	^{13}C2-8:2FTS	960			4.800
	Tetrafluoro-2-heptafluoropropoxy-^{13}C3-propanoic acid	STL02255	^{13}C3-HFPO-DA	2000			10.000
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	5000			25.000
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	5000			25.000
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	500			5.000
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	500			5.000
MPFACHIFES	Perfluoro-n-[2,3,4-^{13}C3]butanoic acid	STL02680	^{13}C3-PFBA	1000	0.01		5.000
	Perfluoro-n-[1,2,3,4-^{13}C4]octanoic acid	STL00990	^{13}C4-PFOA	500			2.500
	Perfluoro-n-[1,2-^{13}C2]decanoic acid	STL00996	^{13}C2-PFDA	250			1.250
	Perfluoro-n-[1,2,3,4-^{13}C4]octanesulfonic acid	STL00991	^{13}C4-PFOS	479			2.395
	Perfluoro-n-[1,2,3,4,5-^{13}C5] nonanoic acid	STL00995	^{13}C5-PFNA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]hexanoic acid	STL00993	^{13}C2-PFHxA	500			2.500
	Perfluoro-1-hexane[^{18}O2]sulfonic acid	STL00994	^{18}O2-PFHxS	474			2.370

1633 Labeled Ampulated Standards						
Ampulated Solution Name	**Vendor**	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**
MPFACHIFES	Wellington	MPFACHIFES	Perfluoro-n-[^{13}C4]butanoic acid	STL00992	^{13}C4-PFBA	2000
			Perfluoro-n-[^{13}C5]pentanoic acid	STL01893	^{13}C5-PFPeA	1000
			Perfluoro-n-[1,2,3,4,6-^{13}C5]hexanoic acid	STL02577	^{13}C5 -PFHxA	500
			Perfluoro-n-[1,2,3,4-^{13}C4]heptanoic acid	STL01892	^{13}C4-PFHpA	500
			Perfluoro-n-[^{13}C8]octanoic acid	STL01052	^{13}C8-PFOA	5050
			Perfluoro-n-[^{13}C9]nonanoic acid	STL02578	^{13}C9-PFNA	250
			Perfluoro-n-[1,2,3,4,5,6-^{13}C6]decanoic acid	STL02579	^{13}C6-PFDA	250
			Perfluoro-n-[1,2,3,4,5,6,7-^{13}C7]undecanoic acid	STL02580	^{13}C7-PFUnA	250
			Perfluoro-n-[1,2-^{13}C2]dodecanoic acid	STL02703	^{13}C2-PFDoA	250
			Perfluoro-n-[1,2-^{13}C2]tetradecanoic acid	STL02116	^{13}C2-PFTeDA	250
			Perfluoro-1-[2,3,4-^{13}C3]butanesulfonic acid	STL02337	^{13}C3-PFBS	466
			Perfluoro-1-[1,2,3-^{13}C3]hexanesulfonic acid	STL02581	^{13}C3-PFHxS	474
			Perfluoro-1-[^{13}C8]octanesulfonic acid	STL01054	^{13}C8-PFOS	479
			Perfluoro-1-[^{13}C8]octanesulfonamide	STL01056	^{13}C8 -PFOSA	500
			N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	1000
			N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	1000
			1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]hexan sulfonic acid	STL02395	^{13}C2-4:2FTS	938
			1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]octanesulfonic acid	STL02279	^{13}C2-6:2FTS	951
			1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]decanesulfonic acid	STL02280	^{13}C2-8:2FTS	960
			Tetrafluoro-2-heptafluoropropoxy-^{13}C3-propanoic acid	STL02255	^{13}C3-HFPO-DA	2000
			N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	5000
			N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	5000
			N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	500
			N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	500
MPFACHIFES	Wellington	MPFACHIFIS	Perfluoro-n-[2,3,4-^{13}C3]butanoic acid	STL02680	^{13}C3-PFBA	1000
			Perfluoro-n-[1,2,3,4-^{13}C4]octanoic acid	STL00990	^{13}C4-PFOA	500
			Perfluoro-n-[1,2-^{13}C2]decanoic acid	STL00996	^{13}C2-PFDA	250
			Perfluoro-n-[1,2,3,4-^{13}C4]octanesulfonic acid	STL00991	^{13}C4-PFOS	479
			Perfluoro-n-[1,2,3,4,5-^{13}C5] nonanoic acid	STL00995	^{13}C5-PFNA	250
			Perfluoro-n-[1,2-^{13}C2]hexanoic acid	STL00993	^{13}C2-PFHxA	500
			Perfluoro-1-hexane[^{18}O2]sulfonic acid	STL00994	^{18}O2-PFHxS	474

Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

Inj Std	Internal Standard/Injection Standard
I13C3-PFBA	13C3-PFBA
I13C2-PFHxA	13C2-PFHxA
I13C4-PFOA	13C4-PFOA
I13C5-PFNA	13C5-PFNA
I13C2-PFDA	13C2-PFDA
I18O2-PFHxS	18O2-PFHxS
I13C4-PFOS	13C4-PFOS

Extraction Standards

Extraction Standard	Internal Standard
E13C4-PFBA	13C3-PFBA
E13C5-PFPeA	13C2-PFHxA
E13C5-PFHxA	
E13C4-PFHpA	
E13C3-HFPO-DA	
E13C8-PFOA	13C4-PFOA
E13C9-PFNA	13C5-PFNA
E13C6-PFDA	13C2-PFDA
E13C7-PFUnA	
E13C2-PFDoA	
E13C2-PFTeDA	
E13C3-PFBS	18O2-PFHxS
E13C3-PFHxS	
E13C2-4:2-FTS	
E13C2-6:2-FTS	
E13C2-8:2-FTS	

Extraction Standard	Internal Standard
E13C8-PFOS	13C4-PFOS
E13C8-PFOSA	
Ed3-NMeFOSA	
Ed5-NEtFOSA	
Ed3-NMeFOSAA	
Ed7-NMeFOSE	
Ed9-NEtFOSE	

Native PFAS Compounds

Native	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	

Native	Extraction Standard
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

1633 Native Ampulated Standards						
Ampulated Solution Name	**Vendor**	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**
Native Replacement PFAS Solution/Mixture	Wellington	PFAC-MXF	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1890
			9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1870
			4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1890
			Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000
Native PFAS Solution/Mixture	Wellington	PFAC-MXH	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	3840
			1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	3750
			1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	3800
			N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	1000
			N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	1000
			Perfluorobutanesulfonic acid	375-73-5	PFBS	887
			Perfluorobutanoic acid	375-22-4	PFBA	4000
			Perfluorodecanesulfonic acid	335-77-3	PFDS	965
			Perfluorodecanoic acid	335-76-2	PFDA	1000
			Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	970
			Perfluorododecanoic acid	307-55-1	PFDoDA	1000
			Perfluoroheptanesulfonic acid	375-92-8	PFHpS	953
			Perfluoroheptanoic acid	375-85-9	PFHpA	1000
			Perfluorohexanesulfonic acid	355-46-4	PFHxS	914
			Perfluorohexanoic acid	307-24-4	PFHxA	1000
			Perfluorononanesulfonic acid	68259-12-1	PFNS	962
			Perfluorononanoic acid	375-95-1	PFNA	1000
			Perfluorooctanesulfonamide	754-91-6	PFOSA	1000
			Perfluorooctanesulfonic acid	1763-23-1	PFOS	928
			Perfluorooctanoic acid	335-67-1	PFOA	1000
			Perfluoropentanesulfonic acid	2706-91-4	PFPeS	941
			Perfluoropentanoic acid	2706-90-3	PFPeA	2000
			Perfluorotetradecanoic acid	376-06-7	PFTeDA	1000
			Perfluorotridecanoic acid	72629-94-8	PFTrDA	1000
			Perfluoroundecanoic acid	2058-94-8	PFUnDA	1000
Native Perfluoroalkyl Ether Carboxylic Acids and Sulfonate Solution/Mixture	Wellington	PFAC-MXG	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	2000
			Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	2000
			Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	2000
			Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	1780
Native N-NMe/EtFOSA & N-Nme/EtFOSE Solution/Mixture	Wellington	PFAC-MXI	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	10000
			N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	1000
			2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	10000
			N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	1000
Native X:3 Flourotelomer Caroxylic Acid Solution/Mixture	Wellington	PFAC-MXJ	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	4000
			3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	20000
			3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	20000

Attachment 3

Acquisition Method	Mass Spectrometer Method Properties
EPA1633_DOD Mass Spec 10.500 min Period 10.500 min -MRM Integrated Valve Sciex LC System Equilibrate Injection	

Period 1:

Scans in Period: 1050
Min. Dwell Time: 3 ms
Max. Dwell Time: 250 ms
Relative Start Time: 0.00 msec
Scheduled Ionization: Off
Experiments in Period: 1
Use target Cycle Time: No
Target Cycle Time: N/A

Period 1 Experiment 1:

Scan Type: MRM (MRM)
Scheduled MRM: Yes
Polarity: Negative
Scan Mode: N/A
Ion Source: Turbo Spray
sMRM Q1Q3 Resolution: No
MRM detection window: 60 sec
Target Scan Time: 0.6000 sec
Resolution Q1: Unit
Resolution Q3: Unit
Intensity Thres.: 0.00 cps
Settling Time: 0.0000 msec
MR Pause: 5.0070 msec
MCA: No
Step Size: 0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	3.88	DP	-40.00	-40.00	13C3-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
217.000	172.000	3.88	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.000	223.000	4.44	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Attachment 3

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.000	80.000	4.49	DP	-120.00	-120.00	13C3-PFBS
			CE	-65.00	-65.00	
329.000	81.000	4.83	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	
315.000	270.000	4.86	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	
318.000	273.000	4.86	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	
287.000	169.000	5.00	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	
367.000	322.000	5.27	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	
402.000	80.000	5.27	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	
359.000	294.000	5.42	DP	-40.00	-40.00	13C2-6:2 FTUCA
			CE	-25.00	-25.00	

Attachment 3

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
379.000	294.000	5.43	DP	-30.00	-30.00	13C2-6:2 FTCA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.000	81.000	5.63	DP	-100.00	-100.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
415.000	370.000	5.65	DP	-50.00	-50.00	13C2-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.000	172.000	5.65	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.000	376.000	5.65	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
503.000	99.000	5.98	DP	-100.00	-100.00	13C4-PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.000	99.000	5.98	DP	-100.00	-100.00	13C8-PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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472.000	427.000	5.99	DP	-50.00	-50.00	13C9-PFNA	
			CE	-18.00	-18.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
459.000	394.000	6.13	DP	-50.00	-50.00	13C2-8:2 FTUCA	
			CE	-25.00	-25.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
479.000	394.000	6.13	DP	-35.00	-35.00	13C2-8:2 FTCA	
			CE	-25.00	-25.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
519.000	474.000	6.30	DP	-50.00	-50.00	13C6-PFDA	
			CE	-18.00	-18.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
515.000	470.000	6.30	DP	-50.00	-50.00	13C2-PFDA	
			CE	-18.00	-18.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
529.000	81.000	6.31	DP	-100.00	-100.00	13C2-8:2-FTS	
			CE	-42.00	-42.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
506.000	78.000	6.40	DP	-100.00	-100.00	13C8-PFOSA	
			CE	-80.00	-80.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
573.000	419.000	6.40	DP	-80.00	-80.00	d3-NMeFOSAA	
			CE	-30.00	-30.00		

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
565.000	520.000	6.58	DP	-70.00	-70.00	13C2-PFUnDA
			CE	-19.00	-19.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.000	6.58	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.000	419.000	6.50	DP	-90.00	-90.00	d5-NEtFOSAA
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
559.000	494.000	6.70	DP	-60.00	-60.00	13C2-10:2 FTUCA
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
579.000	494.000	6.72	DP	-50.00	-50.00	13C2-10:2 FTCA
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.000	570.000	6.81	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.000	59.000	6.85	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	6.86	DP	-100.00	-100.00	d3-NMePFOSA

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			CE	-37.00	-37.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.000	59.000	7.01	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.000	219.000	7.03	DP	-100.00	-100.00	d5-NEtPFOSA
			CE	-38.00	-38.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.000	670.000	7.21	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
163.000	119.000	1.83	DP	-30.00	-30.00	PPF Acid
			CE	-15.00	-15.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
213.000	169.000	3.89	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
249.000	99.000	4.12	DP	-60.00	-60.00	PFPrS
			CE	-40.00	-40.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	85.000	4.17	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	4.49	DP	-60.00	-60.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	4.43	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
299.000	80.000	4.49	DP	-120.00	-120.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.000	4.62	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.000	135.000	4.71	DP	-60.00	-60.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	4.84	DP	-70.00	-70.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.000	307.000	4.83	DP	-100.00	-100.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	4.86	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.000	80.000	4.89	DP	-90.00	-90.00	PFPeS
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
285.000	169.000	5.00	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.000	319.000	5.27	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
399.000	80.000	5.27	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
377.000	251.000	5.32	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.000	5.40	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
357.000	293.000	5.42	DP	-45.00	-45.00	6:2 FTUCA
			CE	-25.00	-25.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
377.000	293.000	5.44	DP	-45.00	-45.00	6:2 FTCA
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
461.000	381.000	5.63	DP	-70.00	-70.00	PFECHS
			CE	-40.00	-40.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.000	407.000	5.62	DP	-100.00	-100.00	6:2-FTS
			CE	-35.00	-35.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	80.000	5.63	DP	-100.00	-100.00	PFHpS
			CE	-90.00	-90.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	5.65	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
499.000	80.000	5.90	DP	-100.00	-100.00	PFOS
			CE	-100.00	-100.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	5.99	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	317.000	6.13	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
457.000	393.000	6.13	DP	-50.00	-50.00	8:2 FTUCA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
477.000	393.000	6.15	DP	-45.00	-45.00	8:2 FTCA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.000	351.000	6.12	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
549.000	80.000	6.28	DP	-100.00	-100.00	PFNS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
513.000	469.000	6.30	DP	-50.00	-50.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.000	507.000	6.30	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	78.000	6.40	DP	-100.00	-100.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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570.000 419.000 6.40 DP -80.00 -80.00 NMeFOSAA
CE -30.00 -30.00

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	80.000	6.54	DP	-100.00	-100.00	PFDS
			CE	-120.00	-120.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.000	519.000	6.58	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.000	419.000	6.50	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
557.000	493.000	6.70	DP	-70.00	-70.00	10:2 FTUCA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
631.000	451.000	6.68	DP	-100.00	-100.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
577.000	493.000	6.72	DP	-60.00	-60.00	10:2 FTCA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.000	569.000	6.99	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
627.000	607.000	6.84	DP	-100.00	-100.00	10:2-FTS
			CE	-47.00	-47.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.000	59.000	6.85	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.000	219.000	6.86	DP	-100.00	-100.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.000	80.000	6.99	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	59.000	7.01	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.03	DP	-100.00	-100.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	619.000	7.03	DP	-60.00	-60.00	PFTrDA
			CE	-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.000	669.000	7.21	DP	-60.00	-60.00	PFTeDA

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				CE	-22.00	-22.00

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
813.000	769.000	7.51	DP	-100.00	-100.00	PFHxDA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
913.000	869.000	7.74	DP	-100.00	-100.00	PFODA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
299.000	99.000	4.50	DP	-100.00	-100.00	PFBS_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	85.000	4.45	DP	-25.00	-25.00	PFECA B_2
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.000	81.000	4.83	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	119.000	4.86	DP	-50.00	-50.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.000	99.000	4.89	DP	-100.00	-100.00	PFPeS_2
			CE	-50.00	-50.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
285.000	185.000	5.00	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
385.000	185.000	5.00	DP	-75.00	-75.00	HFPODA_3
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.000	169.000	5.27	DP	-60.00	-60.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
399.000	99.000	5.27	DP	-100.00	-100.00	PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	5.40	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
461.000	99.000	5.63	DP	-60.00	-60.00	PFECHS_2
			CE	-60.00	-60.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.000	81.000	5.62	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	99.000	5.63	DP	-100.00	-100.00	PFHpS_2
			CE	-80.00	-80.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	5.65	DP	-60.00	-60.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
499.000	99.000	5.97	DP	-100.00	-100.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	5.99	DP	-60.00	-60.00	PFNA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
549.000	99.000	6.28	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
513.000	219.000	6.30	DP	-50.00	-50.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.000	81.000	6.30	DP	-100.00	-100.00	8:2 FTS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	483.000	6.40	DP	-80.00	-80.00	NMeFOSAA_2
			CE	-24.00	-24.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	99.000	6.54	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.000	269.000	6.58	DP	-80.00	-80.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.000	526.000	6.50	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.000	319.000	6.81	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
627.000	81.000	6.84	DP	-120.00	-120.00	10:2 FTS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	169.000	7.03	DP	-60.00	-60.00	PFTrDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.000	169.000	7.21	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
813.000	169.000	7.51	DP	-80.00	-80.00	PFHxDA_2
			CE	-45.00	-45.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
913.000	169.000	7.74	DP	-80.00	-80.00	PFODA_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
179.000	85.000	2.90	DP	-15.00	-15.00	PFMOAA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	241.000	3.92	DP	-80.00	-80.00	R-PSDA
			CE	-32.00	-32.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
405.000	217.000	3.92	DP	-60.00	-60.00	R-EVE
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
439.000	343.000	3.94	DP	-80.00	-80.00	Hydrolized PSDA
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	185.000	4.06	DP	-20.00	-20.00	PMPA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
297.000	135.000	4.17	DP	-80.00	-80.00	NVHOS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

Attachment 3

245.000	85.000	4.37	DP	-10.00	-10.00	PFO2HxA	
			CE	-15.00	-15.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	235.000	4.59	DP	-10.00	-10.00	PEPA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
311.000	85.000	4.97	DP	-20.00	-20.00	PFO3OA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.000	283.000	5.27	DP	-40.00	-40.00	Hydro-EVE Acid
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
397.000	217.000	5.27	DP	-80.00	-80.00	R-PSDCA
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	263.000	5.26	DP	-80.00	-80.00	Hydro-PS Acid
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
379.000	185.000	5.38	DP	-35.00	-35.00	PFECA-G
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
377.000	84.000	5.48	DP	-20.00	-20.00	PFO4DA
			CE	-40.00	-40.00	

Attachment 3

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
443.000	147.000	5.53	DP	-70.00	-70.00	PS Acid
			CE	-32.00	-32.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
407.000	263.000	5.55	DP	-40.00	-40.00	EVE Acid
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
443.000	85.000	5.93	DP	-7.00	-7.00	PFO5DA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
175.000	97.000	1.46	DP	-45.00	-45.00	MTP
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	5.99	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	84.000	5.27	DP	-100.00	-100.00	1802-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	69.000	4.43	DP	-40.00	-40.00	PFPeA_2
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	478.000	6.40	DP	-100.00	-100.00	PFOSA_2

Attachment 3

				CE	-80.00	-80.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.000	169.000	6.86	DP	-100.00	-100.00	NMePFOSA_2
				CE	-37.00	-37.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.03	DP	-180.00	-180.00	NEtPFOSA_2
				CE	-40.00	-40.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
377.000	85.000	5.32	DP	-40.00	-40.00	DONA_2
				CE	-20.00	-20.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
533.000	353.000	6.12	DP	-100.00	-100.00	9Cl-PF3ONS_2
				CE	-38.00	-38.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
633.000	453.000	6.68	DP	-180.00	-180.00	11Cl-PF3OUdS_2
				CE	-40.00	-40.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	4.49	DP	-60.00	-60.00	3:3 FTCA_2
				CE	-12.00	-12.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	337.000	6.13	DP	-80.00	-80.00	7:3 FTCA_2
				CE	-20.00	-20.00

Attachment 3

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.000	83.000	4.71	DP	-60.00	-60.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.000	99.000	6.99	DP	-100.00	-100.00	PFDoS_2
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.000	4.86	DP	-180.00	-180.00	13C5-PFHxA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.000	99.000	4.49	DP	-120.00	-120.00	13C3-PFBS_2
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.000	99.000	5.27	DP	-100.00	-100.00	13C3-PFHxS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.000	80.000	5.98	DP	-100.00	-100.00	13C8-PFOS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.000	309.000	4.83	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.000	409.000	5.63	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Attachment 3

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.000	509.000	6.31	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
287.000	185.000	5.00	DP	-20.00	-20.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.000	119.000	4.86	DP	-30.00	-30.00	13C2-PFHxA_2
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
503.000	80.000	5.98	DP	-100.00	-100.00	13C4-PFOS_2
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
CAD:	10.00
IS:	-3000.00
TEM:	350.00
GS1:	40.00
GS2:	50.00
EP	-10.00
CXP	-14.00

ST1927333A

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

PFAC-MXC

Native Perfluorinated Compound Solution/Mixture

PRODUCT CODE:	PFAC-MXC
LOT NUMBER:	PFACMXC0617
SOLVENT(S):	Methanol / Water (<1%)
DATE PREPARED: (mm/dd/yyyy)	06/14/2017
LAST TESTED: (mm/dd/yyyy)	03/19/2019
EXPIRY DATE: (mm/dd/yyyy)	03/19/2024
RECOMMENDED STORAGE:	Store ampoule in a cool, dark place

DESCRIPTION:

PFAC-MXC is a solution/mixture of thirteen native perfluoroalkylcarboxylic acids (C_4-C_{14}, C_{16}, and C_{18}) and eight native perfluoroalkylsulfonates (C_4-C_{10} and C_{12}). The full name, abbreviation and concentration for each of the components are given in Table A.

The individual perfluoroalkylcarboxylic acids and perfluoroalkylsulfonates all have chemical purities of >98%.

DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture
Figure 1: LC/MS Data (SIR)
Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compounds it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters $x_1, x_2, \ldots x_n$ on which it depends is:

$$u_c(y(x_1, x_2, \ldots x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of ±5% (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A 1226), and ISO 17034 by ANSI-ASQ National Accreditation Board (ANAB; AR-1523).

CALA Testing Accreditation No. A1226

ANAB ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at www.well-labs.com or contact us directly at info@well-labs.com

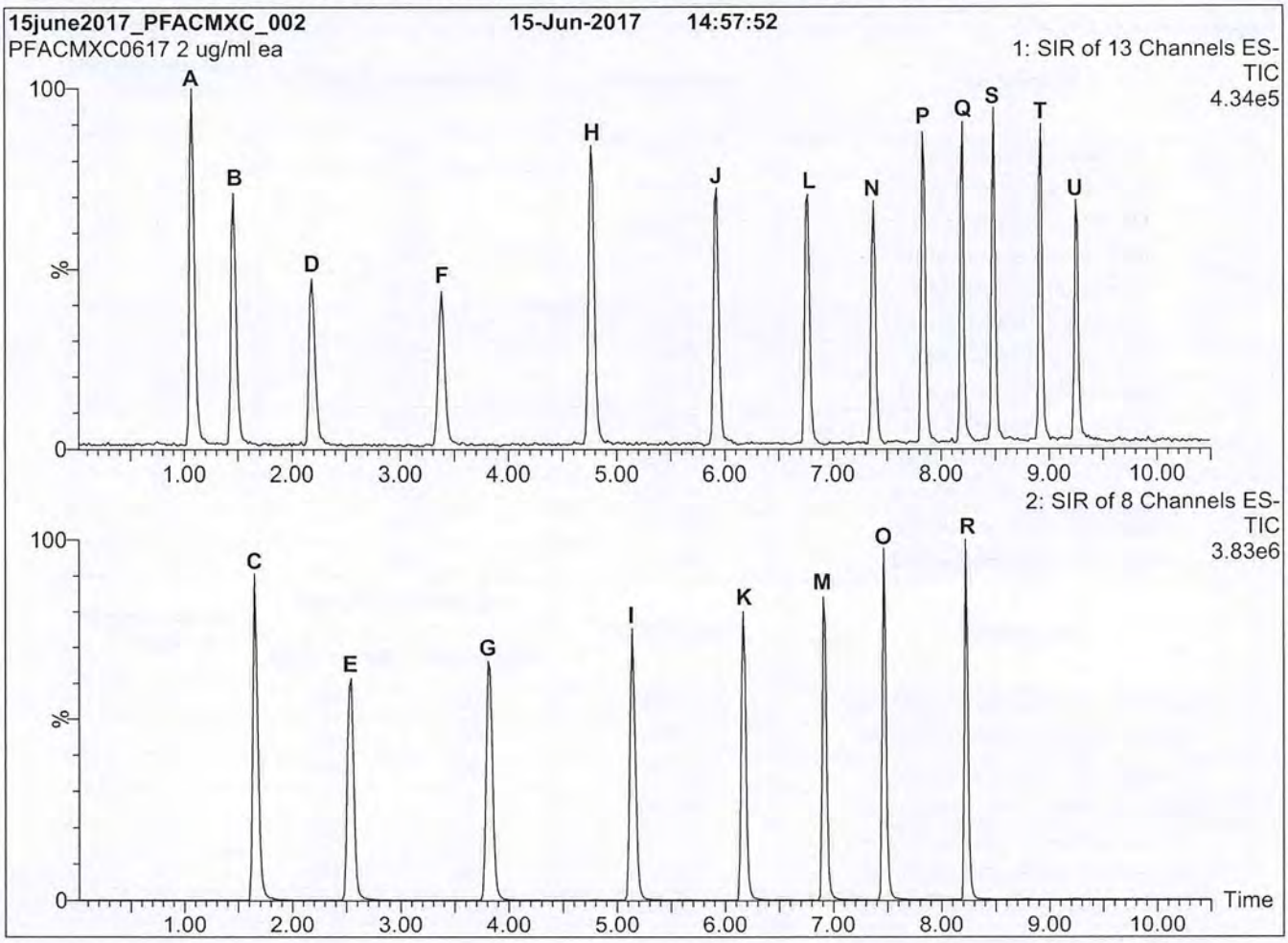
Table A: **PFAC-MXC; Components and Concentrations (ng/ml, ± 5% in Methanol / Water (<1%))**

Compound	Abbreviation	Concentration (ng/ml)*		Peak Assignment in Figure 1
Perfluoro-n-butanoic acid	PFBA	2000		A
Perfluoro-n-pentanoic acid	PFPeA	2000		B
Perfluoro-n-hexanoic acid	PFHxA	2000		D
Perfluoro-n-heptanoic acid	PFHpA	2000		F
Perfluoro-n-octanoic acid	PFOA	2000		H
Perfluoro-n-nonanoic acid	PFNA	2000		J
Perfluoro-n-decanoic acid	PFDA	2000		L
Perfluoro-n-undecanoic acid	PFUdA	2000		N
Perfluoro-n-dodecanoic acid	PFDoA	2000		P
Perfluoro-n-tridecanoic acid	PFTrDA	2000		Q
Perfluoro-n-tetradecanoic acid	PFTeDA	2000		S
Perfluoro-n-hexadecanoic acid	PFHxDA	2000		T
Perfluoro-n-octadecanoic acid	PFODA	2000		U
Compound	**Abbreviation**	**Concentration (ng/ml)***		**Peak Assignment in Figure 1**
		As the salt	**As the anion**	
Potassium perfluoro-1-butanesulfonate	L-PFBS	2000	1770	C
Sodium perfluoro-1-pentanesulfonate	L-PFPeS	2000	1880	E
Sodium perfluoro-1-hexanesulfonate	L-PFHxS	2000	1890	G
Sodium perfluoro-1-heptanesulfonate	L-PFHpS	2000	1900	I
Sodium perfluoro-1-octanesulfonate	L-PFOS	2000	1910	K
Sodium perfluoro-1-nonanesulfonate	L-PFNS	2000	1920	M
Sodium perfluoro-1-decanesulfonate	L-PFDS	2000	1930	O
Sodium perfluoro-1-dodecanesulfonate	L-PFDoS	2000	1940	R

* Concentrations have been rounded to three significant figures.

Certified By: _____ **Date:** 06/06/2019
(mm/dd/yyyy)
B.G. Chittim, General Manager

Figure 1: **PFAC-MXC; LC/MS Data (Total Ion Current Chromatogram; SIR)**



Conditions for Figure 1:

LC: **Waters Acquity Ultra Performance LC**
MS: **Micromass Quattro *micro* API MS**

Chromatographic Conditions

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H_2O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions in 1 min.

Time: 12 min

Flow: 300 µl/min

MS Parameters

Experiment: SIR of 21 Channels

Source: Electrospray (negative)
Capillary Voltage (kV) = 3.00
Cone Voltage (V) = variable (10-80)
Cone Gas Flow (l/hr) = 50
Desolvation Gas Flow (l/hr) = 750

Figure 2: **PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)**

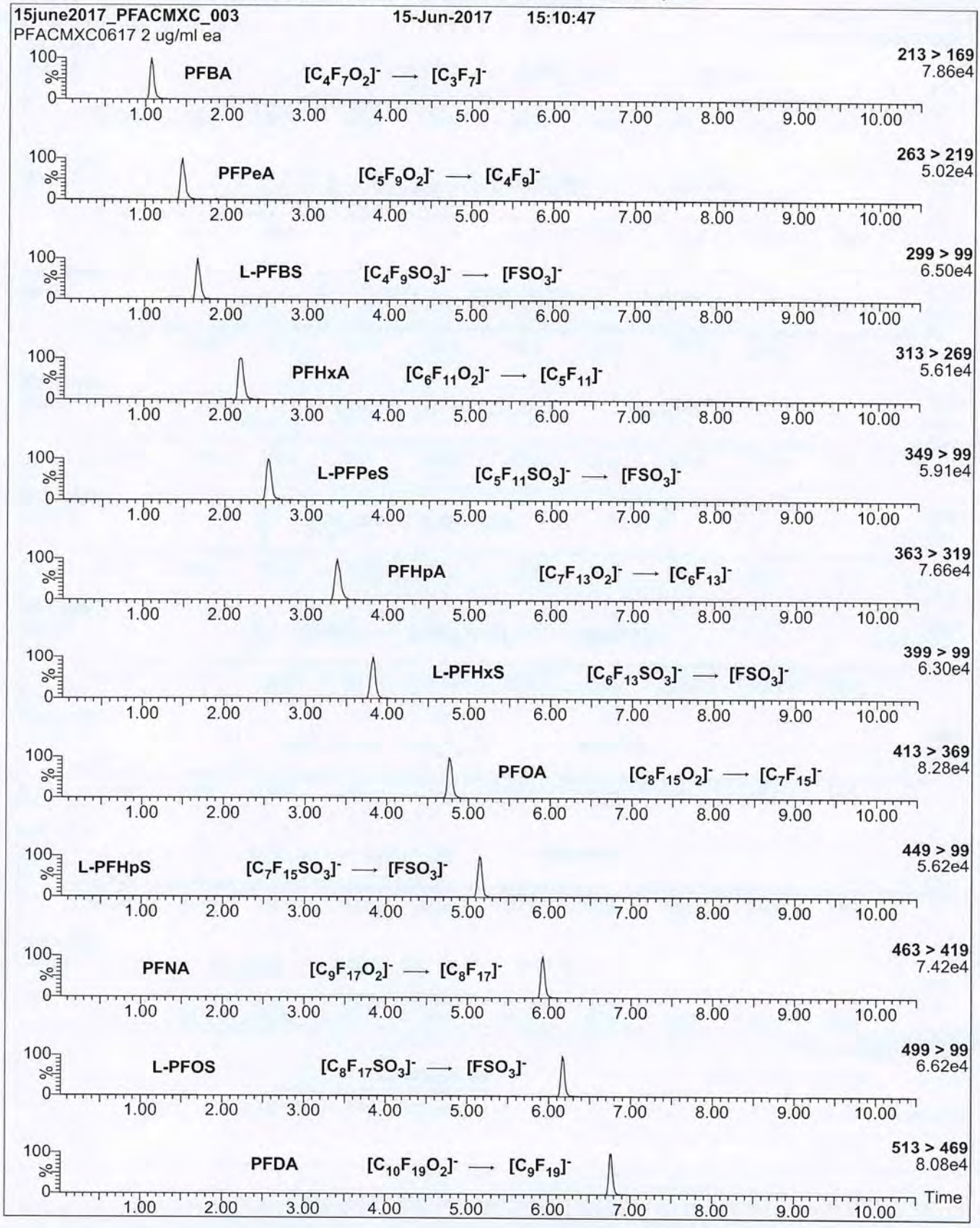
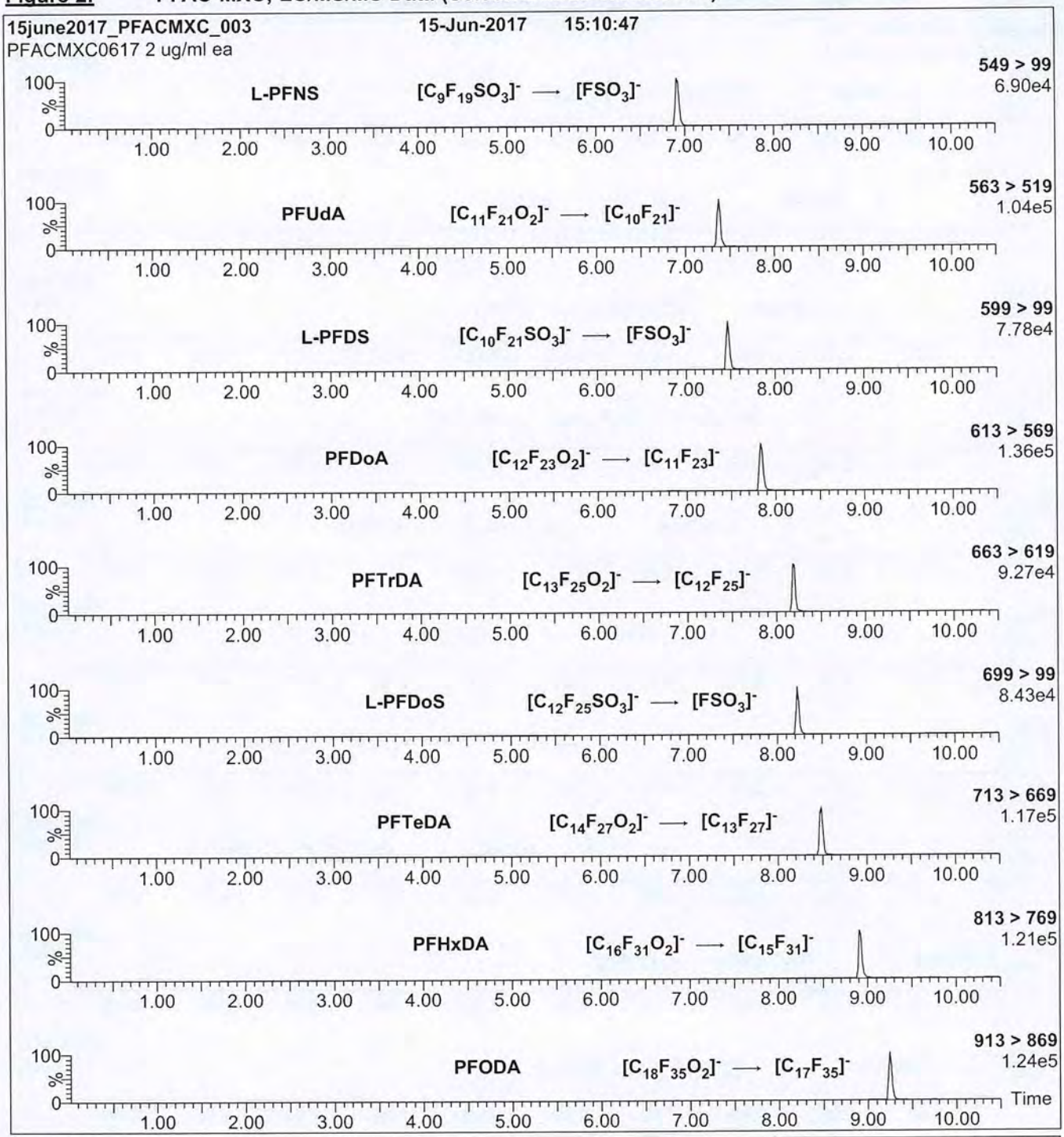


Figure 2: **PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (PFAC-MXC)

Mobile phase: Same as Figure 1

Flow: 300 µl/min

MS Parameters
Collision Gas (mbar) = 3.46e-3
Collision Energy (eV) = 8-50 (variable)

Native PFAS Intermediate A								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Native PFAS Intermediate A (ppb)**
Wellington	PFAC-MXF	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1890	0.10	2mL	94.500
		9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1870			93.500
		4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1890			94.500
		Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000			100.000
Wellington	PFAC-MXH	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	3840	0.05		93.750
		1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	3750			95.000
		1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	3800			96.000
		N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	1000			25.000
		N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	1000			25.000
		Perfluorobutanesulfonic acid	375-73-5	PFBS	887			22.175
		Perfluorobutanoic acid	375-22-4	PFBA	4000			100.000
		Perfluorodecanesulfonic acid	335-77-3	PFDS	965			24.125
		Perfluorodecanoic acid	335-76-2	PFDA	1000			25.000
		Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	970			24.250
		Perfluorododecanoic acid	307-55-1	PFDoDA	1000			25.000
		Perfluoroheptanesulfonic acid	375-92-8	PFHpS	953			23.825
		Perfluoroheptanoic acid	375-85-9	PFHpA	1000			25.000
		Perfluorohexanesulfonic acid	355-46-4	PFHxS	914			22.850
		Perfluorohexanoic acid	307-24-4	PFHxA	1000			25.000
		Perfluorononanesulfonic acid	68259-12-1	PFNS	962			24.050
		Perfluorononanoic acid	375-95-1	PFNA	1000			25.000
		Perfluorooctanesulfonamide	754-91-6	PFOSA	1000			25.000
		Perfluorooctanesulfonic acid	1763-23-1	PFOS	928			23.200
		Perfluorooctanoic acid	335-67-1	PFOA	1000			25.000
		Perfluoropentanesulfonic acid	2706-91-4	PFPeS	941			23.525
		Perfluoropentanoic acid	2706-90-3	PFPeA	2000			50.000
		Perfluorotetradecanoic acid	376-06-7	PFTeDA	1000			25.000
		Perfluorotridecanoic acid	72629-94-8	PFTrDA	1000			25.000
		Perfluoroundecanoic acid	2058-94-8	PFUnDA	1000			25.000
Wellington	PFAC-MXG	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	2000	0.05		50.000
		Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	2000			50.000
		Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	2000			50.000
		Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	1780			44.500
Wellington	PFAC-MXI	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	10000	0.05		250.000
		N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	1000			25.000
		2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	10000			250.000
		N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	1000			25.000

Native PFAS Intermediate B								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ug/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Native PFAS Intermediate B (ppb)**
Wellington	PFAC-MXJ	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	4	0.05	2mL	100.000
		3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	20			500.000
		3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	20			500.000

Working Labeled Extraction Standard Spike							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Working Labeled Extraction Standard Spike (ppb)**
MPFACHIFES	Perfluoro-n-[^{13}C4]butanoic acid	STL00992	^{13}C4-PFBA	2000	0.01	5mL	10.000
	Perfluoro-n-[^{13}C5]pentanoic acid	STL01893	^{13}C5-PFPeA	1000			5.000
	Perfluoro-n-[1,2,3,4,6-^{13}C5]hexanoic acid	STL02577	^{13}C5 -PFHxA	500			2.500
	Perfluoro-n-[1,2,3,4-^{13}C4]heptanoic acid	STL01892	^{13}C4-PFHpA	500			2.500
	Perfluoro-n-[^{13}C8]octanoic acid	STL01052	^{13}C8-PFOA	5050			2.500
	Perfluoro-n-[^{13}C9]nonanoic acid	STL02578	^{13}C9-PFNA	250			1.250
	Perfluoro-n-[1,2,3,4,5,6-^{13}C6]decanoic acid	STL02579	^{13}C6-PFDA	250			1.250
	Perfluoro-n-[1,2,3,4,5,6,7-^{13}C7]undecanoic acid	STL02580	^{13}C7-PFUnA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]dodecanoic acid	STL02703	^{13}C2-PFDoA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]tetradecanoic acid	STL02116	^{13}C2-PFTeDA	250			1.250
	Perfluoro-1-[2,3,4-^{13}C3]butanesulfonic acid	STL02337	^{13}C3-PFBS	466			2.330
	Perfluoro-1-[1,2,3-^{13}C3]hexanesulfonic acid	STL02581	^{13}C3-PFHxS	474			2.370
	Perfluoro-1-[^{13}C8]octanesulfonic acid	STL01054	^{13}C8-PFOS	479			2.395
	Perfluoro-1-[^{13}C8]octanesulfonamide	STL01056	^{13}C8 -PFOSA	500			2.500
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	1000			5.000
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	1000			5.000
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]hexan sulfonic acid	STL02395	^{13}C2-4:2FTS	938			4.690
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]octanesulfonic acid	STL02279	^{13}C2-6:2FTS	951			4.755
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]decanesulfonic acid	STL02280	^{13}C2-8:2FTS	960			4.800
	Tetrafluoro-2-heptafluoropropoxy-^{13}C3-propanoic acid	STL02255	^{13}C3-HFPO-DA	2000			10.000
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	5000			25.000
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	5000			25.000
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	500			5.000
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	500			5.000

Working Internal Standard Spike							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Working Internal Standard Spike (ppb)**
MPFACHIFIS	Perfluoro-n-[2,3,4-^{13}C3]butanoic acid	STL02680	^{13}C3-PFBA	1000	0.01	5mL	5.000
	Perfluoro-n-[1,2,3,4-^{13}C4]octanoic acid	STL00990	^{13}C4-PFOA	500			2.500
	Perfluoro-n-[1,2-^{13}C2]decanoic acid	STL00996	^{13}C2-PFDA	250			1.250
	Perfluoro-n-[1,2,3,4-^{13}C4]octanesulfonic acid	STL00991	^{13}C4-PFOS	479			2.395
	Perfluoro-n-[1,2,3,4,5-^{13}C5] nonanoic acid	STL00995	^{13}C5-PFNA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]hexanoic acid	STL00993	^{13}C2-PFHxA	500			2.500
	Perfluoro-1-hexane[^{18}O2]sulfonic acid	STL00994	^{18}O2-PFHxS	474			2.370

Native 1633 Mid-Level Spike								
Solution Name	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Native 1633 Mid-Level Spike (ppb)**
Wellington	PFAC-MXF	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1890	0.63	5mL	236.250
		9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1870			233.750
		4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1890			236.250
		Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000			250.000
Wellington	PFAC-MXH	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	3840	0.31		480.000
		1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	3750			468.750
		1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	3800			475.000
		N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	1000			125.000
		N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	1000			125.000
		Perfluorobutanesulfonic acid	375-73-5	PFBS	887			110.875
		Perfluorobutanoic acid	375-22-4	PFBA	4000			500.000
		Perfluorodecanesulfonic acid	335-77-3	PFDS	965			120.625
		Perfluorodecanoic acid	335-76-2	PFDA	1000			125.000
		Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	970			121.250
		Perfluorododecanoic acid	307-55-1	PFDoDA	1000			125.000
		Perfluoroheptanesulfonic acid	375-92-8	PFHpS	953			119.125
		Perfluoroheptanoic acid	375-85-9	PFHpA	1000			125.000
		Perfluorohexanesulfonic acid	355-46-4	PFHxS	914			114.250
		Perfluorohexanoic acid	307-24-4	PFHxA	1000			125.000
		Perfluorononanesulfonic acid	68259-12-1	PFNS	962			120.250
		Perfluorononanoic acid	375-95-1	PFNA	1000			125.000
		Perfluorooctanesulfonamide	754-91-6	PFOSA	1000			125.000
		Perfluorooctanesulfonic acid	1763-23-1	PFOS	928			116.000
		Perfluorooctanoic acid	335-67-1	PFOA	1000			125.000
		Perfluoropentanesulfonic acid	2706-91-4	PFPeS	941			117.625
		Perfluoropentanoic acid	2706-90-3	PFPeA	2000			250.000
		Perfluorotetradecanoic acid	376-06-7	PFTeDA	1000			125.000
		Perfluorotridecanoic acid	72629-94-8	PFTrDA	1000			125.000
		Perfluoroundecanoic acid	2058-94-8	PFUnDA	1000			125.000
Wellington	PFAC-MXG	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	2000	0.31		250.000
		Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	2000			250.000
		Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	2000			250.000
		Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	1780			222.500
Wellington	PFAC-MXI	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	10000	0.39		1250.000
		N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	1000			125.000
		2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	10000			1250.000
		N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	1000			125.000
Wellington	PFAC-MXJ	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	4000	0.03		312.500
		3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	20000			1562.500
		3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	20000			1562.500

eurofins	Always check on-line for validity.	Level:
Document number: **T-PFAS-WI48593**	**Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24**	**Work Instruction**
Old Reference:		
Version: **1**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date **26-MAY-2022**	Document users: **5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep**	Responsible: **5_EUUSLA_PFAS_Manager**

Revision Log

Revision:	1	Effective date: This version
Section	**Justification**	**Changes**
Revision Log	NEW	NEW

Reference

1. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft method 1633), Department of Defense Quality System Manual Version 5.4, Table B-24.

2. US EPA Method 1633, *Analysis of Per and Polyfluoroalkyl Substances(PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS*, Version DRAFT, August 2021.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-PFAS-WI21568	Manifold and N-EVAP Cleaning for PFAS Extractions
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
QA-SOP11178	Demonstrations of Capability
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in solid samples. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2

Analyte	Acronym	CAS#
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonic acid	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6

Analyte	Acronym	CAS#
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

A solid sample is fortified with isotopically-labeled extraction standards. The sample extract is shaken, centrifuged, and the supernatant decanted. Carbon cleanup is performed on each sample extract. Sample extract is diluted to volume and then concentrated. The sample is then passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. The extract is fortified with Isotopically-labeled injection internal standards and filtered. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.

3. All parts of the SPE manifold must be cleaned as per *T-PFAS-WI21568*.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste

disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level.

See *QA-SOP11178* for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 500 mL HDPE widemouth sample bottle or jar with linerless HDPE or polypropylene caps. Collect samples as grab samples using wide-mouth jar and fill no more than ¾ full. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Solid and Biosolid samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.

2. Solid and Biosolid Samples stored in the lab must protected from light and held at a temperature of 0° to 6°C, or $\leq$ -20°C until extraction.

3. Solid and Biosolid samples must be extracted within 90 days. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C.

Note: Biosolid samples stored under refrigeration may produce gases that may cause sample to be expelled from the container when opened. This may produce noxious odors. It is recommended to store frozen if extraction will not occur for a few days.

Apparatus and Equipment

A. Apparatus

1. 500-mL HDPE bottles: Scientific Specialties; # 334008-blk-1, or equivalent.
2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 05-539-5 or equivalent
3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288 or equivalent.
4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC.
5. Analytical Balance – Capable of weighing to 0.0001 g
6. Top-Loading Balance – Capable of weighing to 0.01 g
7. Solid phase extraction (SPE) Weak Anion Exchange ("WAX") cartridge – Agilent; Sampli-Q WAX Polymer; 150mg/6mL; Cat. # 5982-3667.
8. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product # 54258-U.
9. SPE Tube Adapter - Millipore-Sigma; Product # 57020-U.
10. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.
11. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.
12. Centrifuge – "Q-Sep 3000"; Restek Corp. Cat. No. 26230, or equivalent, capable of a minimum rotational speed of 3000 rpm.
13. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent.
14. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. FisherScientific cat # 14-287-150, or equivalent.
15. Polypropylene pipette tips: 0-200µl. Fisher; Cat. No. 02-681-135
16. Polypropylene pipette tips: 101-1000µl. Fisher, Cat. No. 02-707-508
17. Pipettes – Disposable transfer. FisherScientific, Cat. No. 13-711-7M
18. Vortex mixer, variable speed, Fisher Scientific or equivalent.
19. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control. Organomation, Inc. Cat. #11250, or equivalent.

20. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTPF08831.

21. Thermo Target PP Polyspring inserts, catalog number C4010-630P

22. Agilent 9mm vial kit pack, catalog number 5190-2278, or equivalent

23. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent

24. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125

25. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product # 11-865-130.

26. pH paper, range 0-14, Whatman Panpeha or equivalent, 0.5 unit readability

27. Syringe filter - Acrodisc, Syringe Filter, GHP,13 mm, 0.2 µm, Aqueous, 100/pkg, Part # WAT097962.

28. Silanized glass wool (Sigma-Alrich, Cat #20411 or equivalent

29. Disposable syringe filter, 25-mm, 0.2um Nylon membrane, PALL/Acrodisc or equivalent

30. Glass fiber filter, 47 mm, 1 um, PALL A/E or equivalent

31. Variable speed mixing table (Fisherbrand™ Nutating mixer or equivalent

32. Evaporation/concentrator tubes: 60 mL clear glass vial, 30x125 mm, without caps (Wheaton Cat # W226060 or eqvalent).

33. Wooden Tongue Depressors - Fisher; Cat. # 11-700-555, or equivalent.

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

   ExionLC Controller
   ExionLC AC Pump
   ExionLC AC Autosampler
   Exion AC Column Oven
   Data system –Analyst 1.6.3

2. HPLC columns

   a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-YO or equivalent

   b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

1. Methanol (MeOH) – Honeywell Burdick and Jackson "Chromasolv LC-MS" grade Cat. No. BJ34966-4L or equivalent
2. Acetonitrile (ACN) – Fisher Scientific, Optima Cat. No. A955-4 or equivalent
3. Ammonium acetate – Fisher Scientific, Cat. No. A637-500 or equivalent
4. Ammonium hydroxide (NH_4OH), 5.0 M; Ricca, Cat. No. 644-32 or equivalent
5. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
6. Aqueous ammonium hydroxide (3%) – add ammonium hydroxide (10 mL, 30%) to reagent water (90 mL), store at room temperature, replace after 3 months
7. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
8. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
9. Acetic Acid – ACS grade or equivalent, store at room temperature
10. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
11. Formic acid
    a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years
    b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years
    c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years
    d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years

e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years

12. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.

13. Solids reference matrix – Ottawa or reagent-grade sand

14. 20 mM ammonium acetate solution – Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 2 L Milli-Q water and mix well. The solution is prone to volatility losses and is replaced weekly. Store at room temperature for up to one week. Different volumes can be prepared as long as final concentrations are equivalent.

15. 20 mM ammonium acetate solution in 0.5% Milli-Q water/methanol – Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 10mL of Milli-Q water to dissolve the Ammonium Acetate. Bring up to 2 L with methanol and mix well. Store at room temperature for up to one week or until degradation is observed. Different volumes can be prepared as long as final concentrations are equivalent.

B. Standards:

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10 ml Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent. After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in glass or HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 4*.
If the compound purity is assayed to be 96% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent, i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.

Attachment 5 - Native PFAS Intermediate A
Attachment 6 - Native PFAS Intermediate B
Attachment 7 - Working Labeled Extraction Standard Spike
Attachment 8 - Working Internal Standard Spike
Attachment 9 - Native 1633 Mid-Level Spike
Attachment 10 - Native 1633 Low-Level Spike
Attachment 11 - 1633 Initial Calibration Standards Preparation
Attachment 12 - 1633 Initial calibration Standards Concentrations
Attachment 13 - TDCA Stock Solution
Attachment 14 - TDCA Working Solution A
Attachment 15 - TDCA Working Solution B
Attachment 16 - 1633 Linear/Branched TDCA Intermediate
Attachment 17 - 1633 Linear/Branched TDCA Solution
Attachment 18 - PFAS 1633 ICV Working Standard
Attachment 19 - 1633 Labeled Ampulated Standards
Attachment 20 - 1633 Native Ampulated Standards

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit(quadratic). In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.

2. Analyze a Cal4 level standard that contains TDCA retention time marker and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

   Example Initial Calibration Sequence:
   1. Instrument Blank
   2. Instrument Blank
   3. Instrument Blank
   4. CAL 1
   5. CAL 2
   6. CAL 3
   7. CAL 4
   8. CAL 5
   9. CAL 6
   10. CAL 7
   11. ICB (Instrument Blank)
   12. ICV
   13. MDL
   14. WDM (Linear Branched/TDCA standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 2* for additional information about compound relationships.

4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

   NOTE: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA.

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	

PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS

6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

5. Fit the curve

   a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

   b. If the %RSD is greater than 20%, a linear regression with a concentratoin weighing factor of 1/x is tried for the compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%

   c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

   d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

   NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See *Attachment 4*.

6. Initial Calibration Verification (ICV)

   A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must

be within ±30% of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

2. Acceptance criteria

   a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

   b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

A. Sample Preparation

NOTE: Prior to weighing out samples, thoroughly mix each sample using a wooden tongue depressor or stainless steel spoon to ensure a homogeneous sample matrix. Stir from the bottom to the top in a circular motion along the sides of the jar, breaking particles to less than 1 mm by pressing against the side of the container. Remove rocks, invertebrates, and foreign objects. Vegetation can either be removed or cut into smaller pieces based on project requirements.

1. On a calibrated, top-loading balance, accurately weigh 5.0g ± 0.10g (0.5 g for biosolids) of solid sample into a tared, labeled 15-mL centrifuge tube using a disposable polypropylene spatula. Record sample weight in the prep entry system.
2. For each batch - maximum 20 samples - include the following quality control samples:
   a. Method Blank: Weigh 5.0g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 g (0.25 g for biosolids) of reagent water
   b. LCS: Fortify 5.0g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 g (0.25 g for biosolids) reagent water and spiked with 200 µL of Native Spiking Solution (PFC_1633_MID_XXXXX).
   c. LLCS: Fortify 5.0g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 g (0.25 g for biosolids) reagent water and spiked with 400 µL of Native Spiking Solution (PFC_1633_LOW_XXXXX).
   d. Matrix Spike/Matrix Spike Duplicate (MS/MSD): Fortify 5.0g ± 0.10 g (0.5 g for biosolids) of sample as specified in sample preparation log with 200 µL of Native Spiking Solution (PFC_1633_MID_XXXXX).
3. Add 25 µl working labeled extraction surrogate solution (PFC_1633_SS_XXXXX) to each sample/QC tube.
4. Cap and vortex for approximately 30 seconds.
5. Allow samples/QC to equilibrate for at least 30 minutes.
6. Add 10 mL of 0.3% methanolic ammonium hydroxide to each centrifuge tube.
7. Cap and vortex

8. Shake for 30 minutes on a variable speed mixing table
9. Centrifuge for 10 minutes and transfer supernatant to a clean 50 mL polypropylene centrifuge tube.
10. Add 15 mL of 0.3% methanolic ammonium hydroxide to the remaining solid sample in each centrifuge tube. Cap and vortex.
11. Shake for 30 minutes on a variable speed mixing table
12. Centrifuge for 10 minutes and decant the supernatant from the second extraction into the centrifuge tube with the supernatant from the first extraction.
13. Add another 5 mL of 0.3% methanolic ammonium hydroxide to the remaining sample in each centrifuge tube.
14. Shake by hand to disperse.
15. Immediately decant the supernatant from the third extraction into the centrifuge tube with the supernatant from the first and second extraction.
16. Using a 10 mg scoop, add 10 mg of Superclean Envi-Carb to the combined extract, mix by occasionally hand shaking for no more than 5 minutes.
17. Centrifuge for 10 minutes.
18. Immediately decant the extract into a new labeled 50ml PP centrifuge tube.
19. Concentrate the extracts at no more than 40°C with an N_2 flow of approximately 1.2 L/min to a final volume of approximately 3-5 mL.
20. Allow extracts to concentrate for 25 minutes, then mix (by vortex if the volume is < 20 mL or using a glass pipette if the volume is >20 mL).
21. Continue concentrating and mixing every 10 minutes until the extract has been reduced to the required volume.
22. Add enough reagent water to the extract to reach the "40ml" mark on the centrifuge tube and vortex. Check that the pH is 6.5 ± 0.5 and adjust as necessary with 50% formic acid or 30% ammonium hydroxide (or with 5% formic acid and 4% aqueous ammonium hydroxide).

Solid Phase Extraction (SPE)

1. Pack clean silanized glass wool to half the height of the WAX SPE cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

3. Condition each SPE cartridge with the following reagents in the following order:

    a. 15 mL 1% methanolic ammonium hydroxide

    b. 5 mL 0.3M formic acid

    c. Discard conditioning eluent(s)

4. Label each sample bottle, cap and reservoir with the same number to insure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges. Allow full volume to pass through each cartridge by gravity, if possible. Apply light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.

6. After full volume has passed through the cartridges, dry the cartridges with vacuum - no more than 15" of Hg - for approximately five minutes. After five minutes, visually inspect the cartridge to determine if the sorbent is dry. This done by comparing the cartridge to a visual standard (an unused SPE cartridge). If the sorbent is not dry, continue to check at one minute intervals until the cartridge is dry.

7. Discard the waste and rinse the waste reservoir with DI water. Wipe each needle with a Kim-wipe/methanol.

8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by **1:1 0.1M formic acid/methanol** and pass the rinses through the cartridge using vacuum. Dry the cartridge by pulling air through for 15 seconds. Discard the rinse solution.

9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge.

10. Rinse the inside of the evaporation/concentrator tube using 5mL of 1% methanolic ammonium hydroxide.

11. Using a glass pipette, transfer the rinse to the SPE reservoirs, washing the walls of the reservoirs.

12. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

13. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

14. Add 25 uL of concentrated acetic acid to each collection tube and swirl to mix.

Note: The instrument lab chemis performs the next steps.

15. Add 25 uL of Mass Labeled PFAS Injection Standard Solution (PFC_ST_XXXXX) to each sample extract.

16. QS each sample extract to 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

17. Cap and vortex to mix.

18. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 3 mL polypropylene syringe. Take the plunger out and carefully decant ~1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter ~1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

19. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for dilution or reinjection if needed.

C. LC/MS/MS Analysis

1. Mass Calibration and Tuning

   a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

   b. When masses fall outside of the ±0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer’s instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions.

Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Acquisition method: See *Attachment 3*. Mass Transitions: See *Attachment 1*.
4. Instrument Sensitivity Check (ISC) and Instrument Blanks
   a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within ±30% of their true value. The signal-to-noise ratio must be greater than or equal to 3:1. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
   b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes TDCA RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

   Example Sample Sequence:
   1. Instrument blank
   2. Instrument blank
   3. Instrument blank
   4. Instrument Sensitivity Check (CCVIS _CAL1)
   5. CCV 1_CAL4
   6. Linear Branched/TDCA marker (WDM)
   7. Instrument Blank (ICB)
   8. Method Blank (MB)
   9. Low Level LCS (LLCS)
   10. LCS
   11. Sample (10 or less)
   12. CCV 2_CAL4
   13. Instrument Blank

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

    a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.

    b. If LLCS and LCS recoveries are acceptable, proceed to sample quantitation.

    c. If the LCS and LLCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS/LLCS must be reextracted.

    d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) is(are) again outside the QC limit(s), consult a supervisor to determine the appropriate course of action based on batch and sample results.

11. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

12. Compare the retention times of all of the analytes, surrogates, and internals standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than

    a. 0.4 minutes for isotopically-labeled compounds

    b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound

    c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

    If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

13. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of 13C4-PFBA, 13C5-PFPeA, 13C4-PFHpA, 13C8-PFOA, 13C9-PFNA, 13C6-PFDA, 13C7-PFUnA, 13C2-PFDA,13C2-PFDoDA 13C2-PFTeDA, 13C8-PFOSA, D3-NMePFOSA, D5-NEtFOSAA, D3-NMeFOSAA, D5-NEtPFOSA, D7-NMePFOSAE, D9-NEtPFOSAE, 13C3-PFBA, 13C4-PFOA, 13C5-PFNA, 13C2-PFOA, 18O2-PFHxS, PFBA, PFECA F(PFMPA), PFECA A(PFMBA), NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.

14. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.

15. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydfrtoxide and 0.625% acetic acid solution and adjust the amount of labeled internal injection standard in the diluted extract. Select the dilution so tha the expected EIS recoveries in the diluted extract are >5%. Extracts requiring greater than a 10x dilution should be reextracted using a reduced aliquot.

    Dilution Example 1/10: Mix 895 µl of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 µl of sample extract and 5 uL of injection standard. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

    On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

    On-column Concentration = (peak area ratio - intercept) ÷ slope

4. Sample Concentration

    Sample concentration (ng/g) = (On-column concentration x Final Sample Volume x DF) ÷ Initial Sample Volume

5. Ion Ratio

    ion ration = (peak area or height of quantifier)/(peak area or height of qualifier)

5. See *T-PEST-WI9847* for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are statistically derived when sufficient data points are available. If sufficient data points are not available to generate statistical windows advisory limits will be used.

QC parameter	Lower acceptance limit	High acceptance limit
Extracted Internal standard (EIS)	20%	150%
Non-extracted Internal Standard (NIS)	>30% of the average NIS from the initial calibration	200%
Analyte recoveries LCS/LLCS/MS/MSD	40%	150%

Note: lower acceptance limit for EIS cannot not be <20%, lower acceptance limit for analyte recovery cannot be <40%.

Historical data for MS/Ds, LCSs, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to *QA-SOP11892* for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank and an LCS/LLCS (Milli Q water spiked with all compounds to be determined carried through the entire procedure) must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Attachment:
Attachment 1 - Mass Transitions (.doc)
Attachment 10 - Native Low Level Spike (.pdf)
Attachment 11 - 1633 Initial Calibration Standard Concentrations (.pdf)
Attachment 12 - 1633 Initial Calibration Standard Concentrations (.pdf)
Attachment 13 - TDCA Stock Solution (.pdf)
Attachment 14 - TDCA working Solution A (.pdf)
Attachment 15 - TDCA Working Solution B (.pdf)

Attachment 16 - 1633 Linear Branched and TDCA Intermediate (.pdf)
Attachment 17 - 1633 Linear Branched and TDCA Solution (.pdf)
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Attachment 19 - 1633 Labeled Ampulated Standards (.pdf)
Attachment 2 - Standards relationships (.docx)
Attachment 20 - 1633 Native Ampulated Standards (.pdf)
Attachment 3 - Acquisition Parameters (.pdf)
Attachment 4 - Example Certificate of Analysis (.pdf)
Attachment 5 - 1633 Native PFAS Intermediate A (.pdf)
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Attachment 9 - Native Mid Level Spike (.pdf)

QA-SOP11178 Demonstrations of Capability
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PFAS-WI21568 Manifold and N-EVAP Cleaning for PFAS Extractions
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Attachment: Attachment 9 - Native Mid Level Spike (pdf)

End of document

Version history

Version	Approval	Revision information	
1	26.MAY.2022		

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.8
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

Attachment 1

Compound	Parent Ion	Daughter Ion
13C8-PFOA	421.1	376.0
PFOA	413.0	369.0
PFOA (2)	413.0	169.0
13C4-PFOS	502.8	79.9
13C4-PFOS (2)	502.8	98.9
13C8-PFOS	507.1	79.9
13C8-PFOS (2)	507.1	98.9
PFOS	498.9	79.9
PFOS (2)	498.9	98.8
13C5-PFNA	468.0	423.0
13C9-PFNA	472.1	427.0
PFNA	463.0	419.0
PFNA (2)	463.0	219.0
13C8-PFOSA	506.1	77.8
PFOSA	498.1	77.9
PFOSA (2)	498.1	478.0
PFNS	548.8	79.9
PFNS (2)	548.8	98.8
13C2-PFDA	515.1	470.1
13C6-PFDA	519.1	474.1
PFDA	512.9	469.0
PFDA (2)	512.9	219.0
13C2-8:2-FTS	529.1	80.9
13C2-8:2-FTS (2)	529.1	509.0
8:2-FTS	527.1	507.0
8:2-FTS (2)	527.1	80.8
d7-NMePFOSAE	623.2	58.9
NMePFOSAE	616.1	58.9
d3-NMePFOSA	515.0	219.0
NMEPFOSA	511.9	219.0
NMEPFOSA (2)	511.9	169.0
d3-NMeFOSAA	573.2	419.0
NMeFOSAA	570.1	419.0
NMeFOSAA (2)	570.1	483.0
d9-NEtPFOSAE	639.2	58.9
NEtPFOSAE	630.0	58.9
d5-NETPFOSA	531.1	219.0
NEtPFOSA	526.0	219.0
NEtPFOSA (2)	526.0	169.0
PFDS	599.0	79.9

Attachment 1

Compound	Parent Ion	Daughter Ion
PFDS (2)	599.0	98.8
13C7-PFUnDA	570.0	525.1
PFUnDA	563.1	519.0
PFUnDA (2)	563.1	269.1
d5-NEtFOSAA	589.2	419.0
NEtFOSAA	584.2	419.1
NEtFOSAA (2)	584.2	526.0
13C2-PFDoDA	615.1	570.0
PFDoDA	613.1	569.0
PFDoDA (2)	613.1	319.0
PFDoS	699.1	79.9
PFDoS (2)	699.1	98.8
PFTrDA	663.0	619.0
PFTrDA (2)	663.0	168.9
13C2-PFTeDA	715.2	670.0
PFTeDA	713.1	669.0
PFTeDA (2)	713.1	168.9
13C3-HFPODA	286.9	168.9
13C3-HFPODA (2)	286.9	184.9
HFPODA	284.9	168.9
HFPODA (2)	284.9	184.9
DONA	376.9	250.9
DONA (2)	376.9	84.8
9Cl-PF3ONS	530.8	351.0
9Cl-PF3ONS (2)	532.8	353.0
11Cl-PF3OUdS	630.9	450.9
11Cl-PF3OUdS (2)	632.9	452.9
PFECA B (NFDHA)	295.0	201.0
PFECA B(NFDHA) (2)	295.0	84.9
PFECA F (PFMPA)	229.0	84.9
3:3 FTCA	241.0	177.0
3:3 FTCA (2)	241.0	117.0
PFECA A (PFMBA)	279.0	85.1
PFEESA (PES)	314.8	134.9
PFEESA (PES) (2)	314.8	82.9
5:3 FTCA	341.0	237.1
5:3 FTCA (2)	341.0	217.0
7:3 FTCA	441.0	316.9
7:3 FTCA (2)	441.0	336.9

Native 1633 Low-Level Spike								
Solution Name	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Native 1633 Low-Level Spike (ppb)**
Wellington	PFAC-MXF	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1890	0.05	5mL	236.250
		9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1870			233.750
		4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1890			236.250
		Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000			250.000
Wellington	PFAC-MXH	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	3840	0.03		480.000
		1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	3750			468.750
		1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	3800			475.000
		N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	1000			125.000
		N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	1000			125.000
		Perfluorobutanesulfonic acid	375-73-5	PFBS	887			110.875
		Perfluorobutanoic acid	375-22-4	PFBA	4000			500.000
		Perfluorodecanesulfonic acid	335-77-3	PFDS	965			120.625
		Perfluorodecanoic acid	335-76-2	PFDA	1000			125.000
		Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	970			121.250
		Perfluorododecanoic acid	307-55-1	PFDoDA	1000			125.000
		Perfluoroheptanesulfonic acid	375-92-8	PFHpS	953			119.125
		Perfluoroheptanoic acid	375-85-9	PFHpA	1000			125.000
		Perfluorohexanesulfonic acid	355-46-4	PFHxS	914			114.250
		Perfluorohexanoic acid	307-24-4	PFHxA	1000			125.000
		Perfluorononanesulfonic acid	68259-12-1	PFNS	962			120.250
		Perfluorononanoic acid	375-95-1	PFNA	1000			125.000
		Perfluorooctanesulfonamide	754-91-6	PFOSA	1000			125.000
		Perfluorooctanesulfonic acid	1763-23-1	PFOS	928			116.000
		Perfluorooctanoic acid	335-67-1	PFOA	1000			125.000
		Perfluoropentanesulfonic acid	2706-91-4	PFPeS	941			117.625
		Perfluoropentanoic acid	2706-90-3	PFPeA	2000			250.000
		Perfluorotetradecanoic acid	376-06-7	PFTeDA	1000			125.000
		Perfluorotridecanoic acid	72629-94-8	PFTrDA	1000			125.000
		Perfluoroundecanoic acid	2058-94-8	PFUnDA	1000			125.000
Wellington	PFAC-MXG	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	2000	0.03		250.000
		Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	2000			250.000
		Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	2000			250.000
		Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	1780			222.500
Wellington	PFAC-MXI	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	10000	0.03		1250.000
		N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	1000			125.000
		2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	10000			1250.000
		N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	1000			125.000
Wellington	PFAC-MXJ	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	4000	0.03		25.000
		3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	20000			125.000
		3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	20000			125.000

1633 Initial Calibration Standards Preparation

Solution Name	MDL	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Native Replacement PFAS Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.020	0.050	0.250
Native Perfluoroalkyl Ether Carboxylic Acids and Sulfonate Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.010	0.025	0.125
Native PFAS Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.010	0.025	0.125
Native N-NMe/EtFOSA & N-Nme/EtFOSE Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.010	0.025	0.125
Native X:3 Flourotelomer Caroxylic Acid Solution/Mixture Aliquot (mL)	NA	NA	NA	NA	NA	0.0125	0.0312	0.1560
Native PFAS Intermediate A Aliquot (mL)	0.008	0.016	0.040	0.100	0.200	NA	NA	NA
Native PFAS Intermediate B Aliquot (mL)	0.010	0.020	0.050	0.125	0.250	NA	NA	NA
Mass-Labelled PFAS Injection Standard Solution/Mixture - IS Aliquot (mL)	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010
Mass-Labelled PFAS Extraction Standard Solution/Mixture - ES Aliquot (mL)	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010
Final Volume (mL)	**2**	2	2	2	2	2	2	2

1633 Initial Calibration Standards Concentrations							
	1	2	3	4	5	6	7
Compound Name	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)
PFBA	0.8	2	5	10	20	50	250
PFPeA	0.4	1	2.5	5	10	25	125
PFHxA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFHpA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFOA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFNA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFUnA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDoA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFTrDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFTeDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFBS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFPeS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFHxS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFHpS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFOS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFNS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDS	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDoS	0.2	0.5	1.25	2.5	5	12.5	62.5
4:2FTS	0.8	2	5	10	20	50	NA
6:2FTS	0.8	2	5	10	20	50	NA
8:2FTS	0.8	2	5	10	20	50	NA
PFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSAA	0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSAA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSE	2	5	12.5	25	50	125	625
NEtFOSE	2	5	12.5	25	50	125	625
HFPO-DA	0.8	2	5	10	20	50	250
ADONA	0.8	2	5	10	20	50	250
PFMPA	0.4	1	2.5	5	10	25	125
PFMBA	0.4	1	2.5	5	10	25	125
NFDHA	0.4	1	2.5	5	10	25	125
9Cl-PF3ONS	0.8	2	5	10	20	50	250
11Cl-PF3OUdS	0.8	2	5	10	20	50	250
PFEESA	0.4	1	2.5	5	10	25	125
3:3FTCA	1	2.5	6.26	12.5	25	62.4	312
5:3FTCA	5	12.5	31.3	62.5	125	312	1560
7:3FTCA	5	12.5	31.3	62.5	125	312	1560
^{13}C4-PFBA	10	10	10	10	10	10	10
^{13}C5-PFPeA	5	5	5	5	5	5	5
^{13}C5-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C4-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C8-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C9-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C6-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C7-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C2-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C2-PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C3-PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C3-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C8-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C2-4:2 FTS	5	5	5	5	5	5	5
^{13}C2-6:2 FTS	5	5	5	5	5	5	5
^{13}C2-8:2 FTS	5	5	5	5	5	5	5
^{13}C8-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSAA	5	5	5	5	5	5	5
D5-NEtFOSAA	5	5	5	5	5	5	5
D7-NMeFOSE	25	25	25	25	25	25	25
D9-NEtFOSE	25	25	25	25	25	25	25
^{13}C3-HFPO-DA	10	10	10	10	10	10	10
^{13}C3-PFBA	5	5	5	5	5	5	5
^{13}C2-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C4-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C5-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{13}C2-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
^{18}O2-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
^{13}C4-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5

TDCA Stock Solution								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (mg)**	**Aliquot (g)**	**Final Volume**	**Final Conc. TDCA Stock Solution (ppb)**
Sigma Alrich	T0557-500MG	Sodium Taurodeoxycholate hydrate	207737-97-1	TDCA	1000000	0.05	50mL	2000000

TDCA Working Solution A							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mg)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. TDCA Working Solution A (ppb)**
TDCA Stock Intermediate	Sodium Taurodeoxycholate hydrate	207737-97-1	TDCA	2000000	1.25	4mL	625000

TDCA Working Solution B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mg)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. TDCA Working Solution B (ppb)**
TDCA Working Solution A	Sodium Taurodeoxycholate hydrate	207737-97-1	TDCA	625000	0.16	5mL	20000

1633 Linear/Branched TDCA Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. 1633 Linear/Branched TDCA Intermediate (ppb)**
Wellington	T-PFOA	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7TG	T-PFOA	500	0.02	2mL	500
Camridge Isotope Laboratories, Inc.	ULM-11036-S	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500	0.02		500
Camridge Isotope Laboratories, Inc.	ULM-11034-S	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500	0.02		500
Camridge Isotope Laboratories, Inc.	ULM-10780-S	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500	0.01		500
Camridge Isotope Laboratories, Inc.	ULM-10779-S	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500	0.01		500
Camridge Isotope Laboratories, Inc.	ULM-10977-S	Perfluorooctanesulfonamide	754-91-6	PFOSA	500	0.02		500
Wellington	ipPFNA0516	Perfluoro-7-methyloctanoic acid	15899-31-7	PF7MOA	500	0.02		500

1633 Linear/Branched TDCA Solution							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. 1633 Linear/Branched TDCA Solution (ppb)**
TDCA Working Solution B	Sodium Taurodeoxycholate hydrate	207737-97-1	TDCA	5000	0.01	2mL	25
1633 Linear/Branched TDCA Intermediate	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500	0.02		5
	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500			5
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500			5
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			5
	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			5
	Perfluoro-7-methyloctanoic acid	15899-31-7	PF7MOA	500			5

PFAS 1633 ICV Working Standard							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ug/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. PFAS 1633 ICV Working Standard (ppb)**
Native PFAS Intermediate A	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	94.500	0.20	2mL	9.450
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	93.500			9.350
	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	94.500			9.450
	Perfluoro(2-propoxypropanoic) acid	13252-13-6	HFPODA	100.000			10.000
	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	93.750			4.690
	1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	95.000			4.755
	1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	96.000			4.800
	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	25.000			2.500
	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	25.000			2.500
	Perfluorobutanesulfonic acid	375-73-5	PFBS	22.175			2.218
	Perfluorobutanoic acid	375-22-4	PFBA	100.000			10.000
	Perfluorodecanesulfonic acid	335-77-3	PFDS	24.125			2.413
	Perfluorodecanoic acid	335-76-2	PFDA	25.000			2.500
	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	24.250			2.425
	Perfluorododecanoic acid	307-55-1	PFDoDA	25.000			2.500
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	23.825			2.383
	Perfluoroheptanoic acid	375-85-9	PFHpA	25.000			2.500
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	22.850			2.285
	Perfluorohexanoic acid	307-24-4	PFHxA	25.000			2.500
	Perfluorononanesulfonic acid	68259-12-1	PFNS	24.050			2.405
	Perfluorononanoic acid	375-95-1	PFNA	25.000			2.500
	Perfluorooctanesulfonamide	754-91-6	PFOSA	25.000			2.500
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	23.200			2.320
	Perfluorooctanoic acid	335-67-1	PFOA	25.000			2.500
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	23.525			2.353
	Perfluoropentanoic acid	2706-90-3	PFPeA	50.000			5.000
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	25.000			2.500
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	25.000			2.500
	Perfluoroundecanoic acid	2058-94-8	PFUnDA	25.000			2.500
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50.000			5.000
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50.000			5.000
	Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50.000			5.000
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44.500			4.450
	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	250.000			25.000
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	25.000			2.500
	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	250.000			25.000
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	25.000			2.500
Native PFAS Intermediate B	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	100.000	0.25		12.500
	3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	500.000			62.500
	3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	500.000			62.500
MPFACHIFES	Perfluoro-n-[^{13}C4]butanoic acid	STL00992	^{13}C4-PFBA	2000	0.01		10.000
	Perfluoro-n-[^{13}C5]pentanoic acid	STL01893	^{13}C5-PFPeA	1000			5.000
	Perfluoro-n-[1,2,3,4,6-^{13}C5]hexanoic acid	STL02577	^{13}C5 -PFHxA	500			2.500
	Perfluoro-n-[1,2,3,4-^{13}C4]heptanoic acid	STL01892	^{13}C4-PFHpA	500			2.500
	Perfluoro-n-[^{13}C8]octanoic acid	STL01052	^{13}C8-PFOA	5050			2.500
	Perfluoro-n-[^{13}C9]nonanoic acid	STL02578	^{13}C9-PFNA	250			1.250
	Perfluoro-n-[1,2,3,4,5,6-^{13}C6]decanoic acid	STL02579	^{13}C6-PFDA	250			1.250
	Perfluoro-n-[1,2,3,4,5,6,7-^{13}C7]undecanoic acid	STL02580	^{13}C7-PFUnA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]dodecanoic acid	STL02703	^{13}C2-PFDoA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]tetradecanoic acid	STL02116	^{13}C2-PFTeDA	250			1.250
	Perfluoro-1-[2,3,4-^{13}C3]butanesulfonic acid	STL02337	^{13}C3-PFBS	466			2.330
	Perfluoro-1-[1,2,3-^{13}C3]hexanesulfonic acid	STL02581	^{13}C3-PFHxS	474			2.370
	Perfluoro-1-[^{13}C8]octanesulfonic acid	STL01054	^{13}C8-PFOS	479			2.395
	Perfluoro-1-[^{13}C8]octanesulfonamide	STL01056	^{13}C8 -PFOSA	500			2.500
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	1000			5.000
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	1000			5.000
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]hexan sulfonic acid	STL02395	^{13}C2-4:2FTS	938			4.690
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]octanesulfonic acid	STL02279	^{13}C2-6:2FTS	951			4.755
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]decanesulfonic acid	STL02280	^{13}C2-8:2FTS	960			4.800
	Tetrafluoro-2-heptafluoropropoxy-^{13}C3-propanoic acid	STL02255	^{13}C3-HFPO-DA	2000			10.000
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	5000			25.000
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	5000			25.000
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	500			5.000
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	500			5.000
MPFACHIFES	Perfluoro-n-[2,3,4-^{13}C3]butanoic acid	STL02680	^{13}C3-PFBA	1000	0.01		5.000
	Perfluoro-n-[1,2,3,4-^{13}C4]octanoic acid	STL00990	^{13}C4-PFOA	500			2.500
	Perfluoro-n-[1,2-^{13}C2]decanoic acid	STL00996	^{13}C2-PFDA	250			1.250
	Perfluoro-n-[1,2,3,4-^{13}C4]octanesulfonic acid	STL00991	^{13}C4-PFOS	479			2.395
	Perfluoro-n-[1,2,3,4,5-^{13}C5] nonanoic acid	STL00995	^{13}C5-PFNA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]hexanoic acid	STL00993	^{13}C2-PFHxA	500			2.500
	Perfluoro-1-hexane[^{18}O2]sulfonic acid	STL00994	^{18}O2-PFHxS	474			2.370

1633 Labeled Ampulated Standards						
Ampulated Solution Name	**Vendor**	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**
MPFACHIFES	Wellington	MPFACHIFES	Perfluoro-n-[^{13}C4]butanoic acid	STL00992	^{13}C4-PFBA	2000
			Perfluoro-n-[^{13}C5]pentanoic acid	STL01893	^{13}C5-PFPeA	1000
			Perfluoro-n-[1,2,3,4,6-^{13}C5]hexanoic acid	STL02577	^{13}C5 -PFHxA	500
			Perfluoro-n-[1,2,3,4-^{13}C4]heptanoic acid	STL01892	^{13}C4-PFHpA	500
			Perfluoro-n-[^{13}C8]octanoic acid	STL01052	^{13}C8-PFOA	5050
			Perfluoro-n-[^{13}C9]nonanoic acid	STL02578	^{13}C9-PFNA	250
			Perfluoro-n-[1,2,3,4,5,6-^{13}C6]decanoic acid	STL02579	^{13}C6-PFDA	250
			Perfluoro-n-[1,2,3,4,5,6,7-^{13}C7]undecanoic acid	STL02580	^{13}C7-PFUnA	250
			Perfluoro-n-[1,2-^{13}C2]dodecanoic acid	STL02703	^{13}C2-PFDoA	250
			Perfluoro-n-[1,2-^{13}C2]tetradecanoic acid	STL02116	^{13}C2-PFTeDA	250
			Perfluoro-1-[2,3,4-^{13}C3]butanesulfonic acid	STL02337	^{13}C3-PFBS	466
			Perfluoro-1-[1,2,3-^{13}C3]hexanesulfonic acid	STL02581	^{13}C3-PFHxS	474
			Perfluoro-1-[^{13}C8]octanesulfonic acid	STL01054	^{13}C8-PFOS	479
			Perfluoro-1-[^{13}C8]octanesulfonamide	STL01056	^{13}C8 -PFOSA	500
			N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	1000
			N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	1000
			1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]hexan sulfonic acid	STL02395	^{13}C2-4:2FTS	938
			1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]octanesulfonic acid	STL02279	^{13}C2-6:2FTS	951
			1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]decanesulfonic acid	STL02280	^{13}C2-8:2FTS	960
			Tetrafluoro-2-heptafluoropropoxy-^{13}C3-propanoic acid	STL02255	^{13}C3-HFPO-DA	2000
			N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	5000
			N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	5000
			N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	500
			N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	500
MPFACHIFES	Wellington	MPFACHIFIS	Perfluoro-n-[2,3,4-^{13}C3]butanoic acid	STL02680	^{13}C3-PFBA	1000
			Perfluoro-n-[1,2,3,4-^{13}C4]octanoic acid	STL00990	^{13}C4-PFOA	500
			Perfluoro-n-[1,2-^{13}C2]decanoic acid	STL00996	^{13}C2-PFDA	250
			Perfluoro-n-[1,2,3,4-^{13}C4]octanesulfonic acid	STL00991	^{13}C4-PFOS	479
			Perfluoro-n-[1,2,3,4,5-^{13}C5] nonanoic acid	STL00995	^{13}C5-PFNA	250
			Perfluoro-n-[1,2-^{13}C2]hexanoic acid	STL00993	^{13}C2-PFHxA	500
			Perfluoro-1-hexane[^{18}O2]sulfonic acid	STL00994	^{18}O2-PFHxS	474

Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

Inj Std	Internal Standard/Injection Standard
I13C3-PFBA	13C3-PFBA
I13C2-PFHxA	13C2-PFHxA
I13C4-PFOA	13C4-PFOA
I13C5-PFNA	13C5-PFNA
I13C2-PFDA	13C2-PFDA
I18O2-PFHxS	18O2-PFHxS
I13C4-PFOS	13C4-PFOS

Extraction Standards

Extraction Standard	Internal Standard
E13C4-PFBA	13C3-PFBA
E13C5-PFPeA	13C2-PFHxA
E13C5-PFHxA	
E13C4-PFHpA	
E13C3-HFPO-DA	
E13C8-PFOA	13C4-PFOA
E13C9-PFNA	13C5-PFNA
E13C6-PFDA	13C2-PFDA
E13C7-PFUnA	
E13C2-PFDoA	
E13C2-PFTeDA	
E13C3-PFBS	18O2-PFHxS
E13C3-PFHxS	
E13C2-4:2-FTS	
E13C2-6:2-FTS	
E13C2-8:2-FTS	

Extraction Standard	Internal Standard
E13C8-PFOS	13C4-PFOS
E13C8-PFOSA	
Ed3-NMeFOSA	
Ed5-NEtFOSA	
Ed3-NMeFOSAA	
Ed7-NMeFOSE	
Ed9-NEtFOSE	

Native PFAS Compounds

Native	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	

Native	Extraction Standard
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

1633 Native Ampulated Standards						
Ampulated Solution Name	**Vendor**	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**
Native Replacement PFAS Solution/Mixture	Wellington	PFAC-MXF	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1890
			9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1870
			4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1890
			Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000
Native PFAS Solution/Mixture	Wellington	PFAC-MXH	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	3840
			1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	3750
			1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	3800
			N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	1000
			N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	1000
			Perfluorobutanesulfonic acid	375-73-5	PFBS	887
			Perfluorobutanoic acid	375-22-4	PFBA	4000
			Perfluorodecanesulfonic acid	335-77-3	PFDS	965
			Perfluorodecanoic acid	335-76-2	PFDA	1000
			Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	970
			Perfluorododecanoic acid	307-55-1	PFDoDA	1000
			Perfluoroheptanesulfonic acid	375-92-8	PFHpS	953
			Perfluoroheptanoic acid	375-85-9	PFHpA	1000
			Perfluorohexanesulfonic acid	355-46-4	PFHxS	914
			Perfluorohexanoic acid	307-24-4	PFHxA	1000
			Perfluorononanesulfonic acid	68259-12-1	PFNS	962
			Perfluorononanoic acid	375-95-1	PFNA	1000
			Perfluorooctanesulfonamide	754-91-6	PFOSA	1000
			Perfluorooctanesulfonic acid	1763-23-1	PFOS	928
			Perfluorooctanoic acid	335-67-1	PFOA	1000
			Perfluoropentanesulfonic acid	2706-91-4	PFPeS	941
			Perfluoropentanoic acid	2706-90-3	PFPeA	2000
			Perfluorotetradecanoic acid	376-06-7	PFTeDA	1000
			Perfluorotridecanoic acid	72629-94-8	PFTrDA	1000
			Perfluoroundecanoic acid	2058-94-8	PFUnDA	1000
Native Perfluoroalkyl Ether Carboxylic Acids and Sulfonate Solution/Mixture	Wellington	PFAC-MXG	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	2000
			Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	2000
			Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	2000
			Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	1780
Native N-NMe/EtFOSA & N-Nme/EtFOSE Solution/Mixture	Wellington	PFAC-MXI	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	10000
			N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	1000
			2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	10000
			N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	1000
Native X:3 Flourotelomer Caroxylic Acid Solution/Mixture	Wellington	PFAC-MXJ	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	4000
			3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	20000
			3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	20000

Attachment 3

Acquisition Method	Mass Spectrometer Method Properties
EPA1633_DOD Mass Spec 10.500 min Period 10.500 min -MRM Integrated Valve Sciex LC System Equilibrate Injection	

Period 1:

Scans in Period: 1050
Min. Dwell Time: 3 ms
Max. Dwell Time: 250 ms
Relative Start Time: 0.00 msec
Scheduled Ionization: Off
Experiments in Period: 1
Use target Cycle Time: No
Target Cycle Time: N/A

Period 1 Experiment 1:

Scan Type: MRM (MRM)
Scheduled MRM: Yes
Polarity: Negative
Scan Mode: N/A
Ion Source: Turbo Spray
sMRM Q1Q3 Resolution: No
MRM detection window: 60 sec
Target Scan Time: 0.6000 sec
Resolution Q1: Unit
Resolution Q3: Unit
Intensity Thres.: 0.00 cps
Settling Time: 0.0000 msec
MR Pause: 5.0070 msec
MCA: No
Step Size: 0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	3.88	DP	-40.00	-40.00	13C3-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
217.000	172.000	3.88	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.000	223.000	4.44	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.000	80.000	4.49	DP	-120.00	-120.00	13C3-PFBS
			CE	-65.00	-65.00	
329.000	81.000	4.83	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	
315.000	270.000	4.86	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	
318.000	273.000	4.86	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	
287.000	169.000	5.00	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	
367.000	322.000	5.27	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	
402.000	80.000	5.27	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	
359.000	294.000	5.42	DP	-40.00	-40.00	13C2-6:2 FTUCA
			CE	-25.00	-25.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
379.000	294.000	5.43	DP	-30.00	-30.00	13C2-6:2 FTCA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.000	81.000	5.63	DP	-100.00	-100.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
415.000	370.000	5.65	DP	-50.00	-50.00	13C2-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.000	172.000	5.65	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.000	376.000	5.65	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
503.000	99.000	5.98	DP	-100.00	-100.00	13C4-PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.000	99.000	5.98	DP	-100.00	-100.00	13C8-PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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472.000	427.000	5.99	DP	-50.00	-50.00	13C9-PFNA	
			CE	-18.00	-18.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
459.000	394.000	6.13	DP	-50.00	-50.00	13C2-8:2 FTUCA	
			CE	-25.00	-25.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
479.000	394.000	6.13	DP	-35.00	-35.00	13C2-8:2 FTCA	
			CE	-25.00	-25.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
519.000	474.000	6.30	DP	-50.00	-50.00	13C6-PFDA	
			CE	-18.00	-18.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
515.000	470.000	6.30	DP	-50.00	-50.00	13C2-PFDA	
			CE	-18.00	-18.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
529.000	81.000	6.31	DP	-100.00	-100.00	13C2-8:2-FTS	
			CE	-42.00	-42.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
506.000	78.000	6.40	DP	-100.00	-100.00	13C8-PFOSA	
			CE	-80.00	-80.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID	
573.000	419.000	6.40	DP	-80.00	-80.00	d3-NMeFOSAA	
			CE	-30.00	-30.00		

Attachment 3

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
565.000	520.000	6.58	DP	-70.00	-70.00	13C2-PFUnDA
			CE	-19.00	-19.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.000	6.58	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.000	419.000	6.50	DP	-90.00	-90.00	d5-NEtFOSAA
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
559.000	494.000	6.70	DP	-60.00	-60.00	13C2-10:2 FTUCA
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
579.000	494.000	6.72	DP	-50.00	-50.00	13C2-10:2 FTCA
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.000	570.000	6.81	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.000	59.000	6.85	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	6.86	DP	-100.00	-100.00	d3-NMePFOSA

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			CE	-37.00	-37.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.000	59.000	7.01	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.000	219.000	7.03	DP	-100.00	-100.00	d5-NEtPFOSA
			CE	-38.00	-38.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.000	670.000	7.21	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
163.000	119.000	1.83	DP	-30.00	-30.00	PPF Acid
			CE	-15.00	-15.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
213.000	169.000	3.89	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
249.000	99.000	4.12	DP	-60.00	-60.00	PFPrS
			CE	-40.00	-40.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	85.000	4.17	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	4.49	DP	-60.00	-60.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	4.43	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
299.000	80.000	4.49	DP	-120.00	-120.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.000	4.62	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.000	135.000	4.71	DP	-60.00	-60.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	4.84	DP	-70.00	-70.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.000	307.000	4.83	DP	-100.00	-100.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	4.86	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.000	80.000	4.89	DP	-90.00	-90.00	PFPeS
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
285.000	169.000	5.00	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.000	319.000	5.27	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
399.000	80.000	5.27	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
377.000	251.000	5.32	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.000	5.40	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
357.000	293.000	5.42	DP	-45.00	-45.00	6:2 FTUCA
			CE	-25.00	-25.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
377.000	293.000	5.44	DP	-45.00	-45.00	6:2 FTCA
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
461.000	381.000	5.63	DP	-70.00	-70.00	PFECHS
			CE	-40.00	-40.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.000	407.000	5.62	DP	-100.00	-100.00	6:2-FTS
			CE	-35.00	-35.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	80.000	5.63	DP	-100.00	-100.00	PFHpS
			CE	-90.00	-90.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	5.65	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
499.000	80.000	5.90	DP	-100.00	-100.00	PFOS
			CE	-100.00	-100.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	5.99	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	317.000	6.13	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
457.000	393.000	6.13	DP	-50.00	-50.00	8:2 FTUCA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
477.000	393.000	6.15	DP	-45.00	-45.00	8:2 FTCA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.000	351.000	6.12	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
549.000	80.000	6.28	DP	-100.00	-100.00	PFNS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
513.000	469.000	6.30	DP	-50.00	-50.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.000	507.000	6.30	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	78.000	6.40	DP	-100.00	-100.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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570.000 419.000 6.40 DP -80.00 -80.00 NMeFOSAA
CE -30.00 -30.00

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	80.000	6.54	DP	-100.00	-100.00	PFDS
			CE	-120.00	-120.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.000	519.000	6.58	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.000	419.000	6.50	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
557.000	493.000	6.70	DP	-70.00	-70.00	10:2 FTUCA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
631.000	451.000	6.68	DP	-100.00	-100.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
577.000	493.000	6.72	DP	-60.00	-60.00	10:2 FTCA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.000	569.000	6.99	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
627.000	607.000	6.84	DP	-100.00	-100.00	10:2-FTS
			CE	-47.00	-47.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.000	59.000	6.85	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.000	219.000	6.86	DP	-100.00	-100.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.000	80.000	6.99	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	59.000	7.01	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.03	DP	-100.00	-100.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	619.000	7.03	DP	-60.00	-60.00	PFTrDA
			CE	-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.000	669.000	7.21	DP	-60.00	-60.00	PFTeDA

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				CE	-22.00	-22.00

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
813.000	769.000	7.51	DP	-100.00	-100.00	PFHxDA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
913.000	869.000	7.74	DP	-100.00	-100.00	PFODA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
299.000	99.000	4.50	DP	-100.00	-100.00	PFBS_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	85.000	4.45	DP	-25.00	-25.00	PFECA B_2
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.000	81.000	4.83	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	119.000	4.86	DP	-50.00	-50.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.000	99.000	4.89	DP	-100.00	-100.00	PFPeS_2
			CE	-50.00	-50.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
285.000	185.000	5.00	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
385.000	185.000	5.00	DP	-75.00	-75.00	HFPODA_3
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.000	169.000	5.27	DP	-60.00	-60.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
399.000	99.000	5.27	DP	-100.00	-100.00	PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	5.40	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
461.000	99.000	5.63	DP	-60.00	-60.00	PFECHS_2
			CE	-60.00	-60.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.000	81.000	5.62	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	99.000	5.63	DP	-100.00	-100.00	PFHpS_2
			CE	-80.00	-80.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	5.65	DP	-60.00	-60.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
499.000	99.000	5.97	DP	-100.00	-100.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	5.99	DP	-60.00	-60.00	PFNA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
549.000	99.000	6.28	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
513.000	219.000	6.30	DP	-50.00	-50.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.000	81.000	6.30	DP	-100.00	-100.00	8:2 FTS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	483.000	6.40	DP	-80.00	-80.00	NMeFOSAA_2
			CE	-24.00	-24.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	99.000	6.54	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.000	269.000	6.58	DP	-80.00	-80.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.000	526.000	6.50	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.000	319.000	6.81	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
627.000	81.000	6.84	DP	-120.00	-120.00	10:2 FTS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	169.000	7.03	DP	-60.00	-60.00	PFTrDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.000	169.000	7.21	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
813.000	169.000	7.51	DP	-80.00	-80.00	PFHxDA_2
			CE	-45.00	-45.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
913.000	169.000	7.74	DP	-80.00	-80.00	PFODA_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
179.000	85.000	2.90	DP	-15.00	-15.00	PFMOAA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	241.000	3.92	DP	-80.00	-80.00	R-PSDA
			CE	-32.00	-32.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
405.000	217.000	3.92	DP	-60.00	-60.00	R-EVE
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
439.000	343.000	3.94	DP	-80.00	-80.00	Hydrolized PSDA
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	185.000	4.06	DP	-20.00	-20.00	PMPA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
297.000	135.000	4.17	DP	-80.00	-80.00	NVHOS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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245.000	85.000	4.37	DP	-10.00	-10.00	PFO2HxA	
			CE	-15.00	-15.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	235.000	4.59	DP	-10.00	-10.00	PEPA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
311.000	85.000	4.97	DP	-20.00	-20.00	PFO3OA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.000	283.000	5.27	DP	-40.00	-40.00	Hydro-EVE Acid
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
397.000	217.000	5.27	DP	-80.00	-80.00	R-PSDCA
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	263.000	5.26	DP	-80.00	-80.00	Hydro-PS Acid
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
379.000	185.000	5.38	DP	-35.00	-35.00	PFECA-G
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
377.000	84.000	5.48	DP	-20.00	-20.00	PFO4DA
			CE	-40.00	-40.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
443.000	147.000	5.53	DP	-70.00	-70.00	PS Acid
			CE	-32.00	-32.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
407.000	263.000	5.55	DP	-40.00	-40.00	EVE Acid
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
443.000	85.000	5.93	DP	-7.00	-7.00	PFO5DA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
175.000	97.000	1.46	DP	-45.00	-45.00	MTP
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	5.99	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	84.000	5.27	DP	-100.00	-100.00	1802-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	69.000	4.43	DP	-40.00	-40.00	PFPeA_2
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	478.000	6.40	DP	-100.00	-100.00	PFOSA_2

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				CE	-80.00	-80.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.000	169.000	6.86	DP	-100.00	-100.00	NMePFOSA_2
				CE	-37.00	-37.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.03	DP	-180.00	-180.00	NEtPFOSA_2
				CE	-40.00	-40.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
377.000	85.000	5.32	DP	-40.00	-40.00	DONA_2
				CE	-20.00	-20.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
533.000	353.000	6.12	DP	-100.00	-100.00	9Cl-PF3ONS_2
				CE	-38.00	-38.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
633.000	453.000	6.68	DP	-180.00	-180.00	11Cl-PF3OUdS_2
				CE	-40.00	-40.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	4.49	DP	-60.00	-60.00	3:3 FTCA_2
				CE	-12.00	-12.00
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	337.000	6.13	DP	-80.00	-80.00	7:3 FTCA_2
				CE	-20.00	-20.00

Attachment 3

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.000	83.000	4.71	DP	-60.00	-60.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.000	99.000	6.99	DP	-100.00	-100.00	PFDoS_2
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.000	4.86	DP	-180.00	-180.00	13C5-PFHxA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.000	99.000	4.49	DP	-120.00	-120.00	13C3-PFBS_2
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.000	99.000	5.27	DP	-100.00	-100.00	13C3-PFHxS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.000	80.000	5.98	DP	-100.00	-100.00	13C8-PFOS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.000	309.000	4.83	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.000	409.000	5.63	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Attachment 3

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.000	509.000	6.31	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
287.000	185.000	5.00	DP	-20.00	-20.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.000	119.000	4.86	DP	-30.00	-30.00	13C2-PFHxA_2
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
503.000	80.000	5.98	DP	-100.00	-100.00	13C4-PFOS_2
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
CAD:	10.00
IS:	-3000.00
TEM:	350.00
GS1:	40.00
GS2:	50.00
EP	-10.00
CXP	-14.00

ST1927333A

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

PFAC-MXC

Native Perfluorinated Compound Solution/Mixture

PRODUCT CODE:	PFAC-MXC
LOT NUMBER:	PFACMXC0617
SOLVENT(S):	Methanol / Water (<1%)
DATE PREPARED: (mm/dd/yyyy)	06/14/2017
LAST TESTED: (mm/dd/yyyy)	03/19/2019
EXPIRY DATE: (mm/dd/yyyy)	03/19/2024
RECOMMENDED STORAGE:	Store ampoule in a cool, dark place

DESCRIPTION:

PFAC-MXC is a solution/mixture of thirteen native perfluoroalkylcarboxylic acids (C_4-C_{14}, C_{16}, and C_{18}) and eight native perfluoroalkylsulfonates (C_4-C_{10} and C_{12}). The full name, abbreviation and concentration for each of the components are given in Table A.

The individual perfluoroalkylcarboxylic acids and perfluoroalkylsulfonates all have chemical purities of >98%.

DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture
Figure 1: LC/MS Data (SIR)
Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compounds it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters $x_1, x_2, \ldots x_n$ on which it depends is:

$$u_c(y(x_1, x_2, \ldots x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of ±5% (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A 1226), and ISO 17034 by ANSI-ASQ National Accreditation Board (ANAB; AR-1523).

CALA Testing Accreditation No. A1226

ANAB ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at www.well-labs.com or contact us directly at info@well-labs.com

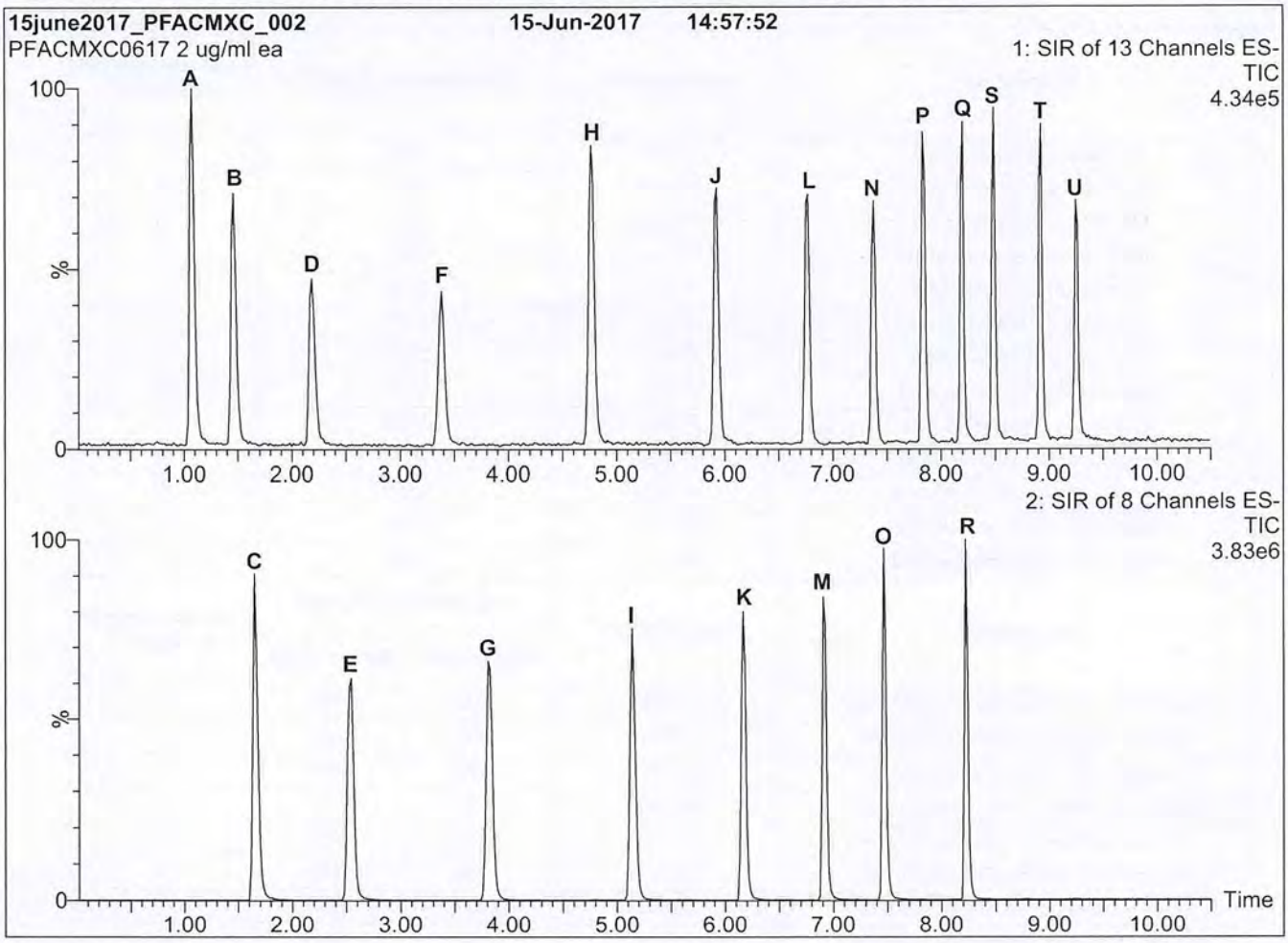
Table A: **PFAC-MXC; Components and Concentrations (ng/ml, ± 5% in Methanol / Water (<1%))**

Compound	Abbreviation	Concentration (ng/ml)*		Peak Assignment in Figure 1
Perfluoro-n-butanoic acid	PFBA	2000		A
Perfluoro-n-pentanoic acid	PFPeA	2000		B
Perfluoro-n-hexanoic acid	PFHxA	2000		D
Perfluoro-n-heptanoic acid	PFHpA	2000		F
Perfluoro-n-octanoic acid	PFOA	2000		H
Perfluoro-n-nonanoic acid	PFNA	2000		J
Perfluoro-n-decanoic acid	PFDA	2000		L
Perfluoro-n-undecanoic acid	PFUdA	2000		N
Perfluoro-n-dodecanoic acid	PFDoA	2000		P
Perfluoro-n-tridecanoic acid	PFTrDA	2000		Q
Perfluoro-n-tetradecanoic acid	PFTeDA	2000		S
Perfluoro-n-hexadecanoic acid	PFHxDA	2000		T
Perfluoro-n-octadecanoic acid	PFODA	2000		U
Compound	**Abbreviation**	**Concentration (ng/ml)***		**Peak Assignment in Figure 1**
		As the salt	**As the anion**	
Potassium perfluoro-1-butanesulfonate	L-PFBS	2000	1770	C
Sodium perfluoro-1-pentanesulfonate	L-PFPeS	2000	1880	E
Sodium perfluoro-1-hexanesulfonate	L-PFHxS	2000	1890	G
Sodium perfluoro-1-heptanesulfonate	L-PFHpS	2000	1900	I
Sodium perfluoro-1-octanesulfonate	L-PFOS	2000	1910	K
Sodium perfluoro-1-nonanesulfonate	L-PFNS	2000	1920	M
Sodium perfluoro-1-decanesulfonate	L-PFDS	2000	1930	O
Sodium perfluoro-1-dodecanesulfonate	L-PFDoS	2000	1940	R

* Concentrations have been rounded to three significant figures.

Certified By: _____ **Date:** 06/06/2019
(mm/dd/yyyy)
B.G. Chittim, General Manager

Figure 1: **PFAC-MXC; LC/MS Data (Total Ion Current Chromatogram; SIR)**



Conditions for Figure 1:

LC: **Waters Acquity Ultra Performance LC**
MS: **Micromass Quattro *micro* API MS**

Chromatographic Conditions

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H_2O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions in 1 min.

Time: 12 min

Flow: 300 µl/min

MS Parameters

Experiment: SIR of 21 Channels

Source: Electrospray (negative)
Capillary Voltage (kV) = 3.00
Cone Voltage (V) = variable (10-80)
Cone Gas Flow (l/hr) = 50
Desolvation Gas Flow (l/hr) = 750

Figure 2: **PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)**

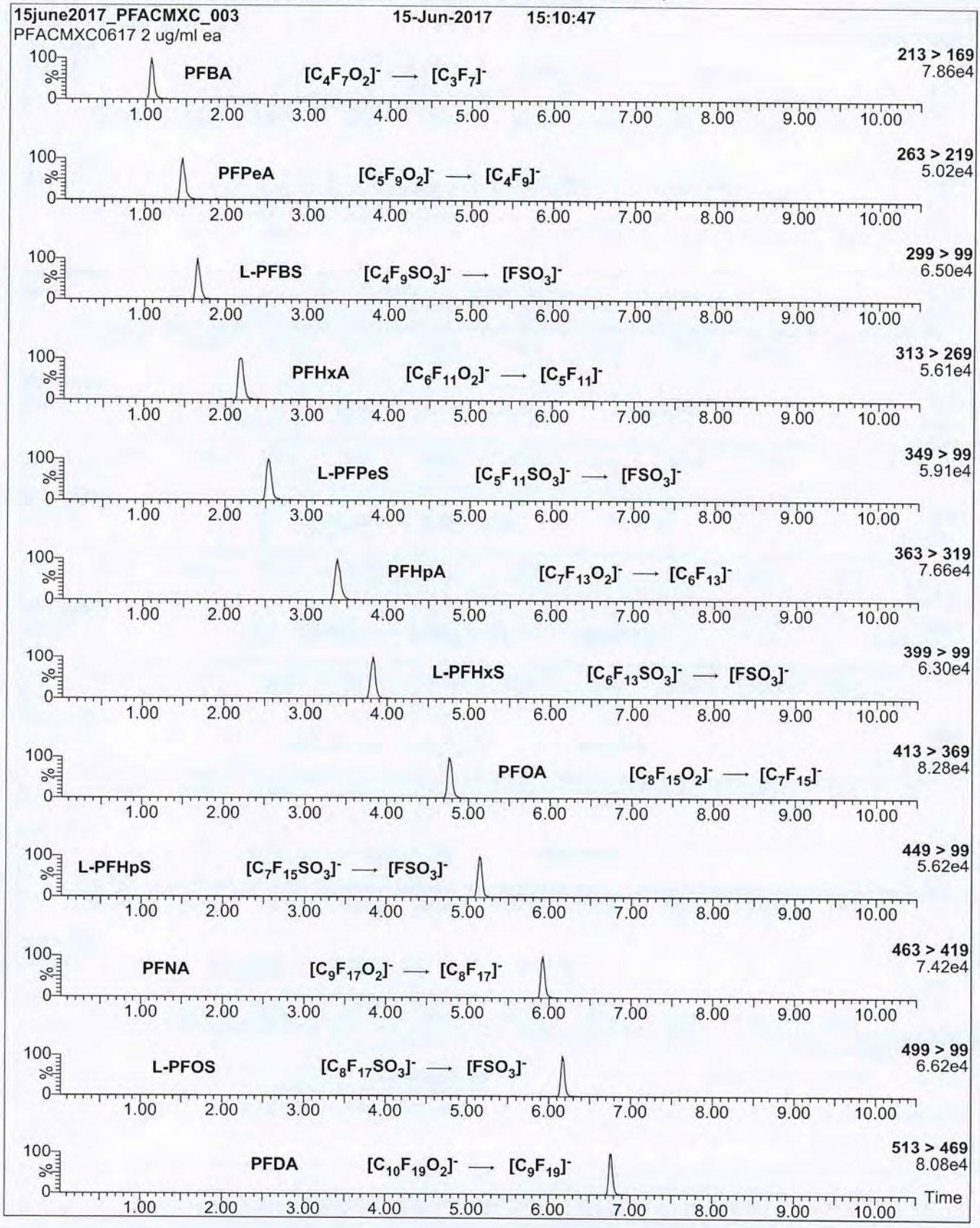
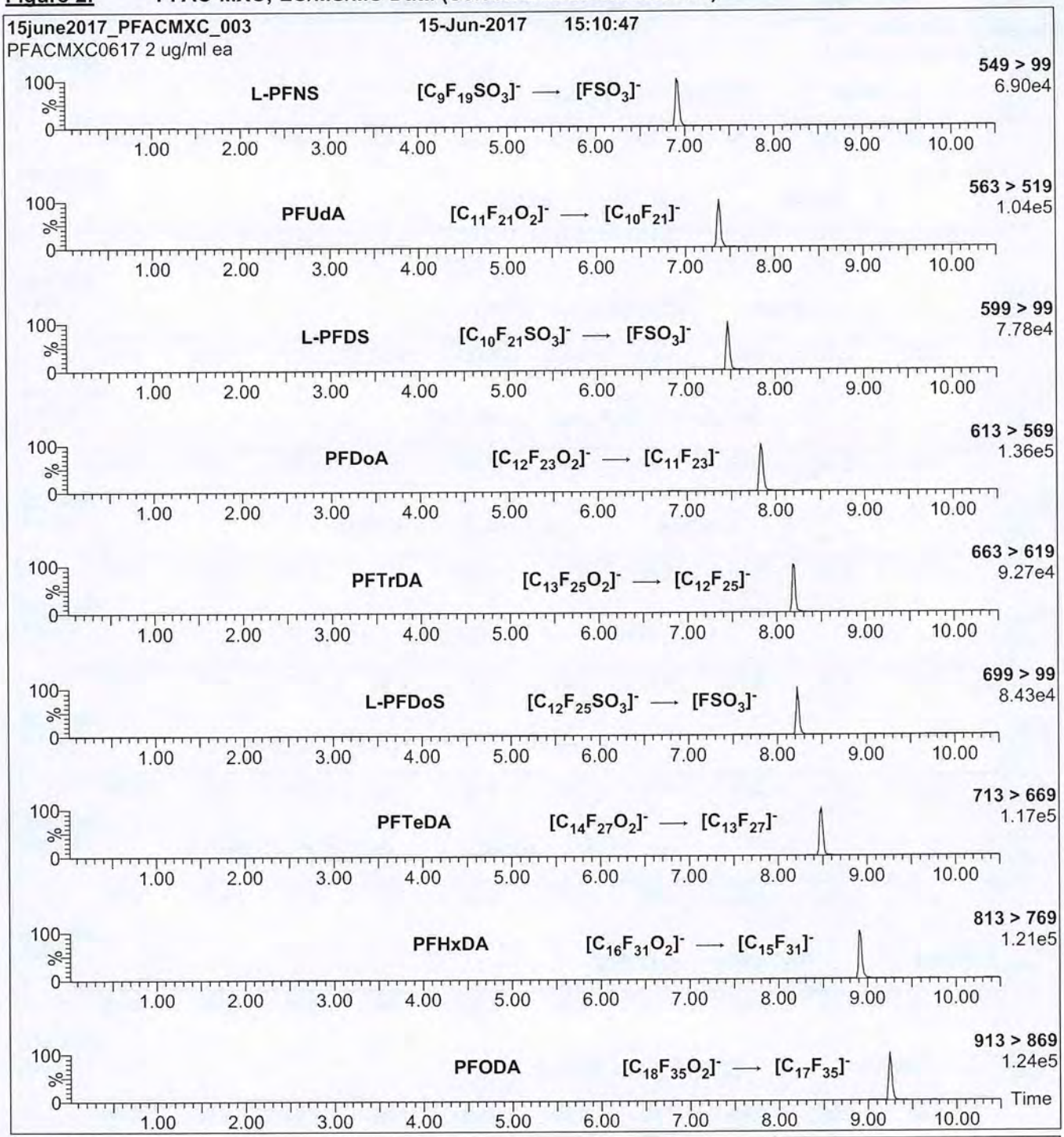


Figure 2: **PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (PFAC-MXC)

Mobile phase: Same as Figure 1

Flow: 300 µl/min

MS Parameters
Collision Gas (mbar) = 3.46e-3
Collision Energy (eV) = 8-50 (variable)

Native PFAS Intermediate A								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Native PFAS Intermediate A (ppb)**
Wellington	PFAC-MXF	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1890	0.10	2mL	94.500
		9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1870			93.500
		4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1890			94.500
		Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000			100.000
Wellington	PFAC-MXH	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	3840	0.05		93.750
		1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	3750			95.000
		1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	3800			96.000
		N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	1000			25.000
		N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	1000			25.000
		Perfluorobutanesulfonic acid	375-73-5	PFBS	887			22.175
		Perfluorobutanoic acid	375-22-4	PFBA	4000			100.000
		Perfluorodecanesulfonic acid	335-77-3	PFDS	965			24.125
		Perfluorodecanoic acid	335-76-2	PFDA	1000			25.000
		Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	970			24.250
		Perfluorododecanoic acid	307-55-1	PFDoDA	1000			25.000
		Perfluoroheptanesulfonic acid	375-92-8	PFHpS	953			23.825
		Perfluoroheptanoic acid	375-85-9	PFHpA	1000			25.000
		Perfluorohexanesulfonic acid	355-46-4	PFHxS	914			22.850
		Perfluorohexanoic acid	307-24-4	PFHxA	1000			25.000
		Perfluorononanesulfonic acid	68259-12-1	PFNS	962			24.050
		Perfluorononanoic acid	375-95-1	PFNA	1000			25.000
		Perfluorooctanesulfonamide	754-91-6	PFOSA	1000			25.000
		Perfluorooctanesulfonic acid	1763-23-1	PFOS	928			23.200
		Perfluorooctanoic acid	335-67-1	PFOA	1000			25.000
		Perfluoropentanesulfonic acid	2706-91-4	PFPeS	941			23.525
		Perfluoropentanoic acid	2706-90-3	PFPeA	2000			50.000
		Perfluorotetradecanoic acid	376-06-7	PFTeDA	1000			25.000
		Perfluorotridecanoic acid	72629-94-8	PFTrDA	1000			25.000
		Perfluoroundecanoic acid	2058-94-8	PFUnDA	1000			25.000
Wellington	PFAC-MXG	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	2000	0.05		50.000
		Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	2000			50.000
		Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	2000			50.000
		Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	1780			44.500
Wellington	PFAC-MXI	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	10000	0.05		250.000
		N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	1000			25.000
		2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	10000			250.000
		N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	1000			25.000

Native PFAS Intermediate B								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ug/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Native PFAS Intermediate B (ppb)**
Wellington	PFAC-MXJ	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	4	0.05	2mL	100.000
		3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	20			500.000
		3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	20			500.000

Working Labeled Extraction Standard Spike							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Working Labeled Extraction Standard Spike (ppb)**
MPFACHIFES	Perfluoro-n-[^{13}C4]butanoic acid	STL00992	^{13}C4-PFBA	2000	0.01	5mL	10.000
	Perfluoro-n-[^{13}C5]pentanoic acid	STL01893	^{13}C5-PFPeA	1000			5.000
	Perfluoro-n-[1,2,3,4,6-^{13}C5]hexanoic acid	STL02577	^{13}C5 -PFHxA	500			2.500
	Perfluoro-n-[1,2,3,4-^{13}C4]heptanoic acid	STL01892	^{13}C4-PFHpA	500			2.500
	Perfluoro-n-[^{13}C8]octanoic acid	STL01052	^{13}C8-PFOA	5050			2.500
	Perfluoro-n-[^{13}C9]nonanoic acid	STL02578	^{13}C9-PFNA	250			1.250
	Perfluoro-n-[1,2,3,4,5,6-^{13}C6]decanoic acid	STL02579	^{13}C6-PFDA	250			1.250
	Perfluoro-n-[1,2,3,4,5,6,7-^{13}C7]undecanoic acid	STL02580	^{13}C7-PFUnA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]dodecanoic acid	STL02703	^{13}C2-PFDoA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]tetradecanoic acid	STL02116	^{13}C2-PFTeDA	250			1.250
	Perfluoro-1-[2,3,4-^{13}C3]butanesulfonic acid	STL02337	^{13}C3-PFBS	466			2.330
	Perfluoro-1-[1,2,3-^{13}C3]hexanesulfonic acid	STL02581	^{13}C3-PFHxS	474			2.370
	Perfluoro-1-[^{13}C8]octanesulfonic acid	STL01054	^{13}C8-PFOS	479			2.395
	Perfluoro-1-[^{13}C8]octanesulfonamide	STL01056	^{13}C8 -PFOSA	500			2.500
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	1000			5.000
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	1000			5.000
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]hexan sulfonic acid	STL02395	^{13}C2-4:2FTS	938			4.690
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]octanesulfonic acid	STL02279	^{13}C2-6:2FTS	951			4.755
	1H,1H,2H,2H-Perfluoro-1-[1,2-^{13}C2]decanesulfonic acid	STL02280	^{13}C2-8:2FTS	960			4.800
	Tetrafluoro-2-heptafluoropropoxy-^{13}C3-propanoic acid	STL02255	^{13}C3-HFPO-DA	2000			10.000
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	5000			25.000
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	5000			25.000
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	500			5.000
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	500			5.000

Working Internal Standard Spike							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Working Internal Standard Spike (ppb)**
MPFACHIFIS	Perfluoro-n-[2,3,4-^{13}C3]butanoic acid	STL02680	^{13}C3-PFBA	1000	0.01	5mL	5.000
	Perfluoro-n-[1,2,3,4-^{13}C4]octanoic acid	STL00990	^{13}C4-PFOA	500			2.500
	Perfluoro-n-[1,2-^{13}C2]decanoic acid	STL00996	^{13}C2-PFDA	250			1.250
	Perfluoro-n-[1,2,3,4-^{13}C4]octanesulfonic acid	STL00991	^{13}C4-PFOS	479			2.395
	Perfluoro-n-[1,2,3,4,5-^{13}C5] nonanoic acid	STL00995	^{13}C5-PFNA	250			1.250
	Perfluoro-n-[1,2-^{13}C2]hexanoic acid	STL00993	^{13}C2-PFHxA	500			2.500
	Perfluoro-1-hexane[^{18}O2]sulfonic acid	STL00994	^{18}O2-PFHxS	474			2.370

Native 1633 Mid-Level Spike								
Solution Name	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume**	**Final Conc. Native 1633 Mid-Level Spike (ppb)**
Wellington	PFAC-MXF	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1890	0.63	5mL	236.250
		9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1870			233.750
		4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1890			236.250
		Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000			250.000
Wellington	PFAC-MXH	1H,1H,2H,2H perfluorotelomersulfonic acid	39108-34-4	4:2-FTS	3840	0.31		480.000
		1H,1H,2H,2H perfluorotelomersulfonic acid	757124-72-4	6:2-FTS	3750			468.750
		1H,1H,2H,2H perfluorotelomersulfonate acid	27619-97-2	8:2-FTS	3800			475.000
		N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	1000			125.000
		N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	1000			125.000
		Perfluorobutanesulfonic acid	375-73-5	PFBS	887			110.875
		Perfluorobutanoic acid	375-22-4	PFBA	4000			500.000
		Perfluorodecanesulfonic acid	335-77-3	PFDS	965			120.625
		Perfluorodecanoic acid	335-76-2	PFDA	1000			125.000
		Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	970			121.250
		Perfluorododecanoic acid	307-55-1	PFDoDA	1000			125.000
		Perfluoroheptanesulfonic acid	375-92-8	PFHpS	953			119.125
		Perfluoroheptanoic acid	375-85-9	PFHpA	1000			125.000
		Perfluorohexanesulfonic acid	355-46-4	PFHxS	914			114.250
		Perfluorohexanoic acid	307-24-4	PFHxA	1000			125.000
		Perfluorononanesulfonic acid	68259-12-1	PFNS	962			120.250
		Perfluorononanoic acid	375-95-1	PFNA	1000			125.000
		Perfluorooctanesulfonamide	754-91-6	PFOSA	1000			125.000
		Perfluorooctanesulfonic acid	1763-23-1	PFOS	928			116.000
		Perfluorooctanoic acid	335-67-1	PFOA	1000			125.000
		Perfluoropentanesulfonic acid	2706-91-4	PFPeS	941			117.625
		Perfluoropentanoic acid	2706-90-3	PFPeA	2000			250.000
		Perfluorotetradecanoic acid	376-06-7	PFTeDA	1000			125.000
		Perfluorotridecanoic acid	72629-94-8	PFTrDA	1000			125.000
		Perfluoroundecanoic acid	2058-94-8	PFUnDA	1000			125.000
Wellington	PFAC-MXG	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	2000	0.31		250.000
		Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	2000			250.000
		Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	2000			250.000
		Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	1780			222.500
Wellington	PFAC-MXI	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	10000	0.39		1250.000
		N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	1000			125.000
		2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	10000			1250.000
		N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	1000			125.000
Wellington	PFAC-MXJ	3-Perfluoropropylpropanoic acid	763051-92-9	3:3 FTCA	4000	0.03		312.500
		3-Perfluoropentylpropanoic acid	756426-58-1	5:3 FTCA	20000			1562.500
		3-Perfluoroheptylpropanoic acid	919005-14-4	7:3 FTCA	20000			1562.500

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Document number: **T-PFAS-WI23588**	**Preventative and Corrective Maintenance for the API 4000 and AB Sciex 4500, 5500, 5500+ Liquid Chromatograph Mass Spectrometers (LC/MS/MS)**	**Work Instruction**
Old Reference:		
Version: **3**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date: **21-FEB-2022**	Document users: **5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Management_Team**	Responsible: **5_EUUSLA_PFAS_Manager**

Revision Log

Revision: 03		**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Procedure	Enhancement	Updated table in A to include current requirements.

Revision: 02		**Effective date:** 26-FEB-2021
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Enhancement	Added Manual for 5500/5500+
Purpose	Enhancement	Added new instruments
Procedure	Enhancement	Added new instruments

Reference

1. *AB Sciex 4500 Series of Instruments System User Guide*, AB Sciex, AB Sciex Pte. Ltd., 2013.

2. 5500 Series of Instruments, AB Sciex, AB Sciex Pte. Ltd., 2017.

3. *Turbo V Ion Source Operator Guide*, AB Sciex, AB Sciex Pte. Ltd., 2015.

4. *Instrument Front-End Cleaning Procedure For Customers*, AB Sciex, AB Sciex Pte. Ltd., 2013.

5. *Hardware Manual: API 4000 LC/MS/MS System*, AB Sciex, AB Sciex Pte. Ltd., 2010.

6. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Purpose

Preventative and corrective maintenance are crucial in maintaining the overall performance of the API 4000, AB Sciex Triple Quad 4500, 5500, 5500+ LC/MS/MS. This SOP explains the basic principles of managing the integrity of the API 4000, AB Sciex Triple Quad 4500, 5500, 5500+ LC-MS/MS.

Scope

This document serves as a guideline for the performance of preventative and corrective maintenance on the API 4000 and AB Sciex Triple Quad 4500, 5500, 5500+ LC-MS/MS.

Routine preventative maintenance is required to maintain an LC/MS/MS system that will perform to method specifications. The performance of preventative maintenance is at the discretion and determination of the chemist. The course of maintenance is based upon experience and meeting method performance specifications. Corrective maintenance may be performed by the chemist, at the chemist's discretion, based on the observed non-conformance. If, however, it is determined that an outside contractor must be contacted to perform certain types of preventative or corrective maintenance on a system, the chemist should consult with his/her supervisor. The supervisor will then assist in determining who will contact the outside contractor to schedule a service call.

Personnel Training and Qualifications

Personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of the SOP. Each new chemist will train with an experienced chemist for the first 12 weeks depending on the individual and his or her previous experience.

During the training period, the new chemist will learn daily preventative maintenance and corrective maintenance procedures, column and source cleaning procedures. S/he is also required to read all relevant SOPs and EPA methods.

Safety Precautions and Waste Handling

Safety glasses must be worn and all necessary safety precautions taken when performing any type of preventative or corrective maintenance.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal and State laws and regulations.

Procedure

All corrective maintenance procedures, whether performed internally or by outside contractor, must be fully documented in the instrument maintenance log. Documentation must include the issue leading to the maintenance, the actions taken, and the condition of the instrument after maintenance

A. MS Maintenance Procedures

Mass Spectrometer (MS)

API 4000* and AB Sciex Triple Quad 4500, 5500, 5500+ LC-MS/MS Preventative Maintenance Schedule (MS Components)

Procedure	Frequency
Clean QJet*	Bi-annually
Clean Q0	Bi-annually
Mass Calibration	After cleaning QJet/0
Clean Electrode	Bi-Monthly
Clean Oriface Plate	Bi-Monthly(when venting)
Clean Curtain Plate	Bi-Monthly
Replace Rough Pump Oil	As needed (yearly)
Divert Valve	Monthly
Injection Valve	Monthly

*The API 4000 does not have a QJet. This system has a Skimmer, which is cleaned bi-annually.

1. 1. Clean Electrode

    a. Deactivate the hardware profile.
    b. Allow the Turbo V Ion source to cool for 15-20 minutes (just until it is comfortable to the touch).
    c. Remove the top fitting and separate from the peek tubing.
    d. Remove the black cap.
    e. Put the top fitting back in place and create a tight seal.
    f. Carefully unscrew the fitting and pull the electrode straight out, careful not to touch the sides of the source housing.
    g. Wipe the electrode with methanol and allow to dry.
    h. Carefully position the electrode back into the source housing.
    i. Screw the black cap on.
    j. Connect the peek tubing from the Analytical Column to the Ion Source.
    k. Connect the peek tubing that leads to the electrode.
    l. Reactivate the hardware profile.

2. Clean the Curtain Plate

    a. Deactivate the hardware profile.
    b. Allow the Turbo V Ion source to cool for 15-20 minutes (just until it is comfortable to the touch).
    c. Remove the Turbo V Ion source from the MS by unlocking the two latches on both sides and pulling straight off. (Before pulling off, disconnect the peek tubing that connects the Analytical Column to the Ion Source).
    d. Ensure that the O-rings on the back of the Ion source do not fall off.
    e. Remove the curtain plate by pulling straight off and place on a Kimwipe.
    f. Clean both sides with Milli-Q water.
    g. Clean both sides with Methanol.

h. Use a disposable spatula to mix 1 scoop of Aluminum Oxide with Milli-Q H2O in a medicine cup. Mix together and rub onto the curtain plate to remove further discoloration.
i. Rinse with Milli-Q and Methanol again.
j. Allow the curtain plate to air dry.

Note: Maintenance beyond the curtain plate will require fully venting the MS. Prior to maintenance past the curtain plate, a performance baseline should be established using negative PPG as outlined in the Mass Calibration Attachment 1; another check should be performed after the maintenance.

3. Vent the MS

a. Deactivate the hardware profile in Analyst.
b. Vent the MS by holding down the Vent button on the side of the MS (hold down the vent button until you see all of the lights flashing on the side panel).
c. Allow the MS to vent naturally (wait at least 20 minutes).
d. Turn off the MS power supply.
e. Turn off the rough pump and wait another 15-20 minutes.

4. Clean the Orifice Plate

a. Vent the MS using the above procedure.
b. Disconnect the peek tubing leading from the analytical column to the Turbo V Ion Source.
c. Remove the Turbo V Ion Source.
d. Remove the Curtain Plate.
e. Remove the Orifice Plate.
f. Clean the Orifice Plate with Milli-Q water.
g. Clean the Orifice Plate with Methanol.
h. If there are deposits or discoloration remaining, use a disposable spatula to mix 1 scoop of aluminum oxide with Milli-Q water in a medicine cup. Mix together and rub onto the curtain plate to remove any remaining discoloration.
i. Rinse with Milli-Q water and methanol again.
j. Allow the Orifice Plate to air dry.

5. Clean QJet

a. Vent the MS and remove the Orifice Plate following the above procedures. Then remove the QJet ion guide and place on a Kimwipe.
b. Remove the Q0 lens from the bottom of the QJet.
c. Place the QJet in a large beaker, and rinse thoroughly with Milli-Q water followed by Methanol.
d. Use a cotton swab to clean in between the poles of the QJet.
e. Rinse again, and allow QJet to completely air dry.
f. Clean the Q0 lens by wiping with Methanol.
g. Return the lens to the QJet.

6. Clean Q0

a. Vent the MS and remove the Orifice Plate and QJet following the above procedures.
b. Prepare the Q0 cleaning tool (this is a long plastic rod with two eyelets in the end).
c. Fold a Kimwipe in-half enough times so that it can be inserted into the top eyelet.
d. Insert the Kimwipe so that equal amounts are protruding on either end of the cleaning tool and dampen both ends with methanol.
e. Gently insert the cleaning tool ¾ of the way into the Q0, rotate two full clockwise motions, and then remove the tool from the Q0.
f. Remove the used Kimwipe and prepare another to be inserted into the bottom eyelet.

g. Repeat procedure for the bottom eyelet.
h. After cleaning, verify that no pieces of Kimwipe have been left behind in Q0. This can be done by a visual inspection of the used Kimwipes and by a visual check inside of Q0 with the help of a flashlight.

B. LC Maintenance Procedures

1. Mobile Phases

Mobile Phase solutions will need to be replaced and or topped off as needed. The current mobile phases used are 20 mM Ammonium Acetate in Milli-Q water and 20 mM Ammonium Acetate in 0.5% Water/Methanol.

Should the mobile phases run dry while the system is acquiring data, perform the following steps:

a. In Analyst, click Abort Sample and Stop Queue.
b. Replace and or top off the mobile phase that ran dry.
c. Purge the pumps on the Exion LC Autosampler (make sure the pump light is OFF and the drain is OPEN prior to selecting the purge function).
d. Let the pumps purge and check for air in the lines.
e. If needed, top off the IPA vials for both LC pumps.
f. Once the pumps have finished purging, close the drains and turn on the pumps.
g. Set the appropriate files to re-acquire in the Analyst queue and click Start Sample.

NOTE: It is important to also keep in mind the needle wash will need to be replaced as needed. The current needle wash used is 100% Acetonitrile. Should the needle wash become dry, there will be noticeable shifts in retention times.

2. ExionLC AC Column Oven

The column oven houses the Analytical Column. In the process of troubleshooting high pressure, contamination, and or peak tailing, an analyst may need to replace the Analytical Column. The current Analytical Column is a Gemini 3µM C18 LC Column (Phenomenex).

To replace the Analytical Column, perform the following steps:

a. Deactivate the hardware profile in Analyst.
b. Open the column oven.
c. Remove the top fitting from the Analytical Column. (This fitting connects the Sample Transfer Line to the Analytical Column.)
d. Hold the Analytical Column while removing the bottom fitting. (This fitting connects the Analytical Column to the Ion Source.)
e. Place the old Analytical Column off to the side (away from the Column Oven) and replace with a new column by carefully screwing the top and bottom fittings.
f. Once the new column is in place, check for leaks by pumping methanol through the system at 0.5 to 1 mL/min. (Note: Prior to pumping methanol through the system, make sure the divert valve is set to position “A” for waste. If the divert valve is set to position “B,” disconnect the peak tubing leading from the Analytical Column to the Ion Source. It is preferable to disconnect the peak tubing at the Ion Source end. For the API 4000 LC\MS\MS System that is not equipped with a divert valve, disconnecting the peak tubing as previously described is the only option to ensure methanol does not get into the Ion Source.)

1. To manually set a flow rate, tap the button on the pump to the left of Sleep. (It looks like an orange square made up of nine dots.)
2. With the key pad lit up in orange, select Function.
3. Click on Function until System appears – Press Enter.
4. Under normal operating conditions, the value to the right hand side will show "0" – change this number to "1" and press Enter. (At this point, the remote button will turn OFF on the pump.)
5. Press CE twice to navigate quickly to the start screen.
6. Press Function once and type in the desired flow rate.
7. Select Enter to save the flow rate.
8. Select Pump to begin pumping the mobile phase through the system.

g. If leaks are present, tighten fittings where appropriate.
h. Set the pump back to remote mode.

1. Tap the button on the pump to the left side of Sleep. (It looks like an orange square made up of nine dots.)
2. With the key pad lit up in orange, select Function.
3. Click on Function until System appears – Press Enter.
4. Type "1" in place of the "0" and press Enter. (At this point, the remote button will turn ON.)
5. Press CE twice to navigate quickly to the start screen.

i. Reactivate the hardware profile and run several solvent blanks followed by CAL standards to check the peak shapes and retention times.
j. If troubleshooting a pressure/contamination issue, additional areas of the LC\MS\MS may need to be investigated.

3. Pre-Column Replacement

Under normal operation, mobile phases are passed through a pre-column prior to sample introduction. The current pre-column is the Luna 5µm C18 LC Column (Phenomenex). Over time, the pre-column can become clogged. This is indicated by an elevated pressure.

To remove the pre-column, perform the following steps:

a. Deactivate the hardware profile. (At a minimum, turn OFF both pumps to ensure the mobile phases do not leak.)
b. With a large wrench wrapped around the center to hold the pre-column in place, unscrew the top and bottom fittings from the pre-column. (Use a smaller wrench to gently twist the fittings off.)
c. Place the old pre-column off to the side and connect a new pre-column.
d. Once the new pre-column is connected, check for leaks by flushing the lines with methanol (step by step instructions in the above section "Exion AC Column Oven").
e. If leaks are present, tighten fittings where appropriate.
f. Set the pump back to remote mode.
g. Reactivate the hardware profile (if deactivated).
h. If pressure issue is not resolved, investigate additional areas of the LC/MS/MS system.

4. Exion AC Pumps

The AC Pumps are critical components in the operation of an LC\MS\MS. If there are injection issues, there are a few areas worth investigating:

a. Plunger Seals

1. Each pump is equipped with two plunger seals, one on the left and another on the right.
2. To remove a plunger seal, deactivate the hardware profile in Analyst (or at a minimum, turn OFF the pumps).
3. Gently loosen the connection to the area housing the drain with a small wrench.
4. Use a small wrench to disconnect the screw from the top of the CV-OUT. (This is a check valve with a single mark.)
5. Use a key wrench to remove the two screws from the pump head.
6. Pull the pump head straight out, careful not to snap the peek tubing connected to the CV-IN. (This is a check valve with a double or triple mark.)
7. Remove the plunger seal using the rough side of the seal remover tool.
8. Inspect the plunger seal for any abnormalities.
9. Clean the pump head with methanol.
10. Place a new plunger seal on the pump head by using the smooth side of the seal remover tool. (The plunger seal should pop into place.)
11. Perform additional maintenance on the pump head if needed before moving to the second pump head (details below).

b. PTFE Diaphragms

1. With the pump head removed, unscrew the unit holding the plunger and the diaphragm.
2. Inspect the diaphragm for any abnormalities and replace if needed. (Make sure the larger protrusion is facing towards the back.)

c. Plunger – replace as needed
d. In-Out Check Valves – replace as needed

Once all the necessary maintenance has been completed on both pump heads, check for leaks.

5. Exion AC Autosampler

The following components require maintenance:

a. Needle – replace as needed
b. Rotor seals - replace as needed
c. Sample transfer line – replace as needed (will most likely replace in the course of troubleshooting, details below)

1. Deactivate the hardware profile
2. Tap the button on the Autosampler to the left side of Sleep (it looks like an orange square made up of nine dots)
3. Press the up arrow (at this point, the following command should appear "ZHOME Enter to Start")
4. Press Enter and wait for the needle to move into the home position
5. Remove the autosampler tray
6. Take off the safety shield by removing the white screws on the corners
7. Gently slide the needle housing to the left side of the autosampler (Note: Please pay careful attention to the latch at the bottom of the autosampler tray. This latch should not be tampered with as to avoid the needle from returning to the injection port when it is not ready for use.)
8. Use a small wrench to disconnect the transfer line from the injection port (the transfer line is marked with a black stripe)

a. Disconnect the fitting from the head of the Analytical Column

b. Weave the transfer line from the Column Oven towards the Autosampler and remove

9. Set the old transfer line off to the side and assemble a new transfer line to put in place. (A ferrule will need to be added to the tip of the transfer line and tightened appropriately to get a good fit in the injection port – pay careful attention not to over tighten the fitting.)
10. Connect the other end of the transfer line to the head of the Analytical Column.
11. Check for leaks by flushing the system with methanol.
12. Once it has been determined there are no leaks, attach the safety shield and close the autosampler door. (At this point, the needle housing should start moving around.)
13. Press Enter to return the needle to the injection port.
14. Reactivate the hardware profile.

6. Peek Tubing
   Replace the peek tubing as necessary. If the system is showing an elevated pressure, it would be helpful to cut a small portion off of the tubing connected to the Ion Source.

C. Mass Calibration - This is required after cleaning of the QJet and Q0 or annually. See attachment 1 for the instructions.

Attachment:
Attachment 1 - Mass Calibration Instructions (.pdf)

End of document

Version history

Version	Approval	Revision information	
1	10.DEC.2018		
2	12.FEB.2021		
3	07.FEB.2022		

Performing a mass calibration using negative polypropylene glycol (PPG) in negative electrospray ionization mode

Purpose:
A step-by-step instruction on how to perform a mass calibration on Q1 and Q3 for SCIEX 4500 triple-quadrupole MS systems (Analyst versions 1.6.3 and 1.7)

When should a mass calibration be performed?

- At designated intervals as indicated by SOPs or department policies
- Before and after extensive cleaning or maintenance of the mass spectrometer to establish baseline instrument performance (should be done before any maintenance beyond the curtain plate – instrument resolution does not need to be adjusted before maintenance)

Table of contents:

I. What is required:

- 1 mL infusion syringe and syringe Luer-lock adapter, or equivalent

- Negative polypropylene glycol (concentration of 3x10e-5 M recommended)

II. Hardware setup:

1) After disabling the active hardware profile, position the lines on the MS so that one line will reach the syringe pump from the grounding unit, and the opposite line connects the grounding unit to the source (ALWAYS leave tubing to the source element connected to the grounding unit)

2) Rinse 1 mL syringe with methanol and pull up ~1/2 mL of negative PPG, removing all air bubbles (to remove air bubbles, pull a small air pocket into the syringe and point the tip of the syringe downward, allowing the air pocket to rise to the plunger – the air pocket will collect smaller air bubbles as it moves; once the pocket is at the plunger, invert the syringe and let the air pocket rise to the needle, then expel it)

3) Connect the needle of the syringe to a Luer-lock adapter and connect the MS line to the adapter

4) Lower the syringe pump plate (pressing the button on the right-hand side of the plate allows manual adjustment), place the syringe in the syringe pump, and bring the plate up to the syringe plunger

III. Software setup:

1) Enable MS only hardware profile, then double click on “Manual Tuning” under “Tune and Calibrate”

2) Select the project folder named “API Instrument” from the dropdown list and open the “Q1 Neg PPGs” acquisition method (or other appropriate method)

3) Under “Source/Gas”, ensure that the current instrument settings for curtain gas, source gases, voltage and source temperature are entered

4) Under “MS Method”, scan type should be “Q1 MS (Q1)”, scan rate should be 10 Da/s, polarity should be negative, and MCA (multiple channel acquisition) should be checked with 1 scan to sum

5) Under “Syringe pump method”, select the proper syringe size and start the syringe pump; allow ~30 seconds for the flow to stabilize

6) Start acquiring data; “Start” will create a temporary data file while “Acquire” will create a permanent data file – the flow rate may need to be adjusted to optimize peak intensity if peaks are too broad

7) Once a scan has completed, right click on the mass spectrum and select “Open File” to review the different mass range spectra; right click one of these and select “List Data”, then select the “Calibration Peak List” tab to see the peak width and mass shift of each target mass (peak width should be between 0.6 and 0.8, and mass shift should be within +/- 0.1) – only masses 44.998 through 933.6360 need to be within spec as that covers our full range of target masses for PFAS analysis
   *****Before performing extensive MS maintenance, print the window displaying the mass spectra and peak list before performing the maintenance to establish a performance baseline**

8) When finished with Q1, repeat the process for Q3

IV. Adjusting the instrument’s resolution settings

1) If the mass spectra are not resolving into peaks with proper width or mass shift values, the instrument’s resolution will need to be adjusted. Peak width must be between 0.6 and 0.8 and mass shift must be within +/- 0.1

2) To modify instrument resolution, go to Tools> Settings> Instrument Options. **Take screenshots of the existing instrument settings before making changes**

3) Ensure that you have the correct polarity and quadrupole selected and begin making adjustments to the Offset under the Resolution Table. Increasing the offset (higher positive value or lower negative value) will decrease sensitivity, while decreasing the offset (lower positive value or higher negative value) will increase sensitivity – as a change to any of the

resolution settings will affect multiple mass ranges, change only one parameter at a time and in increments of no more than 0.005 at a time

4) Once a resolution parameter has been changed, run a new scan to evaluate how performance was affected and if further parameter changes are required

5) Once all applicable mass ranges are in-spec, acquire a permanent data file and use print window to generate a report of the mass calibration (save to Dept 37 drive under PFAS Mass Calibrations) – the TIC, 4 applicable mass range spectra and the calibration peak list should be displayed in the window

6) When finished with Q1, repeat the process for Q3

V. Troubleshooting:

- If the total ion signal is not stable, for instance it drops off and comes back or remains low, there may be air in the lines or at tubing connection points
    - With the syringe pump running, loosen and re-tighten Luer-lock fittings to expel trapped air bubbles – make sure tubing is secure at connection points
    - Check the connection between the syringe needle and Luer-lock adapter
- Changing the resolution offset for one mass range may affect the signal at another mass range – adjust offsets one at a time and in small increments
- If there is one mass range that is being especially troublesome, consider reverting the settings back to what they were before any adjustments were made and first get that mass range in-spec. Then make small adjustments to any other mass ranges that are out-of-spec while keeping the first range in-spec

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ATTACHMENT 2

FIELD STANDARD OPERATING PROCEDURES AND FIELD FORMS

HGL:	SOP 411.01 PFAS Sampling
HGL:	SOP No. 300.04 Field Logbook Use and Maintenance
HGL:	SOP No. 406.02 Monitoring Well Installation
HGL:	SOP No. 406.01 Well Development
HGL:	SOP No. 402.01 Low-Flow (Minimal Drawdown) Groundwater Sampling Procedures
HGL:	SOP No. 403.02 Hand-Operated Auger Soil Sampling
HGL:	SOP No. 403.06 Surface and Shallow Depth Soil Sampling
HGL:	SOP No. 403.08 Sediment Sampling
HGL:	SOP No. 404.01 Surface Water Sampling
HGL:	SOP No. 412.501 Data Validation
HGL:	SOP No. 411.02 Sampling Equipment Cleaning and Decontamination
PARSONS:	SOP ENV-Deer Sampling

ATTACHMENT 2

FIELD SOP DISCLAIMER

Prior to conducting any PFAS sampling, the field crew will refer to SOP 411.01 (PFAS Sampling) for all requirements and prohibited materials. This PFAS sampling SOP and the UFP-QAPP project-specific worksheets supersede the requirements of all other HGL sampling SOPs.

PFAS-Free Definition. All materials related to PFAS sampling, including sample bottles, will be certified PFAS-free by the provider or tested prior to use to document lack of PFAS compounds. The term PFAS-free water is defined here as water that does not contain significant concentrations of any compound in a specific PFAS analyte list that is being analyzed at a project-defined level. The contracted laboratory will provide PFAS-free water defined as less than (<) the method detection limit (MDL) for the target compound analyzed. Site or public water supplies have been identified in many instances to contain detectable levels of PFAS. The project team will determine the acceptability of an on-site source of water for decontamination and well development based on site-specific parameters such as drilling method and sample media. The onsite water source will be defined as PFAS-free if it meets the DoD QSM Table B-24 method blank requirement: "No analytes detected > ½ LOQ or > 1/10th the amount measured in any associated sample or 1/10th the regulatory limit, whichever is greater." The confirmation of PFAS-free water should always be performed prior to the commencement of work. If the potable water source is determined to be unacceptable an alternate source of water with acceptable PFAS levels will be utilized.

Addition to SOP 411.01 (PFAS Sampling), Table 1 – A DEET version of the insect repellent OFF also has been determined to be PFAS-free.

Note: SOPs are reviewed at least every 2 years by HGL and the review date in the SOP is the date the SOP was last reviewed. If no changes were made to the SOP, the revised date was not changed. HGL is in the process of updating all our SOPs to a new format that will address the reviewed and revised dates and this UFP-QAPP will be updated once those SOPs are complete.

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the general requirements, methods, and equipment used to sample for per- and polyfluoroalkyl substances (PFAS). Although the following sections focus on groundwater sampling, the procedures are applicable to other media, such as drinking water, surface water, sediment, and soil. The contents of this SOP are intended to supplement HGL's matrix-specific sampling SOPs by addressing modifications necessary to generate acceptable PFAS data.

PFAS are a class of compounds that are present at low concentrations in many materials and product coatings. PFAS are ubiquitous and have been used to manufacture items for personal use and environmental site investigations. Much of the typical sampling equipment and items used in field activities contain or may contain PFAS (for example, coated Tyvek materials and waterproof logbooks). Standard environmental sampling practices can cross-contaminate samples and lead to analytical results that could be artifacts of the sampling and analysis system caused by the presence of PFAS in materials associated with sample collection at the sampling site, materials used by the sampler, or sample container handling practices. Consequently, additional measures are required to ensure that sample results are representative of PFAS concentrations at the site.

The techniques described in this procedure are in general agreement with the procedures outlined in the following documents:

- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS) (Interstate Technology Regulatory Council [ITRC], 2018).
- PFASs Sampling Fact Sheet, Revision 1.2 (Environmental Data Quality Workgroup [EDQW], 2017).
- Navy Drinking Water Sampling Policy for Perfluorochemicals Perfluorooctane Sulfonate and Perfluorooctonoic Acid (U.S. Navy, 2015).
- ~~PerFluorinated Compound (PFC) Sample Collection Guidance, (New Hampshire Department of Environmental Services [NHDES], 2016).~~
- Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) (Government of Western Australia Department of Environmental Regulation, 2016).

The EDQW's primary mission is to develop and recommend Department of Defense (DoD) policy pertaining to environmental sampling, laboratory testing operations, and data quality, and its fact

sheet should be the primary document consulted when conducting PFAS field sampling for DoD clients. Although the ITRC guidance carries substantial credibility within federal and state agencies, other clients, such as the U.S. Environmental Protection Agency (EPA), may have specific requirements for PFAS sampling, and it is critical that all client and regulatory requirements be understood prior to conducting PFAS sampling for a project.

~~The U.S. Army Corps of Engineers (USACE), Omaha District has issued a supplement to its Chemistry Scope of Services (SOS) that addresses requirements for sampling, analysis, and validation of PFAS for USACE, Omaha District projects (USACE, Omaha District, 2019). HGL must comply with this SOS supplement on all HGL projects performed for the USACE, Omaha District. Note that the specific requirements of this SOS supplement are not universally accepted, and although this document can be consulted for guidance, clear direction must be obtained before incorporating any elements of this guidance into projects performed for other clients. The USACE, Omaha District PFAS SOS supplement is included as Attachment A to this SOP.~~

2.0 SCOPE AND APPLICATION

This SOP applies to all projects that have PFAS sampling as a component. The requirements of this SOP should be followed even when sampling locations at a site that do not include PFAS analysis if sampling materials could potentially cross-contaminate PFAS samples collected on the same day at other site locations. The requirements for sampling for PFAS contained in this SOP represent the current state of knowledge, but the user should be aware that PFAS represent an emerging class of contaminants and that federal, state, and DoD standards and guidance are still being developed. Before each PFAS sampling event, the sampling team must verify that the planned sampling procedures are compliant with all applicable client and regulatory requirements and policies. Use of out-of-date guidance, even on a project where the guidance was previously acceptable, can lead to issues with acceptance of results.

3.0 EQUIPMENT AND SUPPLIES

Many commonly used sampling materials and field supplies contain PFAS and related compounds. Special care must be taken to ensure that all materials used to collect and store samples are free of PFAS to prevent false positive results or high biases caused by cross-contamination. Table 1 shows the items prohibited from use on PFAS sampling sites. This table should be included in all PFAS sample planning documents; however, the contents must be modified on a project-specific basis to ensure that all materials used on the site are acceptable to the client and the regulatory bodies. For example, NHDES allows the use of Sharpie® markers, but EDQW prohibits the use of any markers and requires pens only. Table 1 is based on the recommendations of NHDES, but additional limitations from the EDQW guidance have been incorporated to make the table more applicable to the wide range of clients supported by HGL. Note that the USACE, Omaha District SOS supplement (Attachment A) and the Western Australia guidance prohibit the use of aluminum foil; however, the NHDES and EDQW guidance do not prohibit the use of aluminum foil.

Table 1
Prohibited and Allowed Items for PFAS Sites

Category	Prohibited Items	Allowed Items
Pumps and tubing	Teflon® and other fluoropolymer-containing materials	High-density polyethylene (HDPE) or silicone tubing; peristaltic pump or stainless steel submersible pump (without fluoropolymer-containing components)
Decontamination	Decon 90	Alconox® or Liquinox®, distilled water[1] followed by PFAS-free laboratory reagent water[1] rinse
Sample storage and preservation	LDPE or glass bottles; LDPE Hydrasleeves; PTFE-or Teflon®-lined caps; chemical ice packs; polypropylene bottles[2]; polyethylene bags	HDPE bottles with unlined plastic caps; HDPE Hydrasleeves; regular ice
Field documentation	Waterproof/treated paper or field books; plastic clipboards, binders, or spiral notebooks; markers; Post-it® notes and other adhesive paper products	Plain paper, metal clipboards, pens
Clothing	Clothing or boots made of or with Gore-Tex® or other synthetic water resistant and/or stain resistant materials; Tyvek® material	Cotton material, previously laundered clothing (preferably previously washed more than six times) without the use of fabric softeners; boots made of PVC or polyurethane; polyurethane or wax-coated rain gear
Personal care products (day of sampling)	Cosmetics, moisturizers, hand cream, and other related products	Suncreens: Alba Organics Natural Yes to Cucumbers Aubrey Organics Jason Natural Sun Block Kiss My Face Baby-safe sunscreens ('free' or 'natural') Insect Repellents: Jason Natural Quit Bugging Me Repel Lemon Eucalyptus Herbal Armor California Baby Natural Bug Spray BabyGanics Sunscreen and Insect Repellents: Avon Skin So Soft Bug Guard-SPF 30
Food and beverage	Pre-packaged food, fast food wrappers or containers	Bottled water or hydration drinks

[1] PFAS-free laboratory reagent grade water should be obtained in glass or HDPE containers with unlined HDPE caps.

[2] Polypropylene bottles are allowed by Method 537.1 and can be used for drinking water samples if the target analytes only include perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) and if the laboratory is following Method 537.1 unmodified. However; laboratories frequently make modifications to Method 537.1 for environmental sample analysis, and additional target PFASs are often included as requested analytes for which the adherence issue is less well-understood. As a result, adherence of target analytes to polypropylene bottles may not be sufficiently controlled, and these bottles should not be used for environmental sampling unless required by the regulatory program.

4.0 PROCEDURES

4.1 PROJECT PLANNING

During project planning, it is essential to eliminate potential sources of PFAS cross-contamination and to ensure that project sampling procedures meet the requirements of the client and regulatory agency. Due to the lack of a standardized approach to PFAS sampling within the environmental field, the following measures should be added to the normal project planning process to ensure that client, regulatory, site, and programmatic requirements will be met.

- *Specify the sample preservation requirements.* Method 537 indicates that all PFAS samples be preserved with Trizma®, which is a premixed blend of tris(hydroxymethyl)aminomethane (Tris) and tris(hydroxymethyl)aminomethane hydrochloride (Tris HCL). This preservative acts as a buffer and removes free chlorine in chlorinated finished waters (e.g., potable water). Some laboratory SOPs require the use of Trizma® only when sampling drinking water or other chlorine-treated waters and not for environmental samples, and this may conflict with project-specific requirements to preserve all samples. Method 533, another EPA drinking water method used for shorter-chain PFAS, requires that all samples be preserved with ammonium acetate. EPA regional laboratories may have their own specific requirements. Ensure that the laboratory and project staff understand the project-specific preservation requirements and will provide pre-preserved sample bottles as required.
- *Specify the frequency of ambient blank collection.* Methods 533 and 537 require the collection of a field reagent blank (FRB), which is the equivalent to what is commonly termed an ambient blank in environmental sampling. In routine environmental sampling, ambient blanks are either not required or are only collected at a specified frequency, with additional ambient blanks collected if the field team leader suspects a local source. For some PFAS sampling projects, a specified frequency will be acceptable; however, some programs require that an ambient blank be collected at each sample location.
- *Present the laboratory-specific analytical quality control (QC) requirements.* The analytical method most commonly cited for PFAS analysis is Version 1.1 of EPA Method 537. This method was originally intended to support drinking water sampling under the Safe Drinking Water Act, and the requirements of that method are specific to supporting drinking water sampling from taps. In several instances, the terminology used in Method 537 does not correspond with terms commonly used in the environmental field. Laboratories have made modifications to this method to support types of environmental samples, and the laboratory-specific modifications may conflict with what is specified in the method. Project Quality Assurance Project Plans (QAPPs) should default to the laboratory SOP unless there is a project-specific requirement to use Method 537 as written. Note that DoD has developed method requirements for matrices other than drinking water, which are presented in Appendix B of the *Quality Systems Manual (QSM)*

for Environmental Laboratories, Version 5.3 (2019). The QSM requirements should take precedence for DoD projects.

- *Evaluate any site sampling systems already in place.* Single-use, disposable polyethylene or silicone materials, such as bailers and peristaltic pumps, are preferred for monitoring well purging and sampling. In some cases, a site may have dedicated non-peristaltic pumps already installed in all or part of the monitoring well system. When using positive displacement/submersible pump sampling equipment, the sampling pump/accessory equipment specifications must be examined thoroughly to confirm that device components are neither made of nor contain Teflon® or PTFE. If necessary, test the pump and accessories by collecting a sample of PFAS-free water using the same model of pump.
- *Identify decontamination requirements and verification procedures.* In cases where reuse of materials or sampling equipment across multiple sampling locations is necessary, follow decontamination protocols with allowed materials identified in Section 3.0, and incorporate collection of equipment rinse blanks into the sampling program, as appropriate.

All project-specific modifications required to sample for PFAS should be documented in the project planning documents, including the Work Plan, Field Sampling Plan, and QAPP. In some cases, field quality assurance (QA) split samples will be collected by a third party or will be collected by HGL at a site where a third party is the primary sampler. To ensure that data comparability is maintained, these split sampling efforts should coordinate sample collection methods and material requirements that will apply to all parties. This consensus must be obtained in advance and be documented in the project planning documents.

4.2 MOBILIZATION

During mobilization, project supplies are assembled and transferred to the field. Mobilization includes performing the following:

- Order only allowed items (see Section 3.0) for use in site support and sampling.
- Inspect all supplies coming on site to ensure that no prohibited items are brought to the site or are used in sampling.
- Review the requirements of the project planning documents with the field team, emphasizing any differences from routine sampling required for sampling for PFAS, including sampling procedures (see Section 4.3); personal clothing and cosmetics; personal protective equipment, sunscreens, and insect repellents; support items such as notebooks, pens, and clipboards; and collection of PFAS-specific ambient blanks.

4.3 SAMPLE COLLECTION AND SHIPMENT

The following practices will help reduce the potential for cross-contamination from the sampling process and between wells.

- Disposable nitrile gloves should be worn at all times during site activities. A new pair of nitrile gloves should be donned prior to conducting the following activities at each sampling location:
  - Decontaminating reusable sampling equipment,
  - Coming into contact with sample bottles or decontamination water containers,
  - Inserting anything into a well,
  - Inserting silicone tubing into a peristaltic pump,
  - Completing well purging,
  - Handling QC samples including ambient blanks and equipment blanks, and
  - When judged necessary by field personnel.
- Don a new pair of nitrile gloves after handling any nondedicated sampling equipment or coming into contact with nondecontaminated surfaces.
- Collect the sample for PFAS first, prior to collecting samples for any other parameters into any other containers; this avoids contact with any other type of sample container, bottles or package materials.
- Do not place the sample bottle cap on any surface when collecting the sample, and avoid all contact with the inside of the sample bottle or its cap. It is acceptable to temporarily place the cap, thread side up, on a clean nitrile glove so that the sampler has a free hand to ensure proper sample collection. However, it is preferable for a second member of the field team to hold the cap until the container is ready for capping.
- When a sample is collected and capped, place the sample bottle(s) in an individual sealed plastic bag (e.g., Ziploc®) separate from all other sample parameter bottles, and place it in shipping container packed only with ice.
- Ensure that any site visitors (such as client representatives, regulatory observers, or split sample collectors) adhere to all protocols and honor all prohibitions identified in this SOP. If site visitors are not prepared (e.g., are wearing improper clothing or personal care products), keep them well down-wind from the sample collection area.

5.0 RECORDS

All project information required in HGL SOP 4.07: *Field Logbook Use and Maintenance* must be entered on blank sheets of loose-leaf nontreated paper or forms preprinted on nontreated paper; all documentation must be made using only the types of pens allowed by the project. All project information compiled on loose-leaf paper and forms should be scanned to a PDF file as soon as possible, preferably daily, to ensure that all project field records are captured in a permanent

format. The originals or scanned files should undergo QC in accordance with the requirements of Section 4.4 of HGL SOP 4.07.

6.0 QUALITY CONTROL

The project manager and field team leader are responsible for implementing the requirements of this SOP, including any modifications required to meet project-specific requirements. The QA officer is responsible for reviewing all planning documents, periodically reviewing field documentation, and investigating deviations from plans.

7.0 REFERENCES

Environmental Data Quality Workgroup, 2017. PFASs Sampling Factsheet, version 1.2. July. Internet location: https://www.denix.osd.mil/edqw/home/what-s-new/unassigned/edqw-pfas-sampling-factsheet-rev-1-2-july-2017/.

Government of Western Australia Department of Environmental Regulation. 2016. Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoralkyl Substances (PFAS), Contaminated Sites Guidelines. February 2016. Internet location: https://www.der.wa.gov.au/images/documents/your-environment/contaminated-sites/guidelines/Guideline-on-Assessment-and-Management-of-PFAS-.pdf.

Interstate Technology Regulatory Council, 2018. Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS). Internet location: https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_site_characterization_3_15_18.pdf.

New Hampshire Department of Environmental Services, 2016. PerFluorinated Compound (PFC) Sample Collection Guidance. November. Internet location: https://www.des.nh.gov/organization/commissioner/documents/pfas-sample-guidance-201611.pdf.

U.S Army Corps of Engineers-Omaha District, 2019. Chemistry Scope of Services – PFAS. August 29.

U.S. Environmental Protection Agency, 2009. Method 537: Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS), Version 1.1. EPA/600/R-08/092. September. Internet location:

https://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=198984&simpleSearch=1&searchAll=EPA%2F600%2FR-08%2F092+.

U.S. Environmental Protection Agency, 2019. Method 533: Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry, November. Internet location: https://www.epa.gov/sites/production/files/2019-12/documents/method-533-815b19020.pdf.

U.S. Navy, 2015. Navy Drinking Water Sampling Policy for Perfluorochemicals Perfluorooctane Sulfonate and Perfluorooctonoic Acid. September. Internet location: https://www.navfac.navy.mil/content/dam/navfac/Specialty%20Centers/Engineering%20and%20Expeditionary%20Warfare%20Center/Environmental/Restoration/er_pdfs/d/OPNAV-policy-drinkingwater-PFOS-PFOA-20150914.pdf.

ATTACHMENT A

PFAS SUPPLEMENT TO THE USACE-OMAHA DISTRICT CHEMISTRY SCOPE OF SERVICES

Disclaimer: This attachment is not applicable to the Task Order covered by this UFP-QAPP.

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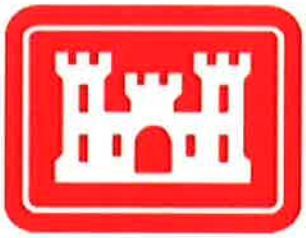
UNITED STATES ARMY CORPS OF ENGINEERS

OMAHA DISTRICT (CENWO)

PFAS CHEMISTRY INSTRUCTIONS FOR SCOPES OF SERVICES

FOR CONTRACTED ENVIRONMENTAL STUDIES



August
2019

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LIST OF ACRONYMS

AGC	Advanced Geophysical Classification
ANSI	American National Standards Institute
ASQ	American Society for Quality
CENWO	United States Army Corps of Engineers Northwestern Division Omaha District
CERCLA	Comprehensive, Environmental Response, Compensation, and Liability Act
COPC	Contaminant of Potential Concern
CSM	Conceptual Site Model
DERP	Defense Environmental Restoration Program
DL	Detection Limit
DoD	Department of Defense
DoDI	Department of Defense Instruction
DOE	Department of Energy
DQCR	Daily Quality Control Report
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
ERPIMS	Environmental Resources Program Information Management Systems
FUDS	Formerly Used Defense Sites
g	gram
IDQTF	Intergovernmental Data Quality Task Force
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
LOD	Limit of Detection
LOQ	Limit of Quantitation
m	meter
mL	milliliter
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols
MFR	Memorandum For Record
NCP	National Contingency Plan
NELAP	National Environmental Laboratory Accreditation Program
OSD	Office of Secretary of Defense
PDT	Project Delivery Team
PFAS	per- and polyfluoroalkyl substances
ppt	parts per trillion
PT	Proficiency Testing (sample)
QA	Quality Assurance
QC	Quality Control
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
SEDD	Staged Electronic Data Deliverable

SOP	Standard Operating Procedure
SOS	Scope of Services
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
U.S.	United States
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency

1 PFAS/PCF Chemistry Supplement

The scope covers all requirements for acceptable sampling, analysis and validation of perfluoroalkyl and polyfluoroalkyl substances (PFAS) for Omaha District contract actions.

2 Background

PFAS, including perfluorooctanesulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) comprise of either a short or a long carbon chain. The carbon chain is lipophilic while the head of the molecule is hydrophilic. The stability of these compounds is due to the strength of the carbon-fluorine bonds. Understanding the analytical implications of factors such as adsorption of PFASs to surfaces, effects of differing matrices, varying PFAS isomer response factors, potential bias effects of sampling, sample preparation, and analysis is critical to measuring highly fluorinated compounds at trace levels. PFAS can be transported to surface waters and groundwater (as a result of runoff and leaching) and are persistent in the environment. As a result, they can be transported long distances from the source site. Requirements for sampling various media (groundwater, soils, surface water and sediments) are discussed below. Also discussed are the analytical and validation requirements.

3 Sample Collection Protocol

All sample ports will be purged, as necessary, prior to sample collection. All purge water shall be collected and either treated or disposed of in accordance with all applicable local, state, federal, and USACE regulations. All samples shall be collected in appropriate containers for the requested analysis. Once collected, the samples will be properly preserved, packaged, placed on wet ice, and shipped under proper chain-of-custody (COC) procedures. The COC forms will be completed by the sampler and will accompany the samples from the field to the lab.

PFAS are present in a wide variety of commercial products including common household items (fabric softeners, sunscreens, low density polyethylene containers, Gore-Tex, cosmetics, moisturizing lotions, etc.). Given the low detection limits associated with PFAS analysis and the many potential sources of trace levels of PFAS, field personnel will strictly adhere to the sampling equipment and protocols summarized in the table below.

Table 1: Summary of Prohibited and Acceptable Items for Sampling of PFAS

Prohibited Items	Acceptable Items
Field Equipment	
Teflon® containing materials	High-density polyethylene (HDPE) materials
Low density polyethylene (LDPE)	Polyvinyl chloride (PVC) or acetate liners
Aluminum foil	Silicon tubing
Waterproof field books	Loose paper (non-waterproof)
Plastic clipboards, binders, or spiral hard cover notebooks	Aluminum field clipboards or with Masonite
	Sharpies®, pens
Post-It Notes	
Field Clothing and Personal Protective Equipment (PPE)	
New cotton clothing or synthetic water resistant, waterproof, or stain- treated clothing, clothing containing Gore-Tex™	Well-laundered clothing, defined as clothing that has been washed 6 or more times after purchase, made of natural fibers (preferably cotton)
Clothing laundered using fabric softener	No fabric softener
Boots containing Gore-Tex™	Boots made with polyurethane and polyvinyl chloride (PVC)
Tyvek®	Cotton Clothing
No cosmetics, moisturizers, hand cream, or other related products as part of personal cleaning/showering routine on the morning of sampling	Sunscreens - Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, Baby sunscreens that are "free" or "natural" Insect Repellents - Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellant, Herbal Armor, California Baby Natural Bug Spray, BabyGanics Sunscreen and insect repellant - Avon Skin So Soft Bug Guard Plus – SPF 30 Lotion
Sample Containers	
LDPE or glass containers	HDPE or polypropylene
Teflon®-lined caps	Unlined polypropylene caps
Rain Events	
Waterproof or resistant rain gear	Gazebo tent that is only touched or moved prior to and following sampling activities
Equipment Decontamination	
Decon 90	Alconox® and/or Liquinox®
Water from an on-site well	PFAS-free water from a tested source
Food Considerations	
All food and drink, with exceptions noted on the right	Bottled water and hydration drinks (i.e., Gatorade® and Powerade®) to be brought and consumed only in the staging area

USACE Omaha District has adopted with minimal modifications sample handling and processing methods as presented in Appendix A of Government of Western Australia, Department of Environmental Regulation, 2016, Interim Guideline on the Assessment and Management of Perfluoroalkyl Substances (PFAS), Contaminated Sites Guidelines, February. The following is a summary:

4 Sampling Methodology

Prior to sampling, the sampling personnel must don a clean, new pair of disposable nitrile gloves. A new pair of nitrile gloves must be worn for each sample collected.

Teflon®-coated materials and aluminum foil may not come into contact with the sample (refer to **Table 1**). Sample handling equipment or tools made of HDPE or stainless steel are acceptable, provided they are decontaminated prior to use via scrubbing and rinsing thoroughly in PFAS-free water to clean away any debris or material and then triple-rinsed in distilled (Grade 3 or better) or deionized water (Millipore water).

Sample containers must be comprised of polypropylene or HDPE (refer to **Table 1**). Glass containers with lined lids are prohibited. Prior to sampling, confirm sample container composition (polypropylene versus HDPE) with the selected analytical laboratory.

For each sample, the required minimum volume of drinking water, surface water, or groundwater is 125 milliliters (mL), and the required minimum amount of soil or sediment is at least 2 grams on a dry weight basis. If quantitation limits lower than 4 parts per trillion (ppt) are needed to meet data quality objectives, the required minimum volume of drinking water, surface water, or groundwater is 250mL. These sampling requirements may vary by laboratory. Prior to sampling, confirm sample size requirements with the selected analytical laboratory. Sampling volume is determined by the analytical laboratory and should be adapted to expected PFAS levels and analytical capacities. The instrumental limit of detection is the main factor limiting the sensitivity and the volume should be enough to reach quantitation levels.

For chlorinated drinking water, each sample bottle may be required to contain a small amount (5g per liter) of Trizma®, a buffering reagent that removes free chlorine from chlorinated drinking water, or similar sample additive as specified by the selected analytical laboratory. Confirm the need for additive with the selected analytical laboratory and the USACE chemist.

The use of chemical or gel-based coolant products (e.g., BlueIce®) to maintain samples at less than 6°C following sample collection is prohibited. The acceptable alternative is wet ice which has been double-bagged (polyethylene plastic) and secured to avoid meltwater from contacting sample containers during overnight or same-day delivery to the analytical laboratory.

Table 1 should be reviewed to identify other products that may contaminate the sampling processing area. If in doubt about a particular product or item in contact with environmental media to be sampled or in close proximity to operations, collect and analyze a rinsate sample using laboratory-supplied PFAS-free water.

Support personnel that are within 2 to 3 meters (m) of the processing area are considered subject to the same restrictions related to precautionary measures for clothing and food, as applied to sampling personnel.

During sample processing and storage, minimize the exposure of the sample to light. Once collected, the samples will be properly preserved, packaged, placed on ice, and shipped under proper COC procedures. The COC forms will be completed by the sampler and will accompany the samples from the field to the lab.

5 Soil Drilling and Surface Water and Sediment Sampling

Decontamination of soil drilling and sampling equipment and of sediment sampling equipment (cores, grabs) must avoid the use of detergents other than those listed in **Table 1**. Equipment must be scrubbed with a plastic brush or steam cleaned and rinsed thoroughly in PFAS-free water to clean away any debris of material on exposed surfaces and then triple rinsed in distilled (Grade 3 or better) or deionized water (or Millipore water). Equipment that contacts soil, sediment, or surface water must not contain or be coated with Teflon® unless the Teflon® is internal to the equipment and does not contact the external environment.

Prior to sample collection, any personnel that handles decontaminated soil, sediment, or surface water sampling equipment that directly contacts the environmental media to be sampled must don a clean, new pair of disposable nitrile gloves. A new pair of nitrile gloves must be worn for each different sampling location. Donning a new pair of gloves is necessary if the old pair of gloves was compromised or if the personnel's ungloved hands touched items that may represent potential PFAS contamination (refer to **Table 1**) since last being washed.

Surface water must be collected by inserting a capped sampling container (polypropylene or HDPE) with the opening pointing down to avoid the collection of surface films. At the time of container opening, the container must be more than 10 centimeters (cm) from the sediment bed and more than 10cm below the surface water level and as close to the center of the channel as possible, where practicable. Point the container up to fill so that gloved hands, sample container, and sampler are downstream of where sample is being collected.

Soil and sediment core samples must be collected directly from single-use PVC or acetate liners that must not be decontaminated or reused at different locations.

For aquatic samples collected from shore or via wading, ensure that waders are constructed of fabric that has not been treated with waterproofing coatings (refer to **Table 1**). **Table 1** should be reviewed to identify other products that may contaminate the sampling area or surface water, sediment, or soil sample. If in doubt about a particular product or item in contact with environmental media to be sampled or in close proximity to operations, collect and analyze a rinsate sample using laboratory-supplied PFAS-free water. Support personnel that handle any part of equipment that directly contacts surface water or aquatic sediment, personnel that are within 2 to 3m of the borehole during soil sampling, or personnel that are within 2 to 3m of the collection and processing area on aquatic vessels during sediment or surface water sampling, are considered subject to the same restrictions related to precautionary measures for clothing and food, as applied to sampling personnel

In saltwater conditions, other measurements should also be collected: conductivity, salinity and TSS. Filtration upon sample collection is not recommended since the filter may absorb PFAS or be a source of contamination.

6 Groundwater Well Drilling, Development, and Sampling

Decontamination of drilling equipment must avoid the use of detergents. All equipment must be scrubbed with a plastic brush or steam cleaned and rinsed thoroughly in PFAS-free water to clean away any debris or material on exposed surfaces and then triple-rinsed in distilled (Grade 3 or better) or deionized water (or Millipore water). Sampling must include submission of sample(s) representing any water collected at the point of use (i.e., water truck or tank on-site) used by the driller for drilling purposes.

Equipment that contacts well water within the well (pumping equipment, water meters, etc.) must not contain or be coated with Teflon® unless the Teflon® is internal to the equipment and does not contact the external environment.

Prior to well development, any personnel that handles decontaminated well development equipment that directly contacts bore water must don a clean, new pair of disposable nitrile gloves. A new pair of nitrile gloves must be worn for each different well developed. Hand washing prior to donning the new pair of gloves is necessary if the old pair of gloves was compromised or if the personnel's ungloved hands touched items that may represent potential PFAS contamination (refer to **Table 1**) since last being washed.

Equipment recommended for obtaining groundwater samples includes low-flow peristaltic pumps using silicone or HDPE tubing or polypropylene HydraSleeves (or similar products). Sampling equipment must not be decontaminated and/or reused at different locations. If the depth to groundwater prevents the use of peristaltic pumps, then bladder pumps may be considered; however, bladders and other internal parts (i.e., check balls, o-rings, and compression fittings) must not be made of Teflon. Bladders must be changed between sample locations and it is recommended that o-rings also be changed between sample locations.

Table 1 should be reviewed to identify other products that may contaminate the well during drilling and development or obtaining the groundwater sample. If in doubt about a particular product or item in contact with environmental media to be sampled or in close proximity to operations, collect and analyze a rinsate sample using laboratory-supplied PFAS-free water.

7 Analytical Requirements

An accredited laboratory shall be contracted and shall ensure that the selected detection and reporting limits are sufficient to meet the project-established limits. Quality control (QC) samples should also be included: field duplicates (1/10 samples), matrix spikes/matrix spike duplicates (1/20), equipment blanks, and laboratory quality measures (per method). Results and evaluation of the QC program compared to the Project QAPP specifications shall be provided in a Quality Control Summary Report.

The laboratory to be used by the Contractor shall be DoD Environmental Laboratory Accreditation Program (ELAP) accredited or equivalent. The Contractor shall ensure that the selected laboratory meets all state and federal requirements. The Contractor shall select a laboratory that complies with the requirements of their current accreditation of the DoD Quality Systems Manual (QSM), currently at version 5.3. DoD ELAP accredited laboratories for PFAS analysis may be found at: https://www.denix.osd.mil/edqw/accreditation/accreditedlabs

Analysis for all matrices (i.e., drinking water, groundwater, surface water, soil, and sediment) shall be performed by an ELAP accredited laboratory using a liquid chromatography tandem mass spectrometry (LC/MS/MS) method that is on the laboratory's ELAP scope of accreditation and is compliant with the requirements in the DoD QSM for Environmental Laboratories, Table B-15. All PFAS analytes in **Table 2** must be reported. Additional PFAS may be added if determined to be site-specific constituents of concern (e.g., HFPO-DA, ADONA, F-53B major and minor). All compounds to be reported should be on the laboratory's ELAP scope of accreditation.

It should be noted that PFAS analysis is improving and method revisions, new methods, or new state requirements are likely to come into existence in the near future. In all cases, the laboratory must be ELAP accredited, have the method and reported analytes on the laboratory's ELAP scope of accreditation, and be in compliance with the version of the DoD QSM to which the laboratory is accredited.

Table 2: PFAS Analyte List

Chemical	CASRN	Acronym
4:2 Fluorotelomer sulfonate	75124-72-4	4:2 FTS
6:2 Fluorotelomer sulfonate	27619-97-2	6:2 FTS
8:2 Fluorotelomer sulfonate	39108-34-4	8:2 FTS
N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA
N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA
Perfluorobutanesulfonic acid	375-73-5	PFBS
Perfluorobutanoic acid	375-22-4	PFBA
Perfluorodecanesulfonic acid	335-77-3	PFDS
Perfluorodecanoic acid	83-89-6	PFDA
Perfluorododecanoic acid	307-55-1	PFDoA
Perfluoroheptanoic acid	374-85-9	PFHpA
Perfluoroheptanesulfonic acid	375-92-8	PFHpS
Perfluorohexanesulfonic acid	355-46-4	PFHxS
Perfluorohexanoic acid	307-24-4	PFHxA
Perfluorononanoic acid	375-95-1	PFNA
Perfluorononanesulfonic acid	68259-12-1	PFNS
Perfluorooctanesulfonamide	754-91-6	PFOSA
Perfluorooctanesulfonic acid	1763-23-1	PFOS
Perfluorooctanoic acid	335-67-1	PFOA
Perfluoropentanoic acid	2706-90-3	PFPA

Chemical	CASRN	Acronym
Perfluoropentanesulfonic acid	2706-91-4	PFPS
Perfluorotetradecanoic acid	376-06-7	PFTeDA
Perfluorotridecanoic acid	72629-94-68	PFTriDA
Perfluoroundecanoic acid	2058-94-8	PFUnA

8 PFAS-specific Laboratory Analysis Specifications

During communication with the selected analytical laboratory prior to sampling or during pre- project communications with candidate analytical laboratories, it is recommended to confirm the following:

The laboratory uses polypropylene or HPDE sample containers with polypropylene lids, and if there is a preference for either sample container type.

Sample results will represent the sum of the linear and branched isomers for each PFAS. Many PFAS (e.g., PFOS) have several isomeric forms that may show up as separate or partially-merged peaks in the analytical chromatograms. It must be confirmed that these peaks will be integrated and the areas summed such that the result represents the concentration of the sum of the linear and branched isomers. Laboratories must also note in their analytical reports the type of analytical standards used (linear and/or branched) and the approach used in quantitation.

Reagent or ultra-pure water used in the laboratory will be confirmed to be free of PFAS above the detection limit during the analyses and that this water can be provided in HPDE containers with polypropylene lids for use at the site for conducting equipment rinsate sampling (as needed).

9 Data Validation

The current version of the QSM can be found at the following website: http://www.navylabs.navy.mil/. The Contractor shall be responsible for assessing the environmental data's quality by performing data validation against criteria established within the analytical method and the DoD QSM and the project specific UFP QAPP. Validation shall be performed to a 90% Stage 2b standard and a 10% Stage 4 standard with recalculation of appropriate data, including DOD QSM Appendix B table requirements. Documentation is evaluated from sampling logs, sample shipment records, the sample's condition upon receipt at the lab and through the analytical process, as well as the various method quality control samples and instrument parameters.

10 Reporting

Support for specific programs may also mandate the submission of chemical and/or sampling data in electronic formats for archival/retrieval within an agency-specific database systems. Analytical data generated from the laboratory shall be submitted as Microsoft® excel, staged electronic data deliverable (SEDD) file, and/or an Environmental Resources Program Information Management System (ERPIMS) file, depending on the

requirements of the project and full electronic PDF Level IV data packages. For example, AFCEC requires ERPIMS; IMCOM requires DOEHRS, and Formerly Used Defense Sites (FUDS) requires FUDSChem format submissions.

11 Investigation Derived Waste

Waste containing PFAS is not classified as a characteristic or listed hazardous waste based solely on the presence of PFAS chemicals; however, given the potential for future liability, it is recommended that project teams design investigations to minimize generation of investigation derived waste (IDW).

Solid IDW may be disposed as non-hazardous solid waste. Investigators should clearly note the presence of PFAS on waste manifests for full disclosure of contents. For liquid IDW (e.g., purge water), a sample shall be analyzed prior to disposal. If the combined concentration of PFOS/PFOA is less than 70 parts per trillion (ppt), and assuming that no other contamination is present and no state or local regulation prohibits it, the water may be discharged to the sanitary sewer after disclosing the nature and concentrations of PFAS constituents contained in the liquid IDW to the local wastewater authority and after obtaining a recordable authorization from the authority. Liquid IDW with a combined PFOS/PFOA concentration greater than 70 ppt shall be held pending written authorization by the facility director of the treatment plant that will receive the liquid. If no treatment facility is available then disposing liquid IDW as liquid non-hazardous waste at an EPA approved Subtitle-D Industrial Waste Landfill or equivalent facility capable of processing liquid non-hazardous waste should be considered, and written authorization and acceptance of the PFAS containing IDW should be obtained from the landfill. Additionally, treatment of liquid IDW to bring the waste to acceptable disposal levels may be conducted.

PFAS are NOT classified as a hazardous waste by definition because PFAS are not regulated by RCRA. However, individual states have been and can be more stringent than the EPA. Waste should be labeled as a "Non-Regulated Waste" or other state mandated labeling requirements with special instruction to the treatment, storage, and disposal facility to use thermal destruction as the means to destroy waste. This classification can change depending on individual state definitions. Due to the uncertainty in the regulatory and legal environment surrounding PFAS, this guidance is subject to frequent updates.

12 References

APHC Technical Information Paper No. 85-067-0117, 2017, Environmental Criteria Perfluorinated Alkyl Compounds.

APHC Chemical and Material Emerging Risk Alert, undated, Aqueous Film Forming Foam (AFFF).

Department of Defense and Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.3, 2019.

Department of Defense Instruction (DoDI) 4715.18, Emerging Contaminants, 11 June 2009.

EDQW Fact Sheet, October 2016, Bottle Selection and other Sampling Considerations When Sampling for Per- and Poly-Fluoroalkyl Substances (PFASs)

Environmental Fate and Effects of Poly-and Perfluoroalkyl Substances (PFAS). 2016. CONCAWE (European Industrial Consortium)

EPA Office of Solid Waste and Emergency Response (OSWER) Directive 9355.0-30, 1991, Role of the Baseline Risk Assessment in Superfund Remedy Selection Decisions.

EPA OSWER Directive 9285.7-53, 5 Dec 2003, Human Health Toxicity Values in Superfund Risk Assessments.

EPA OSWER 9285.7- 02EP, July 2004, Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part E, Supplemental Guidance for Dermal Risk Assessment), EPA/540/R/99/005.

EPA OSWER, 28 October 2009, The Toxicity of Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS).

EPA Fact Sheet, March 2014, Emerging Contaminants - Perfluorooctane Sulfonate (PFOS) and Perfluorooctanoic Acid (PFOA), (http://www2.epa.gov/sites/production/files/2014-04/documents/factsheet_contaminant/ _pfos_pfoa_march2014.pdf).

EPA Office of Water, May 2016, Drinking Water Health Advisory for Perfluorooctanoic Acid (PFOA) EPA 822-R-16-005.

EPA Office of Water, May 2016, Drinking Water Health Advisory for Perfluorooctane Sulfonate (PFOS).

EPA Office of Water, 15 November 2016, 2016. Clarification about the Appropriate Application of the PFOA and PFOS Drinking Water Health Advisories.

Government of Western Australia, Department of Environmental Regulation. 2016. Interim

Guideline on the Assessment and Management of Perfluoroalkyl Substances (PFAS), Contaminated Sites Guidelines. February.

Memorandum, Office of the Assistant Secretary of Defense (OASD) for Energy, Installations and Environment, 10 June 2016, subject: Testing DoD Drinking Water for Perfluorooctane Sulfonate (PFOS) and Perfluorooctanoic Acid (PFOA).

Memorandum, OASD, 9 March 2012, subject: Revised Site Management procedures – Update to DoD Manual 4715.20, Defense Environmental Restoration Program Management.

Memorandum, Headquarters, Department of the Army (HQDA), Assistant Secretary of the Army for Installations, Energy and Environment (ASA IE&E), 10 June 2016, subject: Perfluorinated Compound (PFC) Contamination Assessment.

Memorandum, HQDA ACSIM, 29 August 2016, subject: Department of the Army Guidance to Address Perfluorooctane Sulfonate and Perfluorooctanoic Acid Contamination.

Memorandum, HQDA ACSIM, 21 June 2017, subject: Supplemental Drinking Water Monitoring Guidance for Perfluorooctane Sulfonate and Perfluorooctanoic Acid.

Memorandum, HQDA, Assistant Chief of Staff For Installation Management, February 2018. subject: Army Guidance for Addressing Releases of Per-and Polyfluoroalkyl Substances.

Memorandum, HQDA Assistant Chief of Staff For Installation Management, 5 March 2019, subject: Aqueous Film Forming Foam (AFFF), Removal, and Disposal.

United Nations Environment Programme (UNEP), Division of Technology, Industry and Economics. 2015. PFAS analysis in water for the Global Monitoring Plan of the Stockholm Convention, Set-up and guidelines for monitoring. April.

HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE Approved by: Corporate Quality Manager	
Field Logbook Use and Maintenance		**SOP No.: 300.04 (formerly 4.07)** **SOP Category: QA/QC** **Revision No.: 3** **Revision Date: November 20, 2019** **Review Date: November 2021**

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the methods for use and maintenance of field logbooks. This procedure outlines methods, lists examples for proper data entry into a field logbook, and provides the standardized HGL format. Field logbooks provide a means for recording observations and activities at a site and are intended to provide sufficient data and observations to reconstruct field events. Logbooks are a primary source of evidence referenced during legal proceedings. The overall requirement is to document field activities without having to rely on memory.

2.0 SCOPE AND APPLICATIONS

This procedure provides guidance for logbook use and maintenance during routine field operations on environmental projects. Site-specific deviations from the methods presented herein must be approved by the assigned HGL project manager and the HGL project quality assurance/quality control officer. Consult the project-specific planning documents for other documentation requirements that apply to the project.

3.0 GENERAL REQUIREMENTS

All project work must be performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan for relevant health and safety requirements.

Any deviations from specified project requirements must be justified to and authorized by the project manager and/or the relevant program manager and documented in the planning documents after consultation and approval by the client (refer to change or variance documentation requirements in the planning documents). Deviations from requirements are documented sufficiently to re-create the modified process and/or product and associated approvals.

All field personnel present on site to conduct work related to environmental projects are responsible for documenting field activities in project field logbooks. If field personnel are working in teams, one team member should be assigned to document the work performed. Documentation in the logbooks must be legible, and the logbooks must be maintained over the course of the project in accordance with this SOP.

The HGL field team leader, or approved designee, prepares daily logs to provide clients records of significant events, observations, and measurements taken in the field. These daily logs rely on documentation from the logbooks and should match.

The HGL field team leader should check logbook entries at the end of each field day to ensure that they are complete/adequate and communicate any deficiencies and corrective measures immediately. Logbook entries should be reviewed on a regular basis by the project manager or an approved designee to verify that they have been completed in accordance with this SOP. This could be done as part of the three-phase control inspections for each task or definable feature of work. Regular reviews of logbook ensure that adjustments to the information in the logbook, if needed, can be made early on in the performance of the task and establish expectations for documented information.

4.0 PROCEDURE

4.1 INTRODUCTION

Field logbooks provide a means for recording and documenting for the record observations and activities at a site. Field logbooks are intended to provide sufficient data and observation notes to enable participants to reconstruct events that occurred while performing field activities and to refresh the memory of field personnel when drafting reports or giving testimony during legal proceedings. As such, all entries must be as factual, detailed, and as descriptive as possible so that a particular situation can be reconstructed without reliance on the memory of field crews. Field logbooks are not intended to be used as the sole source of project or sampling information. A sufficient number of logbooks are be assigned to a project to ensure that each field team has a logbook at all times.

4.2 FIELD LOGBOOK IDENTIFICATION

Field logbooks are bound books with consecutively prenumbered pages. Logbooks are permanently assigned to field personnel for the duration of the project or sampling event. When not in use, the field logbooks are to be stored in site project files. If site activities stop for an extended period (2 weeks or more), field logbooks are be stored in the project files in the appropriate HGL office. The field logbooks are be scanned on a regular basis, grouped in files by field event and by logbook, and stored electronically in the proper project file located on SharePoint.

The cover of each logbook contains the following information:

- Organization to which the book is assigned (HGL),
- Site name (including operable unit designation),
- Project number,
- Book number, and
- Start and end dates of the information in the logbook.

4.3 LOGBOOK ENTRY PROCEDURES

Every field team must have a logbook, and each field activity is be recorded in the logbook by a designated field team member to provide daily records of significant events, observations, and measurements during field operations. Beginning on the first blank page and extending through as many pages as necessary, the following list provides examples of useful and pertinent information that may be recorded (optional).

- Serial numbers and model numbers for equipment that will be used for the project duration,
- Formulas, constants, and example calculations,
- Useful telephone numbers, and
- County, state, and site address.

Entries into the logbook may contain a variety of information. At a minimum, the following information must be recorded on the first page of the logbook entry for each workday:

- Date (on all pages),
- Site name, site location, and project number,
- Weather at start of day and projected for the day (changes during the day should be documented at the time of the change),
- Names of field personnel and subcontractors present and directly involved in the field activities, with their initials in order to reference them by initials during the day to facilitate note taking,
- Level of personal protective equipment being used on the site,
- Equipment used and calibration procedures followed,
- Start time, and
- Any field calculations.

In addition, information recorded in the field logbook during the day includes, but is not limited to, the following:

- Sample description including sample numbers, collection time, depth, volume, type and number of containers, preservative, and media sampled;
- Information on field quality control samples (e.g., duplicates, trip blanks, rinsates, and matrix spike/matrix spike duplicates [MS/MSDs]);

- Sample courier airbill numbers and associated chains of custody numbers;
- Observations about site and samples (odors, appearances, etc.);
- Information about any activities, extraneous to sampling activities, that may affect the integrity of the samples;
- Any public involvement, visitors, or press interest, comments, or questions; as well as times present at site;
- Equipment used on site including time and date of calibration along with calibration gas/fluid lot numbers and expiration dates, and calibration results;
- Background levels of each instrument and possible background interferences;
- Instrument readings for the borehole, cuttings, or samples in the breathing zone and from the specified depth of the borehole, etc.;
- Field parameters (pH, specific conductivity, etc., as required by the sampling method and planning documents);
- Unusual observances, irregularities, or problems noted on site or with instrumentation used;
- Maps or photographs acquired or taken at the sampling site, including photograph numbers and descriptions;
- A photographic log that lists subject, person taking photograph, distance to subject, direction, time, photograph number, and noteworthy items for each photograph stating what feature/item the photo is documenting;
- A description of the investigation-derived waste (IDW) generated, the quantity generated, and the manner of IDW storage employed; and
- Forms numbers/titles and any information contained therein used during sampling (Note that a form does not take the place of the field logbook.).

All logbook entries are made in indelible black or blue ink. No erasures are permitted. If an incorrect entry is made, the data is crossed out with a single strike mark and initialed and dated by the originator. Entries are be organized into easily understandable tables if possible. A sample format is shown in Attachment 1. A Logbook Quick Guide, which provides logbook entry requirements and suggestions, is included as Attachment 2. This guide can be copied and taped to the inside cover of a logbooks for quick reference.

All logbook pages are initialed and dated at the top of each page. The time (in 12- or 24-hour format) is recorded next to each entry. No pages or spaces are left blank. If the last entry for a day is not at the end of a page, a diagonal line is drawn through the remaining space, and the line is signed and dated.

Logbooks can become contaminated when used in the field. Every effort should be made by the field team to avoid contaminating the logbook. Logbooks can be kept in seal-top poly bags, or temporary plastic covers may be used.

4.4 REVIEW

The assigned field team leader, or an approved designee, checks field logbooks for completeness and accuracy on an appropriate site-specific schedule determined by the project leader. Any discrepancies in the logbooks are noted and returned to the originator for correction. The originator or other field team member knowledgeable about the field task reviews the comments, makes appropriate revisions, and signs and dates them. The reviewer verifies that revisions have been made before placing the logbook photocopies on the project file in SharePoint.

5.0 REVISION HISTORY

Revision 0		Initial Release
Revision 1	December 2010	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	July 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	November 20, 2019	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.

ATTACHMENTS

Attachment 1 – Example Field Logbook
Attachment 2 – Logbook Quick Guide

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ATTACHMENT 1
EXAMPLE FIELD LOGBOOK

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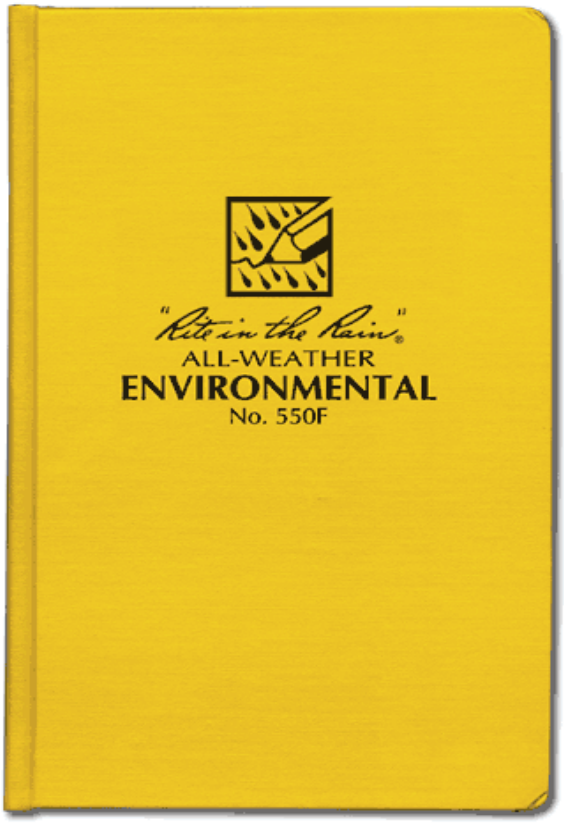
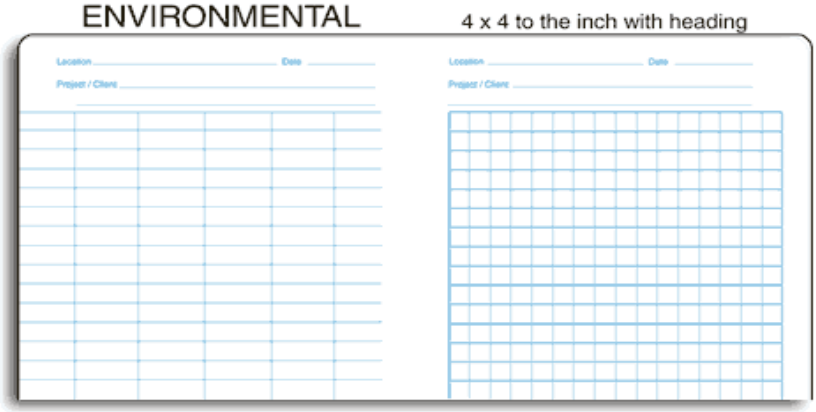
ATTACHMENT 1
Example Field Logbook





INFORMATION RECORDED IN THE FRONT OF LOG BOOKS (OPTIONAL)

- serial/model #s of equipment (meters)
- formulas, constants, example calcs
- useful phone #s
- site address

DAILY RECORDING REQUIREMENTS

- initials and date (top of every page)
- start time
- weather
- deem methods (you may cross reference a previous days method if identical)
- personnel present on site
- ppe
- signature of individual recording info
- equipment/procedures used
- sample descriptions (time, depth, volume, containers, preserv, etc.)
- QC samples (field and lab)
- observations
- field parameters
- maps/photos drawn or taken
- form #s
- lost/voided paperwork

Photo log:
Subject & persons.
distance to subject
person taking photo.
direction of each on.
Time | photo # | roll #.
Note anything else.

When using a field form information recorded in the field does not need to be written twice. Cross reference the field form # in the log book and record the information only on the appropriate field form.

DO NOT LEAVE ANY BLANK SPACES/PAGES. If a page is accidentally left blank or there is unused space at the end of a day's entry draw a diagonal line through the space and initial and date the line.

Anna Vogel AV 11/6/95
1

November 6, 1995, AX1015.13.00

pH Meter
Hach
Model # = 12345
Serial # = 6789

Conductivity Meter
Hach
Model # = 12345
Serial # = 6789

$c^2 = a^2 + b^2$
if $a = 3$
if $b = 4$
then: $c^2 = 3^2 + 4^2$
$c^2 = 25$
$c = \sqrt{25}$
$c = 5$

$\pi = 3.14159$

Anna Vogel home # 123-4567
VOS Denver Office # 303/296-9700
VOS San Francisco # 415/774-2700 (travel)

Smith Site
Badger County, Colorado
Address: 1234 W. Main Street
Manzana, Colorado 00000
Directions to Site:
West on I-70
Exit 93B.
Head south approx. 3 miles
Site is on east side of dirt road.

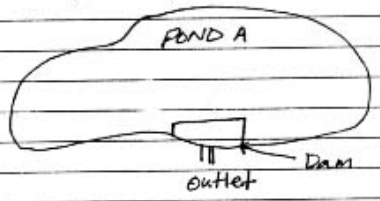
AV 11/6/95
2

November 6, 1995 Site Visit
0700 Arrive on site
Weather: 80°, sunny, slight breeze (~5 mph) from southwest
UOS Field Team: EPA OSC:
M.R. Smith J.P. Scarton
K.W. Wagner
P.R. Lane
PRP representative, L.M. Stein, will be accompanying the UOS Field Team.
Personal Protective Equipment - LEVEL D will be used on-site. (refer to site-specific health & safety plan).
All equipment will be deconed as follows:
- Brush equipment scrub-brush to remove gross particulates.
- Scrub thoroughly with Alconox/water solution.
- Rinse with reagent-grade distilled water.
- Rinse with reagent-grade Methanol.
- Rinse with reagent-grade distilled water.

Allow equipment to gravity drain
Wrap equipment in tinfoil if not immediately used.
Sample procedure:
All surface water samples will be taken using a clean decontaminated TEFLON scoop; stainless steel spoon and stainless steel bowl will be used for sediment samples.

AV 11/6/95
3

The samples will be taken from the ponds at the center of the dam opposite the outlets. (see below; refer to sample plan).
All total suspended solids (TSS) samples will be collected in a 500 ml polystyrene bottle - No preservative is necessary.
All VOA samples will be collected in two 40-ml amber glass vials and will be collected first. Preservation will be 4°C (ice).
→ Meters (pH) Decon = Rinse with reagent-grade distilled water.



0730: leave trailer. Go to sample location SS-1 @ Pond A.
0745: Arrive @ POND A.
Decon. equipment as described on page 2 of this logbook.
Calibrate pH meter - Rinse probe

Time	STD	Reading	
0753	7.00	7.00	Rinse probe
0754	4.00	4.00	Rinse probe

0756 Calibrate Conductivity Meter using 10000 STD - Rinse probe

Time	Sample	Sample#	Label #
0802	VOA	81088 VOAA	101
0803	TSS	8108 TSSA	103*

Decon equipment (scoop only).
*labed 102 fell in mud - destroyed it.
Field parameters

Time	pH	Conductivity
0815	6.35	610

Decon equipment (meters only)
Fill out surface water quality Sheet.
Note — wind speed is picking up — The ponds become turbulent.
0829 - Leave Pond A - go to Pond B.
AV
0840 — Arrive at Pond B
Pond B sampling procedure.
0842 - Decon equipment.
Calibrate pH meter

Time	STD	Reading	
0844	4.00	4.00	Rinse probe
0845	7.00	7.00	Rinse probe

0847 Calibrate conductivity meter using 10000 STD - Rinse probe.
Decon sampling equipment (scoop).

Time	Sample	Sample#	Label #
0902	VOA	81088 VOABD	106
0903	TSS	81088 TSS BD	107

0905 Decon scoop.
AV
Rinsate Samples

Time	Sample	Sample#	Label#
0920	VOA	81088 VOAR	AV 107-108

FIELD PARAMETERS

TIME	pH	Conductivity
0924	6.00	590

Decon meters as noted on page 3 of this logbook.
Fill out surface water quality sheet.
AV
0940 - Leave Pond B - head back to trailer to pack samples for shipment.
0952 - Arrive at Trailer.
0959 - Complete chain-of-custody forms for samples to be shipped.
Wrap Samples according to UOS TSOP.
1020 - Seal Cooler and attach Custody seals.
1030 - Take cooler to Federal Express for shipping.
COC # 1234567.
1035 - Leave Federal express.
Sampling complete.

AV 11/6/95

ATTACHMENT 2
LOGBOOK QUICK GUIDE

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LOGBOOK QUICK GUIDE

TOP

Location: County/City/State
Project/Client: Project/Client Name

MINIMAL REQUIREMENTS

- times of activities (military)
- author of day's entries
- field team members
- field team member assignments
- field activities
- EPA or other regulatory personnel observing - activities
- other personnel
- public or press visitors
- equipment used
- equipment calibration information
- serial numbers of equipment
- weather
- decontamination methods
- level of PPE
- calculations used
- sample information
  - ID
  - depth
  - volume
  - containers
  - preservative
  - media
  - QC samples

LOGBOOK QUICK GUIDE

MINIMAL REQUIREMENTS (cont.)

- background levels and readings
- possible instrument interferences
- photographs
  + number
  + direction
  + description
  + photographer

OTHER REQUIREMENTS

- unusual observations
- strike through mistakes with single line
- diagonal line across unused portion of page with signature and date
- use indelible black or blue ink
- no erasable ink
- generate tables when possible for information
- leave no pages blank
- place North arrow on sketches
- leave no open lines
- staple business cards of visitors in book
- deviations from approved plans
- field forms completed

** Black text applies to all activities*
** Red text applies to activities that include sampling*

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1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the methods for installing groundwater monitoring wells. In addition, this procedure provides guidance for routine field operations on environmental projects.

2.0 SCOPE AND APPLICATIONS

This SOP includes the designs, procedures, and materials used to construct a monitoring well that will produce accurate groundwater level measurements and yield representative groundwater samples. Specific project plans may have well specifications that differ from the design specifications presented in this procedure. In addition, licensing and/or certification of the driller may be required. State and local well installation regulations should be reviewed to confirm that all well construction procedures comply with regulatory agency requirements.

3.0 GENERAL REQUIREMENTS

All work is performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan for relevant health and safety requirements.

Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager and discussed in the approved project plans. Deviations from requirements must be documented sufficiently to re-create the modified process.

4.0 DEFINITIONS

Annulus/Annular Space: The space between the borehole wall and well casing (well screen and blank riser pipe) or the space between the surface/conductor casing and well casing.

Bridging: Gaps or obstructions in the grout, bentonite, or filter pack materials that develop when the well is installed or developed.

Surface/Conductor Casing: The outer casing used to stabilize or seal off a formation to prevent a formation from collapsing or vertical cross contamination from occurring within the well.

Filter Pack: Uniform, clean, and well-rounded sand, gravel, or glass beads placed in the annulus of the well between the borehole wall and the well intake to (1) provide lateral support for the well screen, (2) increase yield, and (3) prevent formation material from entering the well.

Grout: A fluid mixture of water and high-solids sodium bentonite (20% to 30% solids by weight) or neat cement with powdered bentonite (2% to 6% by weight [1.9 pounds to 5.7 pounds per 94-pound bag of Portland cement]) that can be forced through a pipe and emplaced in the annular space between the borehole wall and casing to form a seal. The fluid mixture also may be composed of various additives or bentonite of an appropriate consistency.

Investigation-Derived Waste (IDW): The water, soil, and drill cuttings generated during drilling and decontamination activities.

Pressure Grouting/Sealing: A process by which grout is placed between the borehole and well casing or between a protective surface casing and the borehole using positive pressure to pump grout though a submerged tremie pipe to displace overlying groundwater and drill fluids to maintain grout consistency. In areas designated as special or sensitive habitats, more rigorous pressure grouting methods, not addressed in this SOP, may be required.

Schedule Pipe: The standardization of casing diameters and wall thicknesses where casing wall thickness increases as the schedule number increases.

Screen/Well Intake: A screening device used to keep materials, other than formation fluids, from entering the well.

Slot Size: The width of the slots machined into a slotted well casing (screen) that allows formation fluids into the well.

5.0 PROCEDURE

5.1 INTRODUCTION

The diameter of the monitoring well boring is generally a minimum of 4 inches greater than the outside diameter of the well casing. This is to ensure enough room for insertion of the tremie pipe for filter pack placement and to ensure adequate space for settling of the filter pack so that no bridging occurs during well construction.

During well installation, contamination of the water-bearing zone by drilling equipment or cross-contamination of wells during the drilling process must be avoided. Vertical seepage of surface water into the monitoring well must also be minimized, which may require installation of a surface/conductor casing.

The driller must be trained to operate the specific rig in use and must be licensed to install wells in the state or region in which the work is being performed.

To maintain quality control and obtain accurate formation information, a field geologist must be on the site during well installation to log subsurface conditions (HGL SOP 403.07: *Geologic Borehole Logging*) and construction details for each well.

5.2 PRECAUTIONS

The following precautions should be employed during well installation operations:

- Subsurface utility lines must be identified and cleared before initiating exploratory boring drilling activities. Procedures outlined in HGL SOP 411.03: *Subsurface Utility Avoidance* are followed.
- Aboveground utility lines also must be identified. At a minimum, drilling activities should generally be no closer than 20 feet to an overhead power line unless the power lines are shielded by the local electrical utility. However, if the voltage of the power line is known, the minimum clearance distance can be derived using the table below. Follow the requirements on the Drilling Activity Hazard Analysis included in the project-specific planning documents.

Voltage (Nominal, kV, Alternating Current)	Minimum Clearance Distance
Up to 50	10 feet (3 meters)
51–200	15 feet (4.6 meters)
201–350	20 feet (6 meters
351–500	25 feet (7.6 meters)
501–750	35 feet (10.7 meters)
751–1,000	45 feet (13.7 meters)
Over 1,000	(As established by the utility owner/operator or a Registered Professional Engineer who is a Qualified Person with respect to electrical power transmission and distribution).

Source: Cranes and Derrick in Construction, 29 CFR 1926, Subpart CC Standard Number: 1926.1408

- Every attempt should be made to contain contaminated soil and water and prevent further contamination of the environment.

Potentially contaminated formation materials brought to the surface during drilling activities, IDW, is managed to prevent contamination of the surface area surrounding the borehole. Cuttings should be placed on heavy plastic (6 mil minimum thickness) or plywood, directly into drums, or into a skid-loader bucket if IDW soils are being contained in rolloff boxes. Plastic should be thick enough to prevent it from being puncturing by formation materials, the ground surface, or removal activities. Materials placed on plastic or plywood for an extended period should be covered to protect them from the elements until they can be disposed of properly.

5.3 DECONTAMINATION

All equipment and materials that could spread contamination or that are used directly in the monitoring well installation (for example, well casing, screen, tremie pipe, centralizers, augers) must be thoroughly decontaminated before use or installation in the well unless they have been decontaminated by the manufacturer and shipped in protective plastic sheeting. Decontamination equipment such as steam cleaners and high-pressure hot water cleaners effectively remove potential contaminants left on casings and screens during the manufacturing process. When using polyvinyl chloride (PVC) screen or casing, acid rinse solutions should not be employed for decontamination. All other decontamination procedures must conform with specific protocols outlined in the site-specific field sampling plan and SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.

Decontaminated equipment not used immediately after decontamination should be stored under protective cover, such as aluminum foil or plastic sheeting, until used. Liquid/solid IDW generated during decontamination activities should be managed in accordance with the project-specific planning documents.

5.4 WELL CONSTRUCTION MATERIALS

Materials used in the construction of monitoring wells must be chemically nonreactive to the contaminants suspected to be in the groundwater. The most commonly used well construction materials are PVC and stainless steel. PVC is the most economical and the easiest to use. PVC does not decompose when it comes into contact with groundwater containing low concentrations of organic materials. However, over time, high concentrations of organic contaminants will react with PVC and cause the well screen to decompose. Stainless steel provides greater structural strength, and its use may prove advantageous for large-diameter wells.

Well casing and screen are available in threaded and unthreaded sections, typically in lengths of 5, 10, and 20 feet. Threaded pipe joints may be wrapped with ~~Teflon~~ tape to facilitate joining and to improve the seal of stainless steel products. Sections of casing and screens are assembled on the site to allow inspection immediately before installation. PVC connections must be flush threaded or connected by another mechanical method, as PVC joint sealant will introduce organic contaminants into the well.

Monitoring well construction commonly requires the use of American National Standards Institute (ANSI) Schedule 40 or Schedule 80 PVC pipe to complete monitoring wells. Schedule 40 2-inch pipe is a standard size pipe with a wall thickness of 0.154 inch and an approximate inside diameter of 2.067 inches. Schedule 80 2-inch pipe has a wall thickness of 0.218 inch and an approximate inside diameter of 1.939 inches. Schedule 40 pipe is suitable for most shallow monitoring well applications (total depth less than 100 feet). Schedule 80 pipe is more suitable for wells deeper than 100 feet or for wells to be completed in formations with known swelling properties that could lead to casing collapse.

5.4.1 Well Screen

The purpose of the well screen is to allow sediment-free groundwater to enter the well. The slot size of the well screen is selected based on filter pack material selection. Both the screen and filter pack material are related to the grain size analysis of the aquifer. Typically, for extraction and water supply wells, samples are collected from the formation for grain size analysis to permit design of the filter pack and well screen. However, this is not usually performed for monitoring wells, which typically use 0.010-slot screen, especially for wells completed in clay. Methods for determining the appropriate screen slot and filter pack sizes are available in the U.S. Environmental Protection Agency (EPA)'s *Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells* (EPA, 1991), *Groundwater and Wells* (Driscoll, 1986) and *Practical Handbook of Environmental Characterization and Ground-Water Monitoring* (Nielsen, 2006). Screen slot and filter pack sizes are selected using industrywide accepted methods.

For monitoring well construction, two major types of screens are used: continuous slot wire wrap screen and slotted pipe. Wire wrap provides the greatest open area and results in higher yields. However, it is significantly more expensive than slotted pipe. Continuous slot wire wrap screen is most effective when used to sample low-yield formations.

Slotted pipe is composed of the same Schedule 40 or Schedule 80 casing pipe, but it has been machined to create uniform openings. Slotted pipe has a smaller effective open area than continuous slot wire wrap screen, but it is usually adequate for wells installed in relatively shallow, permeable formation aquifers. The effective open area should be at least 2.70 square inches per lineal foot for 10-slot, 2-inch slotted pipe, and 4.50 square inches per lineal foot for 20-slot, 2-inch slotted pipe.

The well screen length will vary depending on site conditions. However, for typical monitoring well installations, the screen lengths may vary from 5 to 20 feet, but typically they are 10 feet. Well screens lengths greater than 20 feet may permit vertical migration from a contaminated zone to a clean zone through the well screen. Therefore, well screen lengths greater than 20 feet are typically not used. Consideration should be given to whether the well screen should be placed across the water table to monitor for floating product or whether seasonal variations in the water table may require a longer length of screen to be installed.

Note that state and/or local regulations may require that the well screen and riser have centralizers every 50 feet or less to ensure installation of a plumb well centered in the borehole. For wells over 50 feet in depth, centralizers are placed at the base of the well screen and at the top of the filter pack. The specific placement intervals for additional centralizers are based on site-specific conditions and ensure that the placement of the filter pack, bentonite seal, and annular seal will not be hindered. The use of centralizers in wells constructed through hollow stem augers is not required.

5.4.2 Well Filter Pack

The purpose of the well filter pack is to (1) provide lateral support for the well screen, (2) increase yield by improving the hydraulic conductivity in the immediate vicinity of the well, and (3) retain the formation to prevent natural material from entering the well.

Filter packing allows larger screen slot openings to be used, which in turn increase well recharge rates.

The materials used to construct the filter pack must be chemically inert (for example, clean quartz sand, silica, or glass beads), well rounded, and dimensionally stable.

Clean and properly packaged silica sand is the most commonly used pack material and should consist of 90 to 95 percent quartz grains. The filter pack should uniformly envelop the well screen with a thickness of no less than 2 inches or more than 8 inches.

Pack size should be such that it retains 90 percent of the surrounding formation while the screen slot size must retain 90 percent of the filter pack. A tremie pipe may need to be used to install the filter pack to avoid bridging. The tremie pipe should be placed near the bottom of the screen to ensure that the sand settles through turbid groundwater.

Filter pack sand is typically brought up to a depth 2 or 3 feet higher than the top of the screen. The volume of sand required to fill the annular space around the screen should be calculated prior to placement of the sand and compared to the actual volume used, with the volume noted in the field logbook.

5.4.3 Well Seal

The materials used to seal the annulus between the borehole wall and casing, above the filter pack, must prevent contaminant migration from ground surface or intermediate zones and must prevent cross contamination between strata. The materials must be chemically nonreactive to the contaminants found on the site so that they do not affect the quality of the groundwater samples. The permeability of the sealants should be 1 to 2 orders of magnitude less than the surrounding formation.

The seal material is bentonite pellets and/or a slurry of bentonite. The actual mixture of the materials to be used in any boring is determined in the field and is based on drilling and sampling data. Typically, a seal of bentonite pellets with a minimum thickness of at least 2 feet is installed above the filter pack to more effectively seal the screened section of the well and to prevent the intrusion of overlying cement bentonite grout or high-solids bentonite slurry into the filter pack. After the bentonite pellets are in place, they should be hydrated with potable water. Typically, the hydrated bentonite pellets must be allowed to set for 4 hours before placement of grout on top of the seal. Bentonite slurry can be used as the well seal; however, this requires placement of

at least 2 to 3 feet of fined-grained buffer sand on top of the filter pack to prevent grout intrusion into the filter pack.

5.4.4 Annulus Backfill

The annular space above the filter pack and seal is grouted with a high-solids bentonite or bentonite/cement mixture. Grouting is used to minimize the vertical migration of water to the groundwater intake zone and to increase the integrity and stability of the well casing. Site-specific conditions should be considered when selecting grout type. In some geologic settings such as highly fractured zones, or in situations when the bentonite seal has been compromised, it may be possible for the bentonite/cement grout to bypass the bentonite seal and enter the screen interval, raising the pH of the groundwater. For this reason, use of a high-solids bentonite slurry may be preferable to use of a bentonite/cement grout.

For bentonite/cement mixture grouts, between 1.9 (2%) and 5.7 (6%) pounds of bentonite should be mixed with 6 gallons of water per 94-pound bag of cement, with 0.6 gallon of water added for every additional percentage of bentonite used (for example, 7.2 gallons of water for one 94-pound bag of cement with 2% bentonite). The bentonite should be added to the mix water before the mix water is added to the cement to allow the bentonite to disperse. Drillers must be notified of this mixing order to prevent the bentonite from clumping, which will occur if the bentonite s added directly to the cement mixture. For cement grout, the grout must consist of no more than 6 gallons of potable water per 94-pound bag of cement. Cement grout should be mixed thoroughly and be free of lumps. After grouting, the well should not be disturbed or developed for a minimum of 24 hours.

5.5 WELL INSTALLATION

Monitoring wells are constructed in a manner like that shown on the diagram in Attachment 1, Monitoring Well Construction Details. Some exploratory borings may require partial backfilling before the screen and riser are installed. The field geologist determines the well depth and the screen setting for each well, as well as the need for partial backfilling before well installation. Attachment 1, Monitoring Well Construction Details, is an example form used to record well construction data.

Partial backfill materials below the screen consist of bentonite pellets or clean sand. Due to the high pH and ion exchange capacity of bentonite and the related potential for change in groundwater chemistry, special care must be taken to ensure that the backfill and well screen are not near each other. Therefore, construct the well in such a manner that a minimum of 1 to 2 feet of filter pack is placed between the backfill and well screen. The actual mix of the materials to be used in any boring is determined in the field based on drilling and sampling data. The field geologist determines the depth to which annulus well materials are placed after observing the subsurface conditions at each well boring location. The drill crew constantly monitors backfill

depths to the satisfaction of the field geologist by means of a weighted steel or plastic measuring tape.

Monitoring wells installed using hollow stem auger or sonic drilling methods are constructed inside the hollow stem auger or sonic drill pipe. Monitoring wells installed using rotary methods are constructed in open boreholes. Monitoring wells are installed by placing the riser pipe and screen into the completed borehole and backfilling the annulus with clean filter pack material. A tremie pipe may need to be used to install the filter pack to avoid bridging. The borehole annulus is backfilled to a minimum of 2 feet above the well screen. After the depth to filter pack has been confirmed, the seal, bentonite pellets, and/or a slurry of bentonite are installed directly above the filter pack at a minimum thickness of 2 feet. Potable water is added, and the bentonite pellets are allowed to hydrate according to the manufacturer's instructions and regulatory requirements. An annular seal consisting of either a high-solids bentonite slurry or a bentonite/cement slurry is placed above the bentonite pellet seal by pressure grouting using a tremie pipe. Pressure grouting is performed using a positive pressure pump to place grout through a submerged side-discharge tremie pipe and displace water in the annulus, thereby maintaining grout consistency. The side-discharge tremie pipe is used to prevent the integrity of the bentonite seal from being compromised, which could cause the grout to intrude into the screened interval.

An accurate record of the quantity of potable water added to the well during installation must be noted in the field logbook. The remainder of the boring annulus is backfilled with a cement/bentonite grout or high-solids bentonite grout to within 3 feet of the ground surface.

A permanent measuring point reference mark is placed on the casings of completed wells. This mark provides a consistent point from which to collect water-level readings. Typically, this mark is made when well elevations and locations are surveyed.

In cases when wells are drilled through a zone of known contamination into deeper water-bearing zones, the potential for contamination or downward contaminant transport via drilling activities exists. In these cases, deep wells must be constructed in a manner that seals the upper contaminated aquifer from the lower aquifer. Methods typically employed for this procedure include installing permanent a surface/conductor casing or temporary casing to seal zones of known contamination. Permanent surface/conductor casing typically consists of steel or PVC pipe grouted in place. Temporary casing is typically associated with sonic drilling and consists of a drill pipe set as a temporary casing into the confining layer. With either casing method, after a seal is established, a borehole of smaller diameter may be drilled through the conductor casing into the lower zone of unknown contaminant levels, and general well installation procedures may be followed in the lower aquifer. If the temporary casing method is used to seal the confining layer, the drill pipe is removed during the grouting process.

The exact method for isolating a zone of known contamination may vary depending on site-specific conditions and may be specified in state or local regulations for sensitive or special

areas. The field geologist and driller decide the most appropriate method for aquifer isolation and deep well completion based on site-specific field conditions.

A concrete surface seal is placed around the annulus of the well to a minimum depth of 1 foot or to the top of the grout seal, whichever is deeper. A protective steel casing (minimum of 4 inches in diameter, 4 feet in length) equipped with locking caps is installed over the well. Where protective casings are employed, two 0.25-inch-diameter holes are drilled at the base of the protective casing at the ground surface to allow water drainage from inside the casing. Three well guards or post protectors may be placed in a radial pattern around each well if the project leader determines that such protection is necessary to prevent damage to the protective casing or well. The well guards are placed 4 feet from the well, driven 2 to 3 feet below ground surface, and rise 3 feet above the ground surface. A concrete pad is placed around the well on the ground surface according to client or state regulations. The pad is formed in such a manner as to direct surface moisture away from the base of the protective steel casing.

Alternatively, if the well is in an area where frequent vehicular traffic occurs, a commercially supplied traffic-rated box may be used as a protective wellhead. The box is installed flush with the ground surface, and the well may be installed below the ground surface. Appropriate locking mechanisms and locks are used to secure the well. The concrete pad is sloped away from the protective wellhead to prevent surface runoff from entering the flush-mount well.

Upon completion of the monitoring well installation, it should be surveyed in accordance with the project-specific planning documents and developed in accordance with SOP 406.01: *Monitoring Well Development.*

6.0 RECORDS

All activities conducted during monitoring well installation should be documented as follows:

- Document all daily field activities on a daily field activity report.
- Complete the field logbook in accordance with procedures listed in SOP 300.04: *Field Logbook Use and Maintenance*.
- Complete borehole logs in accordance with SOP 403.07: *Geologic Borehole Logging*.
- Complete a monitoring well construction form to record well construction data.

7.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
0	December 2010	Initial Release
1	May 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	February 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
3	November 25, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 2.22 to 406.02.
3	June 23, 2021	Updated to incorporate client editorial comments.

8.0 REFERENCES

Driscoll, Fletcher G., 1986. *Groundwater and Wells*. Johnson Division, St. Paul, Minnesota.

Nielsen, David M., 2006. *Practical Handbook of Environmental Characterization and Ground-Water Monitoring*. Second Edition.

U.S. Environmental Protection Agency (EPA), 1991. *Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells.*

ATTACHMENTS

Attachment 1 – Monitoring Well Construction Details

ATTACHMENT 1
MONITORING WELL CONSTRUCTION DETAILS

WELL CONSTRUCTION FORM
(STANDARD WELL)

FIELD REPRESENTATIVE: _____

DRILLING CONTRACTOR: _____

DRILLING TECHNIQUE: _____
AUGER SIZE AND TYPE: _____

BOREHOLE IDENTIFICATION: _____
BOREHOLE DIAMETER: _____
WELL IDENTIFICATION: _____

WELL CONSTRUCTION START DATE: _____
WELL CONSTRUCTION COMPLETE DATE: _____

SCREEN MATERIAL: _____
SCREEN DIAMETER: _____
STRATUM-SCREENED INTERVAL (FT): _____

CASING MATERIAL: _____
CASING DIAMETER: _____

TYPE OF FILTER PACK: _____
GRADIATION: _____
AMOUNT OF FILTER PACK USED: _____

TYPE OF BENTONITE: _____
AMOUNT BENTONITE USED: _____

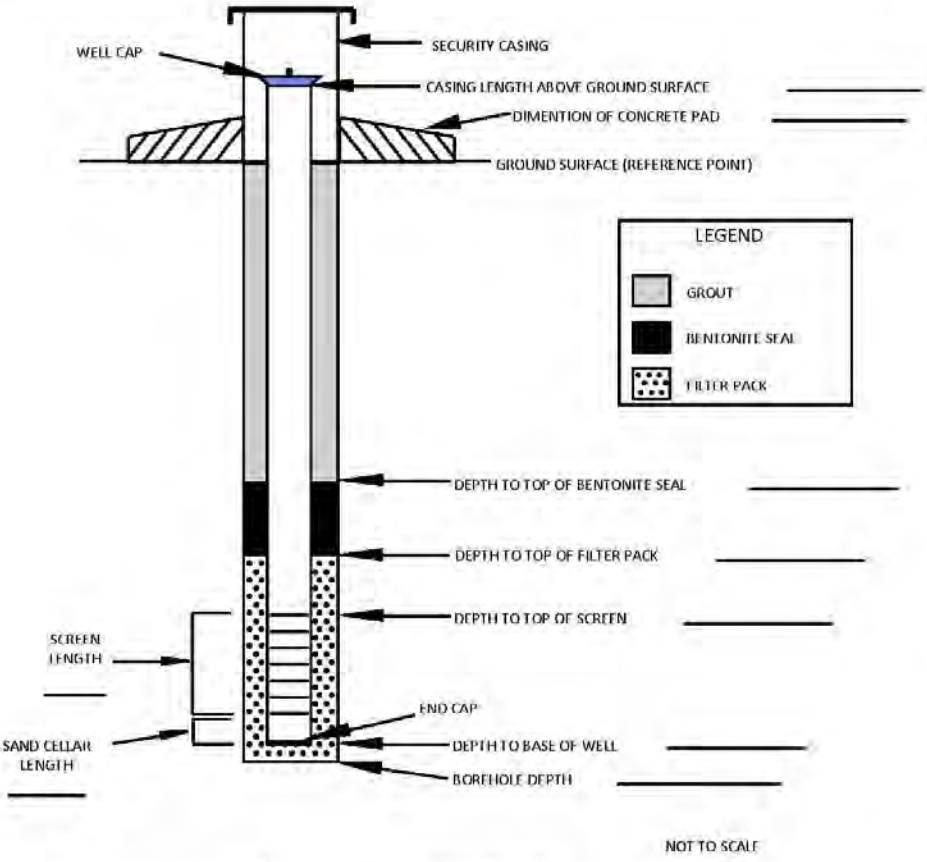
TYPE OF CEMENT: _____
AMOUNT CEMENT USED: _____
GROUT MATERIALS USED: _____

DIMENSIONS OF SECURITY CASING: _____

TYPE OF WELL CAP: _____
TYPE OF END CAP: _____

COMMENTS:

SPECIAL CONDITIONS
(describe and draw)



INSTALLED BY: _____ INSTALLATION OBSERVED BY: _____

DISCREPANCIES: _____

WELL CONSTRUCTION FORM
(FLUSHMOUNT WELL)

FIELD REPRESENTATIVE: _____

DRILLING CONTRACTOR: _____

DRILLING TECHNIQUE: _____
AUGER SIZE AND TYPE: _____

BOREHOLE IDENTIFICATION: _____
BOREHOLE DIAMETER: _____
WELL IDENTIFICATION: _____

WELL CONSTRUCTION START DATE: _____
WELL CONSTRUCTION COMPLETE DATE: _____

SCREEN MATERIAL: _____
SCREEN DIAMETER: _____
STRATUM-SCREENED INTERVAL (FT): _____

CASING MATERIAL: _____
CASING DIAMETER: _____

TYPE OF FILTER PACK: _____
GRADIATION: _____
AMOUNT OF FILTER PACK USED: _____

TYPE OF BENTONITE: _____
AMOUNT BENTONITE USED: _____

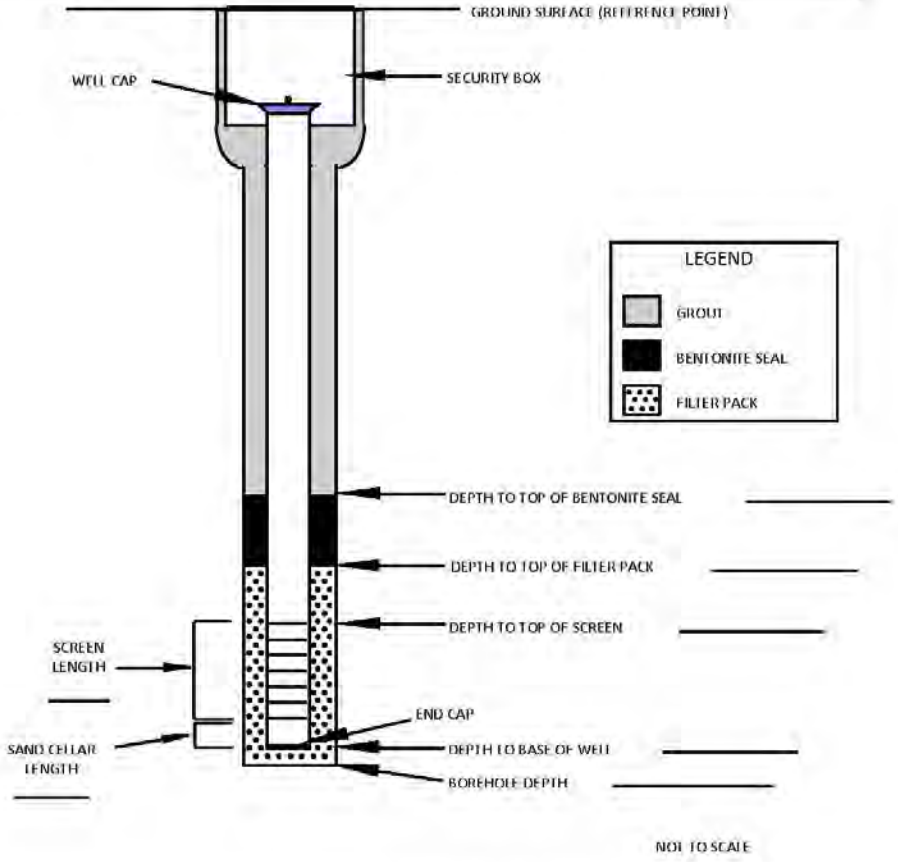
TYPE OF CEMENT: _____
AMOUNT CEMENT USED: _____
GROUT MATERIALS USED: _____

DIMENSIONS OF SECURITY BOX: _____

TYPE OF WELL CAP: _____
TYPE OF END CAP: _____

COMMENTS:

SPECIAL CONDITIONS
(describe and draw)



INSTALLED BY: _____ INSTALLATION OBSERVED BY: _____

DISCREPANCIES: _____

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HGL HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE Approved by: Corporate Quality Manager	
Monitoring Well Development		**SOP No.: 406.01 (formerly 2.10)**
		SOP Category: Environmental Services
		Revision No.: 2
		Revision Date: November 20, 2019
		Review Date: November 2021

1.0 PURPOSE

This standard operating procedure (SOP) describes the methods to be used for developing groundwater monitoring wells on environmental site investigations. Drilling activities associated with installation of groundwater monitoring wells can result in disturbances to the sediments adjacent to the well screen. These disturbances include, but are not limited to, the following:

- Smearing finer-grained sediments along the borehole walls, which can occur in the following types of drilling:
  - Hollow-stem auger drilling: Smearing can occur during the rotation of the hollow-stem augers.
  - Mud-rotary drilling: Smearing can be caused by the creation of a mud-cake on the borehole walls during drilling.
  - Air-rotary drilling: The creation of rock dust lining the borehole during air-rotary drilling can cause similar smearing conditions.
  - Sonic drilling: The drill pipe can compact the soil in the borehole walls and cause similar conditions to smearing.
- Injecting non-native water or use of drilling fluids in the borehole, and
- Sloughing sediments from the borehole sidewall during well construction.

Well development activities are designed to (1) remove the disturbed sediment within the well, (2) remove the drilling fluids and non-native water introduced during drilling activities, (3) clear preferential groundwater flow pathways, and (4) establish a connection with the penetrated aquifer. This SOP describes three well development methods and discusses the advantages and disadvantages of each method. Less frequently used development methods such as jetting and air-lifting are not addressed in this SOP but may be considered if the methods listed within this SOP do not prove effective. Deviations from the methods presented herein as required to accommodate site-specific conditions must be approved by the project manager and documented on field sheets and in subsequent reports.

2.0 GENERAL REQUIREMENTS

All work will be performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan for relevant health and safety requirements.

Any deviations from specified requirements will be justified and authorized by the project manager and/or the relevant program manager. Deviations from requirements will be sufficiently documented to re-create the modified process.

The personnel performing well development or redevelopment activities are responsible for performing the applicable tasks as outlined in this SOP and the project-specific planning documents. The project manager, or an approved designee, is responsible for checking all work performance and verifying that the work satisfies the applicable tasks required by this SOP and planning documents. This will be accomplished by reviewing all documents and data produced during work performance. All activities and data collected will be recorded in the field logbook and on the well development field form (Attachment 1).

3.0 PROCEDURES

3.1 INTRODUCTION

Monitoring well development is the process of flushing the formation interface and cleaning the filter pack and the well screen slots to permit unimpeded flow of groundwater into the monitoring well. The drilling subcontractor has the option of performing initial development prior to installation of the well screen and filter pack in situations where the borehole is stable and the water in the well is very turbid, making development after the filter pack installation difficult. Water produced from a properly developed monitoring well represents formation water and does not contain contaminants introduced during drilling and well construction or formation materials loosened during well installation.

Development is necessary to achieve the following:

- Repair damage done to the formation by drilling so that the natural hydraulic properties are restored and groundwater can enter the well screen freely,
- Remove clays, silts, and fine sands (fines) from the filter pack and well screen so that groundwater samples are not turbid and silting of the well does not occur, and
- Remove any remnant water ,drilling fluids, or contaminants introduced during drilling.

Table 1 presents the three major methods HGL uses to monitor well development. The major considerations for determining which monitoring well development method should be used are the lithologic and stratigraphic characteristics of the interval in which the well is screened. Logistical considerations should be secondary. Methods also can be used with any other method, such as mechanical surging while pumping.

Table 1
Well Development/Redevelopment Methods

Method	Best Application	Avoid
Mechanical Surging (surge block)	Most effective in wells screened in medium- to high-porosity/hydraulic conductivity lithologies. Surging with a pump of similar size to the internal well diameter or surge block with tubing and check valve may be used to remove fine-grained sediment in low hydraulic conductivity wells with low yield.	Less effective in wells screened in low permeability lithologies such as clay sand silts
Bailer (stainless steel)	Wells screened in low-permeability formations. The up and down motion of the bailer also adds some mechanical surging to development if the diameters of the bailer and the well are similar.	Deep or large purge volume wells. Generally only practical in 2-inch-diameter wells or shallow 4-inch-diameter wells.
Pumping (variety of high-volume pumps)	Deep or large-volume wells. May also be applied to low yield wells with some limitations. The pump can also be used to surge the well during pumping.	Wells screened in a combination of high- and low-permeability lithologies. Low yield wells may require alternating periods of pumping followed by recharge to above the top of the screen to avoid only developing a limited portion of the screened interval.

During monitoring well development, organic vapors will be monitored with a photoionization detector (PID) to evaluate the potential for fire, explosion, and toxic effects on field personnel. The maximum sustainable flow (well yield) will be determined, if required, and recorded in the field logbook for each monitoring well. The well yield is the maximum sustainable rate, measured in gallons per minute, at which the well can be pumped before the water level in the well falls below the screened interval. The water level can be measured with an electric water level meter, and the pumping rate can be adjusted until equilibrium is reached. Groundwater recovery data will be recorded on Attachment 1, Well Development Record.

Temperature, pH, specific conductivity, and other field parameters can be measured during development, but these measurements have no real effect on development. A turbidity meter should be used to determine the turbidity of the water. Typically, a turbidity reading of 50 nephelometric turbidity units (NTU) or lower is the goal for well development. The purpose of development is to remove fines from the well and produce clear water.

3.2 DECONTAMINATION

All equipment used for monitoring well development will be thoroughly decontaminated to minimize possible cross contamination of the well. Decontaminate equipment before use according to the methods outlined in HGL SOP 411.02, *Sampling Equipment Cleaning and Decontamination*.

3.3 MONITORING WELL DEVELOPMENT METHODS

Development of a groundwater well should not be started until 48 hours after the well has been grouted or as stated in the project-specific planning documents. Developing a groundwater monitoring well is best accomplished by alternately surging then pumping the well. This process agitates the finer-grain sediments and moves them into the well so that they may be removed. The use of nonformation water for development is not advised but may be necessary under certain conditions. Extreme care should be taken to avoid damaging the borehole, filter pack, and well screen. Each well will be considered developed when the groundwater turbidity has diminished to an acceptable level of clarity. Typically, a turbidity reading of 50 NTUs or under is acceptable to consider the well properly developed. Turbidity levels as low as 20 NTUs may be required for some projects. Stabilization of temperature, pH, and specific conductivity may also be required. Turbidity and water quality parameters should be recorded a minimum of every 15 minutes. Refer to the project-specific planning documents for any deviations.

3.3.1 Mechanical Surging

A surge block is a round plunger, slightly smaller in diameter than the inside diameter of the well screen. Development by mechanical surging produces good results in formations that have medium to high porosities and hydraulic conductivities. This development method is implemented as follows:

- Lower the surge block into the well to a point below the static water level.
- Raise and lower the tool alternately with increasing stroke lengths. As water begins to move easily both into and out of the screen, the surge block is lowered and the procedure resumed.
- Periodically use a bailer or pump to remove accumulated fines from the well. Development should begin at the static water level and move progressively downward to prevent the surge block from becoming sand locked. Surge blocks can be combined with tubing and a check valve to simultaneously surge and remove development water.

Note that surging of low-permeability formations can result in a collapsed screen, especially in wells that have plastic screens. Clayey and silty formations in which screen slot sizes are smaller than 0.015 inch are particularly prone to screen collapse. However, wells screened in fine-grain materials can be surged and pumped simultaneously with low-yield pumps that do not fit tightly against the inner surface of the well screen and are not likely to create a vacuum sufficient to cause collapse of the screen.

3.3.2 Bailer

A bailer heavy enough to sink through the groundwater and drilling fluid can be raised and lowered through the water column to produce an agitating action similar to that of a surge block. Frequently, the water in the well prior to development is very turbid and requires the use of a weighted bailer to

move through the water column. The bailer has the advantage of being able to remove turbid water and fines each time it is brought to the surface. The bailer method is ideal for formations with low permeability because it generally will not produce pressures great enough to cause well screen collapse. Bailing is generally not suitable for deep wells or wells that produce large volumes of water. Large steel or polyvinyl chloride (PVC) bailers attached to a drill rig wireline can be used to develop wells initially. However, the point on the wireline cable where the bailer is at the well bottom should be marked so as to avoid damaging the bottom cap of the well when lowering the bailer.

3.3.3 Pumping

Pumping is the simplest method of removing fines from the water-bearing formation, filter pack, and well screen. Pumping is performed at a rate higher than the recharge rate. Although this method is relatively simple, development action tends to take place in the most permeable zone or close to the top of the well screen. After the permeable zone has been developed, water tends to move preferentially through these zones. This results in the rest of the well being poorly developed and contributing only small volumes of water to the total yield. Pumping from low-permeability formations may compact the finer sediments around the borehole and restrict flow into the well screen. Wells completed in low-yield materials may be pumped using a low flow-rate pump, which can be used to both surge the well screen and remove fine-grain sediments. Surging and bailing should be performed before pumping because coarse material in the development water can damage the impellers in the pump if the pump is not specifically designed for development.

3.4 CONTAINMENT OF DEVELOPMENT WATER

All water generated during well development must be contained, stored, and managed as investigation-derived waste. Development water must be stored in approved containers or, in the case of development water containing nonvolatile constituents, lined impoundments may be used until water sample results are obtained and proper disposal methods are determined. Development water should be screened for volatile constituents using a PID. Generally, development water must be properly disposed of within 90 days of its generation. Proper storage and disposal methods for development water will be determined based on federal, state, and local regulations, and known or suspected contaminants.

4.0 REFERENCES

Driscoll, Fletcher G., 1986. *Groundwater and Wells*. Johnson Division, St. Paul, Minnesota.

Fetter, C. W., 1988. *Applied Hydrogeology*. Merrill Publishing Company. Columbus, Ohio. Second Edition.

U.S. Environmental Protection Agency (EPA), 1987. *Handbook: Ground Water*. Washington, D.C.

EPA, 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA*. Interim Final. Office of Emergency and Remedial Response, OSWER Directive 9355.3-01, EPA/540/G-89/004. Washington, D.C. October.

5.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
0	December 2010	Initial Release
1	July 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	November 20, 2019	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	June 23, 2021	Updated to incorporate client editorial comments.

ATTACHMENTS

Attachment 1 – Well Development Record

ATTACHMENT 1
WELL DEVELOPMENT RECORD

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WELL DEVELOPMENT RECORD

WELL/PIEZOMETER ID: _____

SHEET ___ of ___

PROJECT NAME: _____ PROJECT NO.: _____ DATE: _____

LOCATION: _____ DATE INSTALLED: _____

TOTAL DEPTH (FTOC): _____ CASING DIAMETER: _____

MEASURING POINT HEIGHT ABOVE/BELOW GROUND LEVEL: _____

METHODS OF DEVELOPMENT

o Swabbing o Bailing o Pumping o Describe _____

Equipment decontaminated prior to development o Yes o No

Describe: _____

EQUIPMENT NUMBERS:

pH Meter _____ EC Meter _____ Turbidity Meter _____ Thermometer _____

CASING VOLUME INFORMATION

Casing ID (inch)	1.0	1.5	2.0	2.2	3.0	4.0	4.3	5.0	6.0	7.0	8.0
Unit Casing Volume (A) (gal/ft)	0.04	0.09	0.16	0.2	0.37	0.65	0.75	1.0	1.5	2.0	2.6

PURGING INFORMATION:

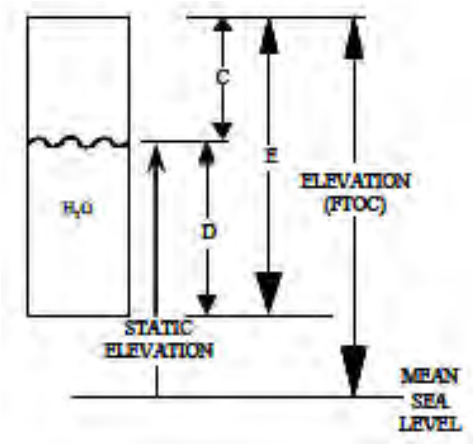
Measured Well Depth (B) _____ ft.

Measured Water Level Depth (C) _____ ft.

Length of Static Water Column (D) _____ - _____ = _____ ft.
(B) (C)

Casing Water Volume (E) + _____ × _____ = _____ gal
(A) (D)

Total Purge Volume = _____ (gal)



Date	Time	Water Level (FTOC)	Volume Removed (gal)	pH	EC	Temperature F or C	Turbidity/ Sand (ppm)	Type, Size, and Amount of Sediments Discharged During Purging

WELL DEVELOPMENT RECORD

Date	Time	Water Level (FTOC)	Volume Removed (gal)	pH	EC	Temperature F or C	Turbidity/ Sand (ppm)	Type, Size, and Amount of Sediments Discharged During Purging

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the standard method and equipment used to perform low-flow (minimal drawdown) groundwater sampling using dedicated or nondedicated low-flow pumping equipment. The general techniques described in this procedure are in general agreement with the procedures outlined in the U.S. Environmental Protection Agency (EPA) publication entitled *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures* (EPA, 1996).

2.0 SCOPE AND APPLICATION

Low-flow (minimal drawdown) groundwater sampling procedures are used to collect depth-specific samples and offer the following advantages:

- There is minimal disturbance of the water column during the purging and sampling procedure.
- The volume of purge water needed to achieve stabilization parameters is greatly reduced.
- There is less mixing of stagnant casing water with the formation water.
- Samples are representative of the mobile load of contaminants present.

3.0 EQUIPMENT AND SUPPLIES

The following equipment is required to perform low-flow groundwater sampling:

- Pump with the capability to produce consistent, low-flow rates ranging from 0.1 to 0.5 liter per minute (L/m). (Dedicated pumps should be equipped with ~~Teflo~~n® tubing to reduce the contamination of the tubing over time. Nondedicated pumps should use disposable one-time-use polyethylene tubing or ~~Teflo~~n® tubing if the tubing (without the pump) is dedicated to the well.);
- Pump controller;
- Portable air compressor or compressed gas;
- Graduated plastic beaker:
- Water level meter or interface probe;

- Multiparameter[1] water quality meter (with flow-through cell) and calibration solutions;
- Pull string or cable for lowering and retrieving submersible pumps (for example, bladder pumps) into and out of the well;
- Graduated 5-gallon buckets;
- Personal protective equipment (PPE), including nitrile gloves;
- 55-gallon drum or equivalent container to collect purge water;
- Fiberglass measuring tape (at least as long as the deepest well);
- Field logbook;
- Well completion information for each well to be sampled, including screen length and pump inlet depth for pre-installed pumps;
- Historical well purge rates/expected drawdown information for each well to be sampled; and
- Groundwater field sampling data sheet.

4.0 PROCEDURES

4.1 WELL INSPECTION/GAUGING

Before groundwater sampling begins, the following tasks are completed:

- Any water in the protective casing or in the vaults around the well casing is removed before venting and purging.
- Each time a casing cap is removed to measure water level or collect a sample, the air in the breathing zone and the air in the well casing are measured with the detector specified in the health and safety plan. Procedures specified in the health and safety plan must be followed when high concentrations of organic vapors or explosive gases are detected. Air monitoring data is recorded.
- The cap of a wells is removed before sampling to allow the well to "breathe." Water levels can change because of pressure changes between the surface and subsurface. This is typically experienced in confined or semiconfined aquifer conditions and is usually not an issue in unconfined aquifers. Remediation sites that use active remediation (for example, air sparging or soil vapor extraction) are especially subject to variations in surface and subsurface pressures. Two water levels should be measured within 1 minute

[1] This analyzer should contain oxidation-reduction potential (ORP, also known as Eh), pH, specific conductivity, temperature, and turbidity sensors. Turbidity may be measured using a separate nephelometer if needed.

of each other to verify level stability in the well. If a variation is noted, measurements should continue until the water level stabilizes.

- Wells should be gauged and sampled beginning with the least contaminated location and continuing to the most contaminated if contamination levels are known or can be anticipated. Typically, a "snapshot" of water level data is collected from all groundwater monitoring wells at a site. Other data requirements, such as vacuum/pressure, vapor concentration, and nonaqueous phase liquid (NAPL) (product) levels, are collected from wells during gauging. Discuss data needs with the project manager and the field team before beginning these activities.
- Wells are inspected for signs of tampering or other damage. If tampering is suspected (that is, a casing is damaged or a lock or cap is missing), this is recorded in the field logbook and reported to the field operations manager. Wells that display signs of tampering must not be sampled until the project manager (PM) has authorized it.
- All nondedicated purging and sampling equipment must be decontaminated.

If a nonconductive product layer is suspected in a well, well gauging is completed using an interface probe. All measurements should be to the nearest 0.01 foot. All these measuring devices must have a test button and a sensitivity setting knob. Before measurements are taken, each instrument is tested.

The following procedure is used to measure both product thicknesses and water levels:

- Water levels are measured from the notch located at the top of the well casing. If well casings are not notched, measurements are taken from the north edge of the top of the well casing, and a notch is made using a decontaminated metal file.
- For floating product, the product thickness is measured with an interface probe before the groundwater level is measured.
- The groundwater level is then measured with the interface probe, with care being taken not to disturb the sediment at the bottom of the well.
- For sinking product, the product thickness is measured after the water level. For sinking product, it may not be possible to avoid touching the bottom of the well with the probe; therefore, when measuring sunken product, the measurement should be performed slowly.
- Water level and product thickness measurements are recorded in the field logbook and on field sheets.
- All electronic water level measuring devices must be checked at least daily for functionality per the manufacturers' instructions.

If the total depth of a well from the top of the casing is known, the depth is confirmed **AFTER** sampling is completed. If the total depth of a well is not known, it is measured using a water level indicator or interface probe, and the data is recorded in the field logbook and/or on field sheets.

4.2 PUMP PLACEMENT

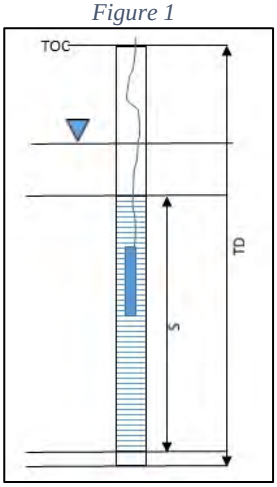
Pumps may be either dedicated or nondedicated. Initial water levels should be measured and recorded before nondedicated pumps are placed because the pumps will displace water in the water column. The elevation or depth of the inlet of the pump must be entered into the field logbook and/or on field sheets.

In many cases the elevation or depth of the pump inlet, prescribed in the project-specific planning documents, is the center of the saturated interval or center of the well screen. In other cases, the inlet elevation may already be given as a set depth. Both cases are described below.

The following information assumes that each well will be completed with a bottom well cap and that the screen will be attached directly to the bottom cap. If the wells are not completed in this manner, the PM should be consulted for modifications to the procedure.

Figure 1 shows some of the depth relationships discussed below. Field personnel must use the following procedures for positioning the pump inlet elevation in the field:

Figure 1



- If the water elevation is above the screen, the inlet of the pump is placed in the center of the screened interval. This elevation (in depth below top of casing [TOC] in feet) is as follows:

  The depth to the center of screen below the TOC in feet = (TD-0.33)-(S/2)

  Where:

  TD = Total well depth from TOC, and
  S = Screen Length.

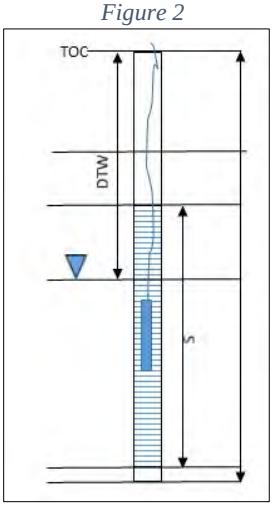
- If the water elevation is below the top of the screen, the inlet of the pump is placed in the center of the saturated screen interval. This elevation (in depth below TOC in feet) is as follows (Figure 2):

The depth to the center of saturated interval below the TOC in feet = (TD-0.33)-(TD-.33-DTW)/2

Where:

TD = Total well depth from TOC, and
DTW = Depth to water below TOC.

Figure 2



Tubing is attached to the nondedicated pump, and the pump is lowered slowly so that the inlet sits at the depth calculated.

4.3 PURGING

Water quality meters should be calibrated before first use and before use each day sampling is conducted. The monitoring wells should be purged and sampled in order of least contaminated to most contaminated. This practice helps reduce the potential for cross contamination between wells by sampling equipment.

After all discharge tubing has been attached to an in-line flow-through cell and the discharge from the cell has been directed to a drum or other purge water container, the purging procedure can begin.

Traditional low-flow purging is the preferred, generally accepted purging procedure. However, deviations are commonly required. Deviations MUST be discussed explicitly in the approved project-specific planning documents or be approved by the PM before they are employed. Table 1 lists some potential alternatives to traditional low-flow sampling.

Table 1
Purge Methods

Purge Process	Flow Rate	Drawdown	Parameter Criteria
Traditional Low Flow	0.1 to 0.5 L/m	<0.33 foot stable	Stabilize
Potential Alternatives (Not a Complete List)			
Modified Low Flow	0.05 to 0.1 L/m	<0.33 foot stable	Stabilize
Purge Dry	Not applicable	Not applicable	Not applicable
High Drawdown-Recover	Variable	Variable	Not applicable

4.3.1 Traditional Low-Flow Purging

4.3.1.1 Traditional Low-Flow Purging Procedures

Low-flow purging relies on both minimal drawdown (generally less than 0.33 foot) and the stabilization of groundwater parameters. Table 2 lists the stabilization criteria that must be achieved for three consecutive readings prior to sampling.

Table 2
Stabilization Parameters

Parameter	Stabilization Criteria
Temperature	± 0.5 °C
pH	± 0.1 units
Specific Conductivity	± 3 percent
Dissolved Oxygen (DO)	± 10 percent
ORP	± 10 millivolts (mV)
Turbidity	<50 nephelometric turbidity units (NTU) or ± 10 percent

The following procedure must be followed for traditional low-flow sampling:

- Commence pumping at a rate between 0.1 to 0.5 L/m, measuring flow rate with a graduated beaker and recording it on the field sheet.
- Inspect the discharge line and flow-through cell for air bubbles and remove air if found, then start recording stabilization parameters every 3 to 5 minutes.
- Continuously monitor drawdown with a water level indicator and record it on the field sheet.
- If drawdown exceeds 0.33 foot immediately, lower the pumping rate to allow the well to recover to a drawdown of not more than 0.33 foot.
- Once all parameters have stabilized for three consecutive readings and after purging has continued for a recommended minimum of 30 minutes, proceed with sampling as discussed in Section 4.4.

4.3.1.2 Traditional Low-Flow Purging Exceptions

4.3.1.2.1 Difficulty Obtaining Water Levels

When pump placement inhibits measuring the water level in the well, purge rates from previous sample events must not be exceeded, and the water discharge line must be monitored closely for air bubbles. If air bubbles are detected at any point during purging, the bladder pump must be shut down, and the validity of lowering the pump or adjusting the purge rate must be evaluated with direction from the PM.

4.3.1.2.2 Turbidity Not Stabilizing Below 50 NTU

Turbidity readings below 50 NTUs are desired. When turbidity is high, the purge time is extended to allow turbidity to reach a value below 50 NTUs. However, if turbidity stabilizes above 50 NTUs (± 10 percent) for 15 to 30 minutes, then turbidity is considered stable, and sampling for both filtered and unfiltered parameters may be needed as indicated in Section 4.4. Furthermore, wells that routinely have high turbidity may require redevelopment or replacement. The PM must be notified of high-turbidity instances so that options can be coordinated with the client.

4.3.1.2.3 Parameters Not Stabilizing

If the parameters do not stabilize, the following procedure is used:

- A subset of water quality parameters, including pH, specific conductivity, and turbidity or DO, is used as the stabilization criteria and noted in the field logbook and/or field sheets.
- Once stable, sampling can proceed as described in Section 4.4.

If the selected subset parameters do not stabilize, then the sample is collected only with the concurrence of the field operations manager **OR** when five well volumes have been removed from the well. This deviation must be recorded in the field logbook and/or on field sheets.

The well volume is defined as the volume of submerged casing and screen. One well volume can be calculated using the following equation:

$$V_w = HV_{ft}$$

Where:

V_w = Well volume (gallons),
H = Well depth minus depth to water (feet),
V_{ft} = Volume of 1-foot length of casing/screen (gallons/feet), and

$$V_{ft} = 7.481\pi\left(\frac{D}{2}\right)^2$$

and Where:

D = Inside diameter of casing/screen (feet).

4.3.1.2.4 Drawdown Equilibrating After Exceeding 0.33 Foot of Drawdown

EPA's guidance concedes that minimal drawdown (that is, less than 0.33 foot) "may be difficult to achieve under some circumstances due to geologic heterogeneities within the screened interval and may require adjustment based on site-specific conditions and personal experience."

Drawdown often exceeds 0.33 foot in formations with lower permeability; however, water levels may subsequently equilibrate after 0.33 foot of drawdown has been exceeded using traditional low-flow sampling techniques. Traditional low-flow sampling can be performed even if 0.33 foot of drawdown from the initial water level has been exceeded as long as the following criteria are met:

- The purge rate is no less than 0.1 L/m.
- The purge rate and the drawdown equilibrate at a level above the top of the sand pack. (This ensures water is not cascading within the screen interval.)
- Stabilization of all water quality parameters has been achieved for three consecutive readings.

4.3.2 Drawdown Greater than 0.33 Foot (Purge Dry Method)

If it is not possible to limit the drawdown to 0.33 foot and the exceptions in Section 4.3.1.2 are not applicable, then the low-flow technique cannot be applied. There are multiple variations of purging procedures that can applied with these very low yield wells. Variations can include lowering the pump and reattempting low-flow pumping, high drawdown, and purging dry. The purging dry procedure is discussed below; however, it should be verified that the project-specific planning documents explicitly state that the purging dry procedure is acceptable before use. When use of this procedure is not specifically approved in the project-specific planning documents, PM approval must be obtained before the procedure can be used.

The purging dry procedure, when authorized, employs the following steps:

- Lower the pump to the bottom of the well, but above any sediment at the bottom.
- Pumping rates may be maximized if there is no flow restriction. The objective is to remove all water from the well.
- Collect parameters and record them in the field logbook and/or on field sheets throughout pumping.
- Once the well is dry, cease pumping and allow the well to recover, typically overnight, and collect samples as described in Section 4.4. Generally, the water level should be allowed to recharge to least 80 percent of the initial water level before sampling.

4.4 SAMPLE COLLECTION

The following procedure is used for sample collection.

- Before sample collection, the discharge tubing should be disconnected from the flow-through cell. After the flow-through cell has been disconnected, head pressure on the

discharge tubing will be reduced and the flow rate may subsequently increase. It is important to maintain the purge rate used to achieve water quality parameter stabilization and drawdown equilibrium while sampling.

- All sample containers are properly labeled before sample collection.
- Samples are collected in the following order:
  - Volatile organic compounds (VOCs),
  - Semivolatile organic compounds, including polynuclear aromatic hydrocarbons,
  - Pesticides/polychlorinated biphenyls,
  - Metals, including mercury and cyanide,
  - Radiological parameters, and
  - Common anions.

4.4.1 Sample Container Filling Requirements

Water samples should be collected immediately after parameter stabilization using the same pump used for purging. Depending on the analyses to be performed, specific preservatives and filling/filtering requirements may be employed. These requirements must be followed as described in the project-specific planning documents and quality assurance plan.

VOCs have specific universal filling requirements, including the following:

- The VOC sample vial is filled using a slow, controlled pour down the side of a tilted sample vial until a meniscus is visible.
- The VOC vial is sealed immediately.
- When the container is capped, it is inverted and gently tapped to ensure that no air bubbles are present in the vial. If, after the initial filling, bubbles are present, the cap is removed, several drops of groundwater are added to the vial to again create a meniscus, and the process to ensure the absence of bubbles is repeated.
- After the VOC vials are sealed, sample degassing may cause bubbles to form: these bubbles must be left in the container.
- VOC sample vials should immediately be stored (inverted) on ice until receipt at the laboratory. These VOC samples must never be composited, homogenized, or filtered.

4.4.2 Sample Collection for High-Turbidity Locations

Filtered and unfiltered samples may be required should turbidity be high (greater than 50 NTU). Preparations for this possibility should occur prior to mobilization.

5.0 RECORDS

Record purging, sampling, and equipment calibration information in the field logbook. Complete the Groundwater Field Sampling Data Sheet (Attachment 1). Record information such as measurement times, parameter values, purge volume, and water levels. Record calibration details on the Equipment Calibration Log (Attachment 2).

6.0 QUALITY CONTROL

The project quality control officer is responsible for ensuring that all equipment is calibrated daily before use and recording the calibration results on the Equipment Calibration Log. The quality assurance coordinator is responsible for periodically reviewing these results.

7.0 REFERENCES

U.S. Environmental Protection Agency (EPA), 1996. Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures, EPA/540/S-95/504. April.

8.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
0	December 2010	Initial Release
1	March 2012	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	January 18, 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
3	January 29, 2019	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
4	December 20, 2019	Updated to address specific client comments.
4	June 23, 2021	Updated to incorporate client editorial comments.

ATTACHMENTS

Attachment 1 – Groundwater Field Sampling Data Sheet
Attachment 2 – Equipment Calibration Log

ATTACHMENT 1
GROUNDWATER FIELD SAMPLING DATA SHEET

ATTACHMENT 1
GROUNDWATER FIELD SAMPLING DATA SHEET

Page __ of ____

Well No.:		Location:	
Sampler(s):		Project Name:	
Well Depth (ft BTOC):		Project No.:	Date: Time:
DTW (ft BTOC):	DTP (ft BTOC):	Courier: _FedEx UPS Hand Other	
MP Ht. Above/Below Ground Surface:		Sample Collection Method: Low-Flow	
Condition of Bottom of Well:		Pump Type:	
Screen Interval (ft bgs):		Weather (sun/clear, overcast/rain, wind direction, ambient temperature):	
Well Diameter (in):			
Placement of Pump Inlet (ft bgs):			

Field Parameters

Time	Depth to Water (ft)	Flow Rate (L/m)	Total Volume (L)	pH	Temp. (°C)	Cond. (mS/cm)	ORP (mv)	DO (mg/L)	Turb. (NTU)	Type, Size, and Amount of Sediment Discharged

Observations

Color (clear, other [describe]):							
Odor:	None	Low	Medium	High	Very Strong	H2S	Fuel-like
Notes:							
Signed/Sampler(s):							

ATTACHMENT 2
EQUIPMENT CALIBRATION LOG

ATTACHMENT 2
EQUIPMENT CALIBRATION LOG

Project Name: _____

Project No. _____

Date/Time	Calibrated by	Instrument	Standard/ Manufacturer Lot No.	Standard Concentration	Instrument Reading	Comments

HydroGeoLogic, Inc — Exceeding Expectations	STANDARD OPERATING PROCEDURE	
	Approved by: Corporate Quality Manager	
Hand-Operated Auger Soil Sampling	**SOP No.: 403.02 (formerly 2.03)**	
	SOP Category: Environmental Services	
	Revision No.: 2	
	Revision Date: August 1, 2019	
	Review Date: August 2021	

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the standard method and equipment used to collect soil samples at the surface or in shallow subsurface using a hand auger.

2.0 SCOPE AND APPLICATION

This procedure yields a disturbed sample and applies to a wide variety of soil types including sands, clays, and silts. A hand auger is typically a small, lightweight metal cylinder (bucket), open at both ends with a cutting bit on the bottom. Diameters typically range between 1 and 4 inches. A T-shaped handle is attached to the top of the bucket by extendable rods. The augers are rotated into the ground until the bucket is full, then lifted out of the borehole and emptied. The maximum depth of hand auger investigations is typically 10 feet below ground surface. The use of an auger is of limited value in rocky soil. This procedure is not appropriate for collecting samples at a discrete depth, but may be used to collect samples at an approximate depth.

3.0 GENERAL REQUIREMENTS

All work must be performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan for relevant health and safety requirements.

Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager. Deviations from requirements must be sufficiently documented to re-create the modified process.

4.0 EQUIPMENT

The equipment required may include hand-operated, spiral-type, ship-type, open-tubular, orchard-barrel, open-spiral, closed-spiral, post-hole, clamshell, Edelman, or Iwan augers. Augers typically are used with 3- to 4-foot-long metal extension rods and T-handles (fixed or ratcheted). The use of stainless steel augers is preferred. Augers plated with chrome or coated with other materials, except Teflon®, cannot be used.

Sampling tools and equipment should be protected from contamination sources before sampling and decontaminated before and between sampling locations, as specified in SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.

5.0 PROCEDURES

1. Don clean gloves. Using a decontaminated stainless steel spoon or other approved utensil, remove surface vegetation and debris from the immediate area around the marked sampling point.
2. Do not allow sampling equipment to touch potentially contaminated surfaces.
3. Record the appropriate information and observations about the sample location in the field logbook.
4. Assemble the decontaminated auger, extension, and T-handle, if necessary, and advance the auger into the soil to the desired depth. Mark the length of the hand auger rods every 0.5 foot to determine auger head depth relative to the ground surface when advancing or tag the bottom of the borehole (if the borehole stays open) with a weighted tape measure or water level meter.
5. Withdraw the auger from the soil.
6. If a sample is not being collected, remove the soil from the auger bucket and repeat Steps 4 and 5. While removing the soil from the auger bucket, the subsurface lithology should be described as specified in SOP 403.07: *Geologic Borehole Logging*. If a sample is to be collected in the next depth interval, replace the auger bucket with a clean decontaminated bucket and repeat Steps 2 through 4. Change gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.
7. Perform any field monitoring required in the project-specific planning documents.

If collecting samples for analyses other than volatile organic compound (VOC) analyses, refer to Steps 8 and 9.

8. Using a decontaminated stainless steel spoon, spatula, disposable scoop, remove soil from the auger bucket and place in a stainless steel or glass container. Food-grade disposable aluminum pans may also be used but cannot be reused. Clean nitrile gloves may be donned to remove soil from the auger bucket by hand. Discard the top 2 or 3 inches of soil in the auger as this soil may consist of borehole slough from above. Mix or composite soil as directed by the project-specific planning documents. Using a decontaminated spoon or other approved utensil, remove any large rocks or other organic material (worms, grass, leaves, roots, etc.). Clean nitrile gloves may also be donned to remove large rocks or other organic material by hand.
9. Using a decontaminated stainless steel spoon, spatula, or disposable scoop, as appropriate, place soil samples in appropriate containers. Clean nitrile gloves may be donned to place soil into appropriate containers. Place samples in containers defined according to analytical needs specified in the project-specific planning documents, label samples, and then (when appropriate) pack on ice as soon as possible.

If collecting samples for VOC analysis, refer to Steps 10 and 11.

10. Remove the hand auger from the boring when the top of the specified sampling depth has been reached. Fit a slide-hammer to the top of the appropriate number of extension rods required to reach the total depth of the hole. Attach an impact sampler to the bottom of the extension rod(s) and drive the impact sampler into the soil to a depth of at least 6 inches. Remove the sampler from the borehole.
11. Collect VOC samples in accordance with SOP 403.01.0: *VOC Soil Sample Collection*. When samples are being collected for multiple analyses, samples that can be degraded by aeration (e.g., VOCs) are collected first and with the least disturbance possible to minimize analyte loss. VOC samples must not be composited.

6.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
0	December 2010	Initial Release
1	April 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	August 1, 2019	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	June 23, 2021	Updated to incorporate client editorial comments.

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HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE
	Approved by: Corporate Quality Manager
Surface and Shallow Depth Soil Sampling	**SOP No.: 403.06 (formerly 2.13)**
	SOP Category: Environmental Services
	Revision No.: 3
	Revision Date: June 24, 2020
	Review Date: June 2022

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the equipment and operations used for sampling surface and shallow depth soils. This procedure outlines the methods for soil sampling with routine field operations on environmental projects.

2.0 SCOPE AND APPLICATIONS

The objective of surface and shallow depth soil sampling is to ascertain the nature and extent of soil contamination at a site. The data can be used to identify contaminant sources, evaluate potential threats to human health or the environment, evaluate potential exposure pathways, or calculate environmental risks. For the purposes of this SOP, soil is defined as all unconsolidated materials above bedrock; surface soils are those that occur 0 to 6 inches below ground surface; and shallow depth soils are soils located above the bedrock surface and from 6 inches to 2 feet below ground surface.

3.0 GENERAL REQUIREMENTS

All work is performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan for relevant health and safety requirements.

Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager and discussed in the approved project plans. Deviations from requirements must be documented sufficiently to re-create the modified process.

4.0 PROCEDURES

4.1 SAMPLING EQUIPMENT

Typically, equipment required for surface and shallow depth soil should be specified in the project field sampling plan or work plan. Equipment includes the following:

- Stainless steel mixing bowl,
- Stainless steel trowels or spoons,
- Stainless steel hand auger,
- Stainless steel core sampler that uses stainless steel or Lexan® liners (optional),
- Stainless steel shovel, and
- Appropriate sample containers.

Disposable sampling equipment items, such as a sampling spoon, may be used instead of stainless steel equipment. An example of a hand auger is provided in Attachment 1.

4.2 DECONTAMINATION

Before initial use, and after each subsequent use, all nondedicated or nondisposable sampling equipment must be decontaminated using the procedures outlined in HGL SOP 411.02: *Sampling Equipment Cleaning and Decontamination.*

4.3 SAMPLING LOCATION/SITE SELECTION

Follow the sample design criteria outlined in the project plan for each sampling event. Relocate the sample sites when conditions dictate, such as when natural or artificial obstructions are present at the proposed sample location (such as boulders or asphalt). Document the actual sample locations on a topographic map or site sketch and photograph all sample locations. GPS coordinates for the new location may also need to be recorded.

4.4 GENERAL

All boreholes and pits are filled in with the material removed during sampling unless otherwise specified in the project-specific planning documents. Where a vegetative turf has been established, fill in with native soil or potting soil and replace the turf if practical in all holes or trenches when sampling is completed.

4.4.1 Homogenizing Samples

Homogenizing is the mixing of a sample to provide a uniform distribution of the contaminants. Proper homogenization ensures that the containerized samples are representative of the total soil sample collected. All samples to be composited or split should be homogenized after all aliquots have been combined. **Do not homogenize (mix or stir) samples for volatile compound analysis. Follow the procedures outlined in HGL SOP 403.01: *VOC Soil Sample Collection* for collection of such samples.**

4.4.2 Compositing Samples

Compositing is the process of physically combining and homogenizing several individual soil aliquots of the same volume or weight. Compositing samples provide an average concentration of contaminants over a certain number of sampling points. Refer to HGL SOP 403.03: *Soil or Sediment Sample Compositing*.

4.4.3 Splitting Samples

Splitting samples is performed when multiple portions of the same samples must be analyzed separately. After preparation, fill the sample containers for the same analyses one after another in

a consistent manner (parent sample for semivolatile organic compounds [SVOCs] analysis, then split sample for SVOC analysis; parent sample for total metals analysis, then split sample for total metals analysis; and so forth).

4.5 SURFACE SOIL SAMPLING

Perform the following steps for surface soil sampling:

- Before sampling, remove leaves, grass, and surface debris from the area using a decontaminated stainless steel trowel or disposable sampling spoon.
- Label the lid of the sample container with an indelible pen or affix the sample label to the side of the jar. Tape over the label to seal out dirt and water before filling the container with soil, if possible.
- Collect surface soil samples with a decontaminated stainless steel trowel, spoon, or hand auger and transfer them to a decontaminated stainless steel bowl for homogenizing. If VOC analyses are to be conducted, collect the VOC sample first following the procedures outlined in HGL SOP 403.01: *VOC Soil Sample Collection*, then transfer the appropriate aliquot of soil to the decontaminated stainless steel bowl for homogenizing.
- Collect samples in the order of volatilization sensitivity. The most common collection order is as follows:
    - VOC,
    - Purgeable organic carbon,
    - Purgeable organic halogens,
    - Total organic halogens,
    - Total organic carbon,
    - Extractable organics,
    - Total metals,
    - Phenols,
    - Cyanide, and
    - Radionuclides.
- Immediately transfer the sample into a container appropriate to the analysis being performed.
- Place the samples in a cooler with ice. The temperature in the cooler must be maintained at approximately 4ºC (if appropriate for analyses) for transport to an analytical laboratory.
- Material removed to collect the samples is returned to the boreholes and pits. Excess soil sample media should be treated as investigation-derived waste (IDW) and managed in accordance with the project-specific planning documents.
- Decontaminate all sampling equipment following HGL SOP 411.02, *Sampling Equipment Cleaning and Decontamination*.

4.6 SURFACE SOIL SAMPLING (COMPOSITE SAMPLES ONLY)

Perform the following steps for surface soil (composite) sampling:

- Before sampling, remove leaves, grass, and surface debris from the area using a decontaminated stainless steel trowel.
- Collect surface soil aliquots with a decontaminated stainless steel spoon, trowel, or hand auger and place them in a stainless steel bowl and homogenize. Homogenize the sample in accordance with HGL SOP 403.03: *Soil or Sediment Sample Compositing*. Follow the procedures outlined in HGL SOP 403.01: *VOC Soil Sample Collection*, for samples collected for VOC analysis.
- Label the sample container and place it in a cooler chilled to 4ºC . Complete the chain of custody record and pack it in the sample cooler.
- Material removed to collect the samples is returned to the boreholes and pits. Excess soil sample media IDW should be managed in accordance with the project-specific planning documents.
- Decontaminate all nondedicated sampling equipment following HGL SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.

4.7 SHALLOW DEPTH SOIL SAMPLING

Perform the following steps to collect shallow depth soil samples:

- Use a decontaminated stainless steel shovel to remove the top layer of soil and leaves, grass, and surface debris.
- Excavate soil to the pre-determined sampling depth using a decontaminated hand auger. Periodically remove the cuttings from the auger.
- When the proper sample depth is reached, remove the hand auger and all cuttings from the hole.
- Lower the decontaminated core sampler or hand auger to the bottom of the hole. When using a core sampler, it must contain a decontaminated liner appropriate for the constituents to be analyzed.
- Mark the sample interval on the hammer stem or auger.
- Operate the slide hammer on the core sampler to drive the sampler head into the soil, or advance the auger until it is flush with the interval mark at ground level.
- Record weight of hammer, length of slide, blow counts, and geologic soil data for all samples collected with a core sampler in the field logbook as outlined in HGL SOP

300.04: *Field Logbook Use and Maintenance*. This information may also be entered on Attachment 2, Surface and Shallow Soil Sampling Log.

- When the core sampler liner or auger has been advanced to the total depth of the required sample, remove it from the bottom of the hole.
- Immediately remove the liner from the core sampler and transfer the sample into a container or stainless steel bowl appropriate to the analysis being performed and then composite and homogenize it in accordance with HGL SOP 403.03: *Soil or Sediment Sample Compositing*. For VOC analysis follow the sample procedures outlined in HGL SOP 403.01: *VOC Soil Sample Collection.*
- Label the sample container and place it in a cooler chilled to 4ºC . Complete the chain of custody record and pack it in the sample cooler.
- Material removed to collect the samples is returned to the boreholes and pits. Excess soil sample media IDW should be managed in accordance with the project-specific planning documents.
- Decontaminate all sampling nondedicated equipment following HGL SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.

4.8 ABANDONMENT PROCEDURES

Abandon boreholes and fill them to grade with the material removed for sampling, if approved, or clean fill.

5.0 DOCUMENTATION

Record applicable sampling information in the field logbook as outlined in HGL SOP 300.04: *Field Logbook Use and Maintenance*. This information can also be entered on Attachment 2, Surface and Shallow Soil Sampling Log.

The project manager or an approved designee checks all field sheets and field logbooks used to record information during sampling for completeness and accuracy as soon as possible after the sampling event. Any discrepancies are noted, and the documents are returned to the originator for correction. The reviewer acknowledges that these review comments have been incorporated by signing and dating the "checked by" and "date" blanks on the field sheets and at the applicable places in the logbook.

6.0 REVISION HISTORY

Revision 0	July 2010	Initial Release
Revision 1	July 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	February 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	June 24, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 2.13 to 403.06.

ATTACHMENTS

Attachment 1 – Example of Hand Auger and Core Sampler
Attachment 2 – Surface and Shallow Soil Sampling Log

ATTACHMENT 1
EXAMPLE OF HAND AUGER AND CORE SAMPLER

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Idaho 83211

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208.226.2017
fax: 208.226.7280
ams@ams-samplers.com
www.ams-samplers.com

The world's finest sampling equipment.

Basic Soil Sampling Kit - 5/8" Threaded

DESCRIPTION:
Hand auger kit includes a Standard type Regular, Mud and Sand Auger plus an AMS Core Sampler* with slide hammer. Included accessories are three 4 foot (1.2m) extensions, cross handle, cleaning brush, 2 crescent wrenches and slip wrench all contained in an AMS Deluxe storage and transport case. Two sizes of kit are available, 3 1/4 inch (8.3 cm) augers with 2 inch (5.1 cm) Core Sampler and 2 1/4 inch (5.7 cm) augers with 1 1/2" Core Sampler. Quick connect is not available with this kit.

APPLICATION:
Use of the augers for accessing the sampling point at depths of up to about 12 feet (3.6 m) with the supplied extensions and AMS slide hammer. The sample may be collected within a removable retaining cylinder (liner). Plastic end caps are included.

FEATURES
AMS Soil augers are designed to rapidly remove soils of all types, using the specially designed bits on the Regular, Mud, and Sand models. The auger tips are tungsten carbide hard surfaced and heat treated before sharpening. The core sampler features a heat treated coring tip on the cylinder and a threaded end cap. All attatchment couplings are 5/8 NC threaded.

BENEFITS
For your convenience, all the items necessary for accessing a sampling point and then taking a sample are included. AMS soil buckets are the most efficient available in terms of effort required and speed. The AMS Core Sampler allows immediate core examination or a sample may be collected in a retaining cylinder for later use.

USE:
Assemble the chosen soil auger with an extension and cross handle. Place at the desired angle on the soil surface and turn three revolutions, or until full. Lift carefully from the hole and empty from the bail by tapping the cross handle on the ground. Repeat until the sampling depth is reached. Assemble core sampler to an extension(s) and slide hammer. Place in the hole and mark the extension six inches (5.1 m) above the soil surface. Use the slide hammer to drive in the the sampler to the mark and carefully remove. Disassemble, remove the liner and place the cap on each end.

HELPFUL HINTS:
Use plumbers wick on 5/8 inch male threads used with Slide Hammer to help threads stay tight. Keep all fittings and samplers clean, dry and free of dirt or Mud. You can clean tooling with soapy water. Always dry to prevent rusting. Use a wire brush on male threads. Use vegetable oil on tools to prevent fittings locking up and rusting. When using augers, use rubber O-rings on male 5/8 inch thread to help take apart.

SPECIFICATIONS:
AMS Soil Auger Kits are manufactured by AMS from all USA made materials. See separate AMS Technical Data Sheets for details on the Regular, Mud, Sand & Soil Augers, Core Sampler, Extensions, Cross Handles, Slide Hammer, and Liners. Crescent wrenches are made from chrome plated forged steel. The cleaning brush is made with nylon bristles, with a twisted wire handle. The AMS Deluxe Case is molded from glass reinforced plastic with a lid gasket and lockable hasps.

Kit Composed of the Following Items

Item	Size	Part #	Size	Part#
1- Regular Auger	3 1/4"	400.06	2 1/4"	400.08
1- Mud Auger	3 1/4"	400.18	2 1/4"	400.20
1- Sand Auger	3 1/4"	400.40	2 1/4"	400.42
1- Cross Handle		406.04		406.04
3- Thrd. Extensions	4'	408.03	4'	408.03
1- Core Sampler*	2" x 6"	404.10	1 1/2" x 6	404.38
* w/slip wrench, liner & caps				
1- Slide Hammer		400.99		400.99
1- AMS Nylon Brush	2"	430.07	1 1/2"	430.11
2- Crescent Wrenches 421.10			421.10	
1- Slip Wrench		421.29		421.29
1- AMS Deluxe Case		430.01		430.01

* Patent Pending, USA & Foreign

ANCILLARY ITEMS:
AMS Extensions, Liners, End Caps, End Cap Inserts, Sieves, Soil Color Charts, and Sample Containers.

Basic Soil Sampling Kit

Basic Soil Sampling Kit	
Size	Basic Kit Regular
2 1/4"	209.53
3 1/4"	209.51

Sampling Equipment
PowerProbe
Well Management
Pest Control
PowerCore

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ATTACHMENT 2
SURFACE AND SHALLOW SOIL SAMPLING LOG

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HGL HydroGeoLogic, Inc. Exceeding Expectations

Surface and Shallow Soil Sampling Log Records Management Data

Project Number	Project Name	Page ____ of ____

General Info			
	Location		
	Surface Elevation ft.	Date Started	Date Completed
	Field Investigator		C of Cr
	Sampling Excavation Method	Sampling Method	
	Depth of Excavation ft.	Depth Water First Encountered ft.	Backfill Material

Sampling Info	Sample Number	Depth (ft)	Lithologic Description[1]	Sample Container	Analyses Requested

Plan View		Legend
		Soil Sampling Location

Recorded By:	Date	Checked By:	Date:

[1] Include such data as OVM, pH, blow counts, or other physical reading observations.

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HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE
	Approved by: Corporate Quality Manager

Sediment Sampling	SOP No.: 403.08 (formerly 2.15)
	SOP Category: Environmental Services
	Revision No.: 2
	Revision Date: March 25, 2020
	Review Date: March 2022

1.0 PURPOSE

This standard operating procedure (SOP) establishes the guidelines for sediment sampling using a variety of sampling devices. Methods for preventing sample and equipment cross-contamination are included. Proper sediment sampling ensures that any evaluations of sediment contamination are based on actual contaminant levels and are not based on improper sampling techniques.

This SOP provides guidance for routine field operations on environmental projects. Site-specific deviations from the methods presented herein must be approved by the HGL project manager.

2.0 SCOPE AND APPLICATIONS

Field personnel collecting sediment samples are responsible for performing the applicable tasks outlined in this procedure when conducting work related to environmental projects.

The project manager or an approved designee is responsible for checking all work performed and verifying that the work satisfies the applicable tasks required by this procedure. This verification will be accomplished by reviewing all documents and data produced during work performance.

3.0 GENERAL REQUIREMENTS

All work will be performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan for relevant health and safety requirements.

Any deviations from specified requirements will be justified to and authorized by the project manager and/or the relevant program manager and documented in the approved project plans. Deviations from requirements will be sufficiently documented to re-create the modified process.

4.0 SAMPLING EQUIPMENT AND TECHNIQUES

Sediment samples may be obtained using on-shore or off-shore techniques. Sediment sampling equipment and techniques must be designed to minimize the risk of dilution or loss of material as the sample is moved through the water column. Sediment sampling devices are described below.

4.1 DIP SAMPLERS

A dip sampler consists of a pole with a jar or scoop attached. The pole may be made of bamboo, wood, ~~Teflon~~®, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is attached by a clamp.

The dip sampler is operated by submerging the jar or scoop and pulling it through the sediments to be sampled. The samples retrieved are then transferred into the appropriate sample container after decanting the liquid. Further decanting can occur while the sample is present in the sample jar. Avoid contact with sampler's gloves. Transferring the sample may require the use of a stainless steel or ~~Teflon~~® spoon/spatula.

4.2 HAND-OPERATED CORE SAMPLERS

Hand-operated sediment core samplers are used to obtain sediment samples in shallow water (less than 3 feet). These samplers operate in a manner similar to soil core samplers. However, because of the saturated conditions of most sediments, provisions must be made to retain the sample within the core. Core samplers are generally constructed of a rigid metal outer tube into which a 2-inch plastic core sleeve fits with minimum clearance. The cutting edge of the core sampler has a recessed lip on which the plastic sleeve rests and that can accommodate a core retainer. This retainer is oriented such that when the sampler is pressed into the sediment, the core is free to move past the retainer. Due to construction of the retainer, the core will not fall through the retainer upon removal of the sampler from the sediment. Some core samplers are also equipped with a butterfly valve below the core barrel that helps retain the material when the sampler is removed from the sediment.

After the sampler has been removed from the sediment, the plastic sleeve is removed. The sediment is removed from the sleeve and placed in the appropriate sample container. Chlorinated organics will not be collected using core samplers because core sleeves and retainers are generally made of plastic. The hand-operated core sampler will not be useful for obtaining samples of gravelly, stony, or consolidated sediments. Examples of hand-operated core samplers are referenced in Attachment 1.

4.3 GRAVITY CORE SAMPLERS

Gravity core samplers are used to obtain sediment samples in water bodies or lagoons with depths greater than 3 to 5 feet. These types of samplers can be used for collecting 1- to 2-foot cores of surface sediments at depths of up to 100 feet beneath the water surface.

As with all core-type samplers, gravity core samplers are not suitable for obtaining samples of coarse, gravelly, stony, or consolidated deposits. They are, however, useful for fine-grained inorganic sediment sampling.

The gravity core sampler operates in a manner similar to the hand-operated core in that a 2-inch plastic sleeve fits within a metal core housing fitted with a cutting edge. Plastic nests are used to retain the core within the plastic sleeve. An opening exists above the core sleeve to allow free flow of water into and through the core as it moves vertically downward to the sediment. The sampler has a field personnel-operated, messenger-activated valve assembly that seals the opening above the plastic sleeve following sediment penetration. This valve is activated by the messenger, creating a partial vacuum to assist in sample retention during retrieval.

Samples are obtained by allowing the sampler, which is attached to approximately 100 feet of stainless steel aircraft cable, to drop to the benthic deposits. The weight of the sampler drives the core into the sediment to varying depths depending on the characteristics of the sediments. The messenger is then dropped by field personnel on the taut aircraft cable to seal the opening above the plastic sleeve. The sampler is then carefully retrieved.

Upon retrieval of the sampler, the plastic core sleeve is removed and the sample is placed in the appropriate sample container. Care should be exercised in labeling to properly identify sample orientation. Examples of gravity core samplers are referenced in Attachment 2.

4.4 DREDGES

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices or when large quantities of materials are required. Various dredge designs are available for sampling in deep or turbulent waters and for obtaining samples from gravelly, stony, or dense deposits.

Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a messenger. Dredges are commonly quite heavy and may require use of a winch and crane assembly for sample retrieval.

Upon retrieval of the dredge, the sample can either be sieved or transferred directly to a sample container for labeling and storage. Examples of dredge types that could be used for sampling include Ponar, Petersen, and Ekman dredges, which are referenced in Attachment 3.

4.5 HAND AUGERS

Sediment samples may be collected using a hand auger. When using a hand auger, provisions must be made to ensure that sediment samples remain in the auger. Hand augers are best utilized when sampling non-subaqueous sediments. Additional information on hand augers can be found in SOP 403.06: *Surface and Shallow Depth Soil Sampling.*

5.0 PROCEDURES

5.1 SAMPLING SEDIMENT WITH NO OVERLYING SURFACE WATER

Sediment samples obtained from areas with no overlying surface water will be collected in accordance with the following procedures:

- Record all data in the field logbooks in accordance with SOP 300.04: *Field Logbook Use and Maintenance.*

- Insert a decontaminated ~~Teflon~~® or stainless steel spoon, scoop, or trowel into the sediment to the desired depth and remove the collected sample, or rotate and push down a decontaminated hand auger into the sediment to the desired depth and remove the collected sample. A disposable scoop may be used for specified media and analytical parameters in accordance with the site-specific project plans.
- Collect samples for volatile organic compounds (VOC) analyses, if applicable, from the sampling device or from unmixed sediment placed into a stainless steel bowl in accordance with SOP 403.01: *VOC Soil Sample Collection*.
- Place the sample in a decontaminated stainless steel bowl. Stir the sample thoroughly (non-VOC samples only) with a decontaminated stainless steel spoon or spatula—or with a dedicated disposable scoop—to provide a homogeneous mixture before filling sampling containers.
- Follow the guidelines in the site-specific project plans and Quality Assurance Project Plan (QAPP) for aliquot size (mass), container type, storage conditions, and holding times. [Note: When sampling in coarse materials, such as gravel, discretion must be used to limit inclusion of large sediment particles. As the analysis of sediments performed by the laboratory is typically restricted to particles less than 2 millimeters in size, care must be taken to ensure that there is sufficient sample volume consisting of particles smaller than 2 millimeters. As a general rule, particles larger than 0.5 inch (12.7 millimeters) in size should be excluded unless a grain size analysis is planned.] Fill the appropriate sample containers as detailed in the site-specific project plans. Identify or label samples carefully and clearly, addressing all the categories or parameters.
- Label the sample containers and place the filled sample containers on ice immediately.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*, after use and between sampling if dedicated disposable scoops are not used. Don new clean gloves before beginning sampling activities and at each sampling point.
- Complete all chain of custody documents and record information in the Field Sampling Report (Attachment 4) and the field logbook (see the project-specific QAPP for sample custody procedures).

5.2 SHALLOW STREAM SEDIMENT SAMPLING

Stream sediment sampling within shallow (less than 2 feet) water will be conducted in accordance with the following procedures. Note that if co-located surface water samples are being collected, the surface water sample should be collected first.

- Collect the sample in an area of sediment accumulation, such as the inside of stream meanders, quiet shallow areas, and low-velocity zones. Avoid areas of net erosion, such as high-velocity, turbulent flow zones.

- If possible, collect the sample while remaining on the stream bank. If the sample cannot be obtained from the bank, enter the stream from a point downstream of the sediment sampling location. Consult the site health and safety plan before entering the river to avoid potential hazards. Collect the sediment sample by reaching into the stream with a decontaminated stainless steel spoon or ~~Teflon~~® scoop and scooping a sample in an upstream direction. Attempt to minimize the loss of fine material. A disposable scoop may be used for specified media and analytical parameters, in accordance with the site-specific project plans.
- Collect samples for VOC analyses, if applicable, from the sampling device or from unmixed sediment placed into a stainless steel bowl in accordance with SOP 403.01: *VOC Soil Sample Collection*.
- Place sample in a stainless steel bowl and gently mix with a stainless steel spoon or dedicated disposable scoop (non-VOC samples only). Transfer the sediment samples to the appropriate sample containers using the stainless steel spoon or dedicated disposable scoop. Do not mix samples for volatile organic analyses.
- Follow the guidelines in the site-specific project plans and QAPP for aliquot size (mass), container type, storage conditions, and holding times. See note under Section 5.1 for sampling coarse materials. Fill the appropriate sample containers as detailed in the site-specific project plans. Identify or label samples carefully and clearly, addressing all the categories or parameters.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*, after use and between sampling if dedicated disposable scoops are not used. Don new clean gloves before beginning sampling activities and at each sampling point.
- Complete all chain of custody documents and record information in the Field Sampling Report (Attachment 4) and the field logbook (see the project-specific QAPP for sample custody procedures).

5.3 SUBAQUEOUS SEDIMENT SAMPLING

Subaqueous sediment sampling from lakes, ponds, lagoons, and surface impoundments will consist of the following:

- Select the most appropriate sediment sampling device (as described in Section 4.0).
- Decontaminate all sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- If sampling from a boat equipped with an engine, attempt to collect the sample with the boat engine off or attempt to ensure that all exhaust fumes are directed away from the sample collection area until the sample has been collected.

- Lower the sampler at a controlled descent of approximately 1 foot per second until the sampler reaches the sediment surface, as indicated by a slackening of the cable. Release the weighted messenger, if applicable, to engage the closing mechanism of the dredge. Slowly retrieve the sampler and raise it at a controlled speed. When the sampler is at the water surface, attach a tag line(s) to steady and pull the sampler back into the boat. If large samplers are used, a motorized winch may be required for retrieval.
- Open and tie back any vent flaps on the sampler and carefully siphon off any overlying water, disposing of it over the side of the boat.
- Visually inspect the sample for acceptability (for example, determine if an undisturbed surface layer is evident, the overlying water is not excessively turbid, and adequate penetration is achieved). If the sample is not acceptable, discard it and collect another sample from an adjacent and upstream location.
- Carefully extrude the sediment from the sampler by slowly lifting on the winch cable and sliding the sample out the bottom of the sampler. If using core liners, remove the front face of the core liner to expose the side of the core.
- Visually inspect the side of the sample to identify any obvious stratification (such as different sediment types, sizes, or colors). If no patterns are evident, collect a sample from the surface and mid-core depth. During some investigations, it may be necessary to collect separate samples from the surface and mid-core depths. This may best be accomplished by gently scraping the side of the core with a decontaminated stainless steel scraper or knife. Scrape from the bottom to the top of the core only. If the sediment is unconsolidated, do not scrape.
- Remove the upper 2 centimeters of the sample using a decontaminated ~~Teflon~~® or stainless steel scoop—or dedicated disposable scoop—and place it in the sample container. From an undisturbed area of the sample surface, scoop a 2-centimeter sample only if grain size analysis is required. After grain size analysis samples are collected, scrape off the upper sediment layer and discard it overboard. Collect samples from the mid-section of the sediment. Sediment must be removed with caution to avoid cross-contaminating the sample (that is, from exposure to engine exhaust, rust, or grease).
- Do not include nonrepresentative materials, such as twigs or debris, in the sample. Do not include sediments that have come into contact with the side of the sampler or core liner for analysis.
- Follow the guidelines in the site-specific project plans and QAPP for aliquot size (mass), container type, storage conditions, and holding times. Fill the appropriate sample containers as detailed in the site-specific project plans. Identify or label samples carefully and clearly, addressing all the categories or parameters;
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination* after use and between sampling if dedicated

disposable scoops are not used. Don new clean gloves before beginning sampling activities and at each sampling point.

- Complete all chain of custody documents and record information in the Field Sampling Report (Attachment 4) and the field logbook (see the project-specific QAPP for sample custody procedures).

6.0 RECORDS

Documentation generated as a result of this procedure is collected and maintained in accordance with requirements detailed in the project-specific planning documents. The field logbook will be completed in accordance with procedures listed in SOP 300.04: *Field Logbook Use and Maintenance.* A Field Sampling Report will be filled out for each sediment sample collected (Attachment 4).

7.0 REVISION HISTORY

Revision 0	December 2010	Initial Release
Revision 1	August 11, 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	February 25, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 2.15 to 403.08.

ATTACHMENTS

Attachment 1 – Core Sampler
Attachment 2 – Gravity Core Sampler
Attachment 3 – Dredges
Attachment 4 – Field Sampling Report

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ATTACHMENT 1
CORE SAMPLER

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CORE SAMPLER

AMS Core Sampler (http://www.ams-samplers.com/hand-tooling/sludge-and-sediment-samplers/sludge-and-sediment-samplers/sludge-and-sediment-samplers.html)

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ATTACHMENT 2
GRAVITY CORE SAMPLER

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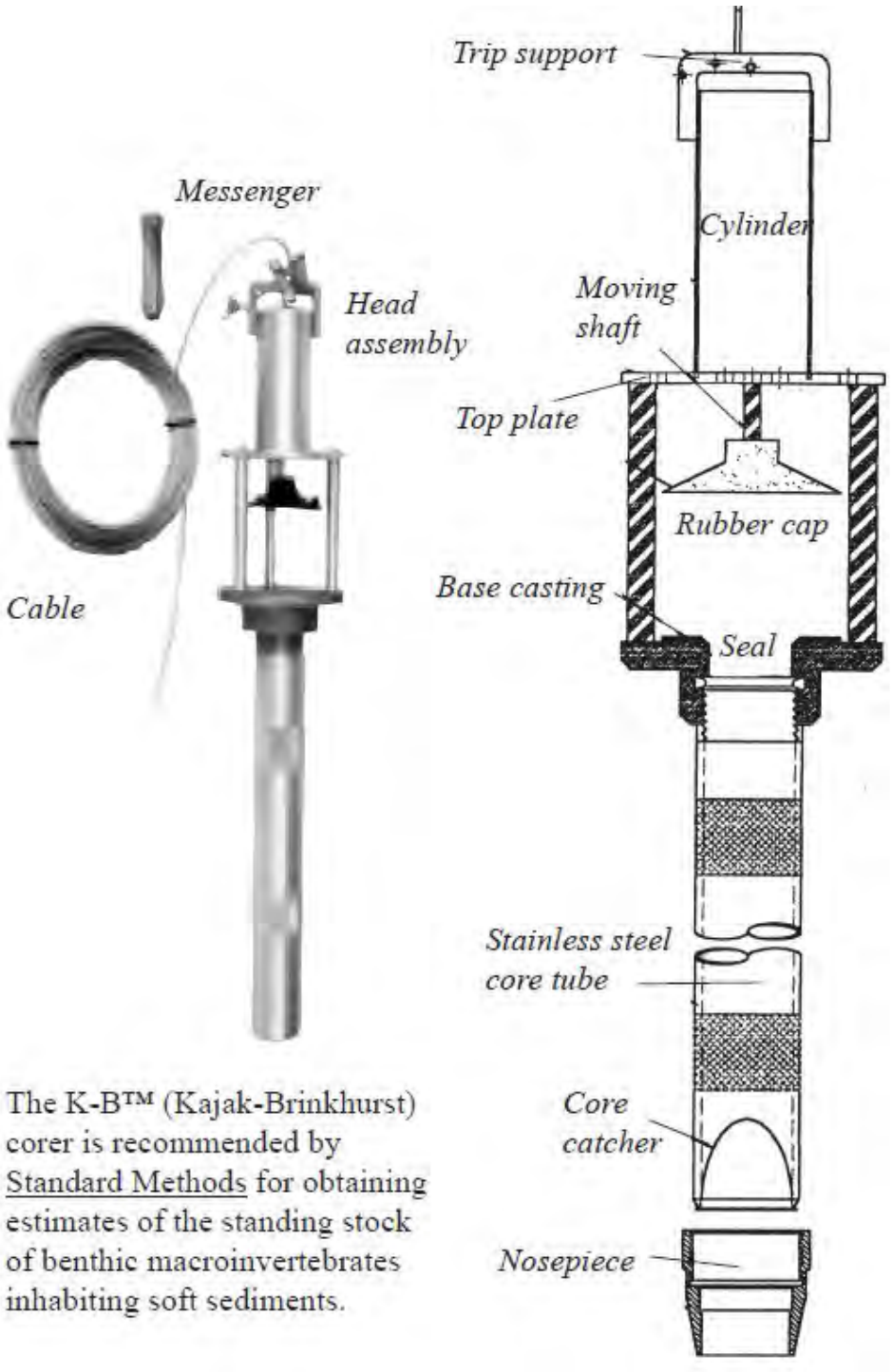
K-B GRAVITY CORER



The K-B™ (Kajak-Brinkhurst) corer is recommended by Standard Methods for obtaining estimates of the standing stock of benthic macroinvertebrates inhabiting soft sediments.

Wildco K-B Corer (http://shop.sciencefirst.com/wildco/k-b-corers/7815-k-b-corer.html)

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ATTACHMENT 3
DREDGES

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PONAR

WILDCO Ponar Dredge (http://www.benmeadows.com/wildco-ponar-grabs_36816477/)

PETERSON

WILDCO Peterson Dredge (https://www.coleparmer.com/p/mn/7270)

EKMAN

EKMAN Dredge (http://www.benmeadows.com/ekman-bottom-grab-sampler_36816471/?searchterm=ekman%2bdredge)

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ATTACHMENT 4
FIELD SAMPLING REPORT

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FIELD SAMPLING REPORT

LOCATION: _____ PROJECT : _____

SITE: _____

SAMPLE INFORMATION

MATRIX _____ SAMPLE ID: _____

SAMPLING METHOD _____ DUP./REP. OF : _____

BEGINNING DEPTH _____ MATRIX SPIKE/MATRIX SPIKE DUPLICATE
YES ( ) NO ( )

END DEPTH _____

GRAB ( ) COMPOSITE ( ) DATE: _____ TIME: _____

CONTAINER		PRESERVATIVE/ PREPARATION	EXTRACTION METHOD	ANALYTICAL METHOD	ANALYSIS
SIZE/TYPE	#				

NOTABLE OBSERVATIONS

PID READINGS	SAMPLE CHARACTERISTICS	MISCELLANEOUS
1st	COLOR:	
2nd	ODOR:	
	OTHER:	

pH _____ Temperature _____ Dissolved oxygen _____ Specific Conductivity _____

GENERAL INFORMATION

WEATHER: SUN/CLEAR _____ OVERCAST/RAIN _____ WIND DRIECTION _____ AMBIENT TEMP _____

SHIPMENT VIA: FEDEX _____ HAND DELIVER _____ COURIER _____ OTHER _____

SHIPPED TO: _____

COMMENTS: _____

SAMPLER: _____ OBSERVER: _____

MATRIX TYPE CODES

DC=DRILL CUTTINGS
WG=GROUND WATER
LH=HAZARDOUS LIQUID WASTE
SH=HAZARDOUS SOLID WASTE
SE=SEDIMENT
SL=SLUDGE
SO=SOIL
GS=SOIL GAS
WS=SURFACE WATER
SW=SWAP\WIPE

SAMPLING METHOD CODES

B=BAILER
BR=BRASS RING
CS=COMPOSITE SAMPLE
C=CONTINUOUS FLIGHT AUGER
DT=DRIVEN TUBE
W=SWAB\WIPE
G=GRAB
HA=HAND AUGER
H=HOLLOW STEM AUGER
HP=HYDRO PUNCH
SS=SPLIT SPOON
SP=SUBMERSIBLE PUMP

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1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the methods for surface water sampling. It describes the procedures and equipment to be used to obtain representative surface water samples capable of producing accurate quantification of water quality.

2.0 SCOPE AND APPLICATIONS

This procedure provides guidance for routine field operations on environmental projects. Procedures are included for collecting grab and composite surface water samples from flowing water bodies (streams, rivers, and creeks) and from standing water bodies (lakes, lagoons, ponds, manholes, basins, tanks, and excavations).

3.0 GENERAL REQUIREMENTS

All work is performed in accordance with the project-specific planning documents. Refer to project-specific health and safety plan for relevant health and safety requirements.

Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager and discussed in the approved project plans. Deviations from requirements are sufficiently documented to re-create the modified process.

4.0 DEFINITIONS

Aliquot: Fractional amount.

Composite Samples: Samples composed of more than one aliquot collected at various sampling sites and/or at separate times.

Epilimnetic Zone: The uppermost layer of water in a lake, characterized by an essentially uniform temperature that is generally warmer than elsewhere in the lake and by a relatively uniform mixing caused by wind and wave action. Specifically, the epilimnetic zone is the light (less dense), oxygen-rich layer of water in a thermally stratified lake.

Grab Samples: Samples that are collected at one particular point and time.

Hypolimnetic Zone: The lowermost layer of water in a lake, characterized by an essentially uniform temperature (except during turnover) that is generally colder than elsewhere in the lake and is often characterized by relatively stagnant or oxygen-deficient water.

Rinsate: Wastewater generated by rinsing sampling equipment during decontamination procedures.

Surface Water Samples: Samples of water collected from streams, ponds, rivers, lakes, or other impoundments open to the atmosphere.

5.0 PROCEDURE

5.1 INTRODUCTION

The objective of surface water sampling is to evaluate the quality of the surface water entering and/or leaving a site. It is also used to obtain data on waste loads, water quality, and characteristics that permit prediction or modeling of the water system (to describe probable water quality) as well as an analysis of the effects of using the water under a variety of conditions. Surface water can also be sampled for waste disposal or discharge purposes.

5.2 SAMPLING EQUIPMENT

Sampling equipment includes all sampling devices and containers that are used to collect or contain a sample prior to final sample analysis. All surface water sampling equipment must have a design that maintains sample integrity and provides the desired level of quality in achieving desired analytical results. There is a variety of equipment available for surface water sampling. Because each site may contain varied surface water conditions, collection of a representative sample may be difficult. Automated samplers (Isco or similar type) can also be used to pull a predetermined-volume grab sample over a set time interval. In general, a sampling device should include the following characteristics:

- Be constructed of disposable or non-reactive material (~~PVC,~~ ~~Teflon~~®-lined, or stainless steel), and
- Have a minimum capacity of 500 milliliters (mL) to minimize sample disturbance.

5.3 DECONTAMINATION

Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*, after use and between sampling if dedicated disposable equipment is not used.

5.4 SAMPLING METHODS

5.4.1 General

The specific sampling method utilized depends on the accessibility to, the size of, and the depth of the water body, as well as the type of samples being collected.

In most ambient water quality studies, grab samples are collected. However, the objectives of the study dictate the sampling method and are specified in the project-specific planning documents.

For rivers, streams and creeks, the type of samples collected are dependent upon the size and the amount of turbulence in the water body. Approximate the depth and location of samples to ensure consistency. Flow rates, if required, are measured using a current meter or flow meter in accordance with the project-specific planning documents.

- With small streams, less than 20 feet wide, a single grab sample collected at mid-depth in the center of the channel is usually adequate to represent the entire cross-section. In small streams and creeks less than 10 feet wide, a single grab sample can be collected by immersing the bottle directly under the surface of the water as close to the center of the channel as possible.
- For slightly larger streams, a vertical composite sample in the center of the channel may be required. The composite sample consists of samples taken just below the surface, at mid-depth, and just above the bottom.
- For rivers, several vertical composite samples are collected across the water body. The vertical composite samples are collected at points in the cross section approximately proportional to flow. The number of vertical composites required and the number of depths sampled for each are usually determined in the field. This determination is based on a reasonable balance between two considerations:
    - The larger the number of subsamples, the more the composite sample represents the water body, but
    - Taking many subsamples is time-consuming and increases the chance of cross-contamination.
- For lakes, ponds, lagoons, and impoundments, the greater tendency to stratify and the relative lack of adequate mixing usually requires that more subsamples be collected.
    - In ponds, lagoons, and small impoundments, a single vertical composite sample at the deepest point is usually adequate.
    - In lakes and larger impoundments, several vertical composites should be combined into a single sample. In some cases, it may be useful to form several composites of the epilimnetic and hypolimnetic zones. Normally, however, a composite consists of several verticals with subsamples collected at various depths.
- For surface water samples that include co-located sediment samples, flow conditions within the water body should be considered to avoid impacts from suspended sediment.
    - In flowing water bodies (rivers, stream, creeks), the surface water sample should be collected before the co-located sediment sample at each location. Co-located surface water and sediment samples should be completed by working in a downstream to

upstream direction. The flow within the water body prevents any potential impacts to adjacent upstream sample locations.

- In low-flow or standing water bodies (lakes, lagoons, ponds), surface water samples should be collected before sediment samples at all locations. Suspended sediment generated during sediment sampling is not removed because of the lack of flow within the water body, and the sediment could affect one or more surface water samples.

5.4.2 Sample Bottle

Collecting a representative sample from small streams and creeks less than 10 feet wide, a single grab sample can be collected by immersing the sample bottle directly under the surface of the water as close to the center of the channel as possible. This method reduces the potential for cross contamination as it does not require the decontamination of equipment. The sample bottle to be used should be new, clean, and not contain any chemical preservatives. If preservatives are required for sample preservation, add the preservatives after the samples have been collected. If pre-preserved bottles are to be used, first collect the sample in an unpreserved bottle and decant the water to the pre-preserved bottle. The following procedures are followed when sampling with a sample bottle:

- All field crew members handling the sampling equipment and/or sample bottles must don new, clean gloves before beginning sampling activities and between each successive sample point.
- When wading to a sample location, approach from the downstream direction to prevent potential impacts caused by suspended sediment.
- Stand facing up stream.
- A single grab sample can be collected by immersing the bottle directly under the surface of the water as close to the center of the channel as possible ensuring the sample is collected upstream of the sampler.
- Raise the sample bottle, then seal, wipe clean, label or identify, and prepare the bottle for transport in accordance with project guidelines.
- Identify or label samples carefully and clearly, addressing all the categories or parameters;
- After labeling of the sample bottles has been completed, place the filled sample containers on ice immediately.
- Complete all chain of custody documents and record information in the field logbook (see the project-specific Quality Assurance Project Plan [QAPP] for sample custody procedures).

- Mark sample location and approximate depth, if possible, and note on maps and in field logbook.

One additional grab sample from each location is collected, and a water quality probe is used to collect pH, conductivity, temperature, turbidity, and salinity (where applicable) data if in situ measurements cannot or should not be made. This sample is not submitted for laboratory analysis. Other odors and significant characteristics are documented on the field sampling sheet and in the field logbook.

5.4.3 Weighted Bottle Sampler

Collecting a representative sample from a larger body of water requires the gathering of samples from various depths and locations. A weighted bottle sampler is typically utilized for this type of sampling. The sampler consists of a ~~Teflon~~® bottle, a weighted sinker, a bottle stopper, and a wire cord used to raise, lower, and open the samples. This type of sampler can be fabricated or purchased. The following procedures are followed when sampling with a weighted bottle sampler (Attachment 1, Weighted Bottle Sampler):

- All field crew members handling the sampling equipment and/or sample bottles must don new clean gloves prior to beginning sampling activities and between each successive sample point.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- Assemble the weighted bottle sampler in accordance with the sampler instruction manual.
- Gently lower the sampler to the desired depth so as not to remove the stopper prematurely. Do not let sampler disturb bottom sediments.
- Pull out the stopper with a sharp jerk of the sampler line.
- Allow the bottle to fill completely, as evidenced by the cessation of air bubbles.
- Raise the sampler, seal, wipe clean, label or identify, and prepare the bottle for transport in accordance with project guidelines.
- Identify or label samples carefully and clearly, addressing all the categories or parameters.
- After labeling of the sample bottles has been completed, place the filled sample containers on ice immediately.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- Complete all chain of custody documents and record information in the field logbook (see the project-specific QAPP for sample custody procedures).

- Mark sample location and approximate depth, if possible, and note on maps and in field logbook.

One additional grab sample from each location is collected, and a water quality probe is used to collect pH, conductivity, temperature, turbidity, and salinity (where applicable) data if in situ measurements cannot or should not be made. This sample is not submitted for laboratory analysis. Other odors and significant characteristics are documented on the field sampling sheet and in the field logbook.

5.4.4 Kemmerer Sampler

A Kemmerer sampler (Attachment 2, Kemmerer Sampler) consists of a hollow stainless steel or copper cylinder with a stopper on the top and bottom that can be triggered to close by a weighted messenger. The Kemmerer is best used in deeper water where the sampler can be deployed from a boat or bridge.

Perform the following procedures when sampling with a Kemmerer sampler:

- All field crew members handling the sampling equipment and/or sample bottles must don new clean gloves before beginning sampling activities and between each successive sample point.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination.*
- Assemble the Kemmerer sampler in accordance with manufacturer's instructions.
- Pull the bottom stopper down until the shaft assembly snaps into the trip head. The sampler should now be in the open position.
- Run a line through the messenger and then attach the line to the sampler. The line should run through the shaft assembly and be secured by knotting it at the bottom of the sampler with a washer.
- Submerge the Kemmerer sampler to the desired sample depth very slowly to minimize surface disturbance.
- Release the messenger down the line to engage the closing mechanism.
- Retrieve the sampling device with minimal surface water disturbance.
- Depress the valve on the bottom of the sampler to release the water into the sample container.
- Empty the sampler slowly, allowing the sample stream to flow gently down the side of the bottle with minimal entry turbulence. Fill the sample bottle and leave the appropriate headspace, if any.

- Identify or label samples carefully and clearly, addressing all the categories or parameters.
- After labeling of the sample bottles has been completed, place the filled sample containers on ice immediately.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- Complete all chain of custody documents and record information in the field logbook (see the project-specific QAPP for sample custody procedures).
- Mark the sample location and approximate depth, if possible, and note the location on maps and in the field logbook.
- Collect additional grab samples to acquire field measurements such as temperature, pH, conductivity, turbidity, salinity (where applicable) and other significant characteristics.

5.4.5 Pond Sampler

The pond or dip sampler (Attachment 3, Pond Sampler) consists of a scoop or container attached to the end of a telescoping or solid pole. The sampler must be made of nonreactive material such as wood, plastic, or stainless steel. The sample is collected in a jar or beaker made of stainless steel or ~~Teflon~~®. Preferably, a disposable beaker that can be replaced before each sampling is used at each station. Liquid wastes from water courses, ponds, pits, lagoons, or open vessels is ladled into the sample container.

Perform the following procedures when using a pond sampler:

- All field crew members handling the sampling equipment and/or sample bottles must don new clean gloves before beginning sampling activities and between each successive sample point.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- Assemble the pond sampler in accordance with manufacturer's instructions.
- Extend the pole to the length that allows safe access to the desired sample location.
- Submerge the pond sampler to desired sample depth. Submerge the sampler very slowly to minimize surface disturbance.
- Allow the sampler to fill very slowly.
- Retrieve the sampling device with minimal surface water disturbance.
- Remove the cap from the sample bottle and slightly tilt the mouth of the bottle below the sampler edge.

- Empty the sampler slowly, allowing the sample stream to flow gently down the side of the bottle with minimal entry turbulence. Fill the sample bottle and leave the appropriate headspace, if any.
- Identify or label the samples carefully and clearly, addressing all the categories or parameters.
- After labeling of the sample bottles has been completed, place the filled sample containers on ice immediately.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- Complete all chain of custody documents and record the information in the field logbook (see the project specific QAPP for sample custody procedures).
- Mark the sample location and approximate depth, if possible, and note the location on maps and in the field logbook.
- Collect additional grab samples to acquire field measurements such as temperature, pH, conductivity, turbidity, salinity (where applicable) and other significant characteristics.

5.4.6 Manual Hand Pump

Manual pumps are available in many sizes and configurations. Manual hand pumps are commonly operated by peristaltic, bellows or diaphragm, and siphon action. Manual hand pumps that operate by bellows/diaphragm and siphon action should not be used to collect samples that will be analyzed for volatile organics (Attachment 4, Manual Hand Pump). These types of pumps should be constructed of inert materials, such as ~~Teflon~~® or stainless steel.

Perform the following procedures when collecting surface water samples with a manual hand pump:

- All field crew members handling the sampling equipment and/or sample bottles must don new clean gloves prior to beginning sampling activities and between each successive sample point.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- Assemble and operate the pump in accordance with the manufacturer's instructions.
- The inlet hose and any surface of the pump used for sampling must be constructed of materials that are operable and non-reactive.
- To avoid agitation, insert the sampling tube into the liquid sample prior to pump activation.

- Insert a liquid trap (preferably the sample container) into the sample inlet hose to collect the sample and to prevent pump contamination.
- Identify or label samples carefully and clearly, addressing all the categories or parameters.
- After labeling of the sample bottles has been completed, place the filled sample containers on ice immediately.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- Complete all chain of custody documents and record information in the field logbook (see the project-specific QAPP for sample custody procedures).
- Mark the sample location and approximate depth, if possible, and note the location on maps and in the field logbook. Record applicable data in the field logbook such as color, turbidity, pH, temperature, turbidity (where appropriate), degree of turbulence, and weather conditions.

5.4.7 Peristaltic Pump

Gathering surface water samples with the assistance of a peristaltic pump is another commonly used sampling technique. In this method, the sample is drawn through heavy-walled tubing and pumped directly into the sample container. This system allows the operator to extend into the liquid body to sample from depth or sweep the width of narrow streams. Medical-grade silicon tubing is often used in the peristaltic pump, and the system is suitable for sampling almost any parameter, including most organics (Attachment 5, Peristaltic Pump).

Peristaltic pumps are available with a range of power sources. The battery-operated units have proven to be the most convenient and dependable.

Perform the following procedures when sampling with a peristaltic pump:

- Prepare the peristaltic pump in accordance with manufacturer's instructions. When using a battery-operated pump, ensure that the battery is fully charged before entering the field.
- In most situations, it is necessary to change the ~~Teflon~~® suction line and the silicon pump tubing between sample locations to avoid cross-contamination.
- All field crew members handling the sampling equipment and/or sample bottles must don new clean gloves prior to beginning sampling activities and between each successive sample point.
- Gently lower the pump intake tube to the desired sample depth. Avoid unnecessary agitation (aeration) of the liquid to be sampled and bottom sediments.

- Before activating the pump, note in which direction the pump will be rotating. (Most peristaltic pumps are capable of rotating in two directions.) Accidental reverse rotation of the pump will cause aeration of the liquid to be sampled.
- Run the pump until no air bubbles are noted in the discharge.
- Discharge water shall be released downstream of the sampling area.
- To prevent excess agitation and/or aeration of the sampler, fill the sample containers by tilting the container and flow the sample water down the side of the sampling container.
- Identify or label samples carefully and clearly, addressing all the categories or parameters.
- After labeling of the sample bottles has been completed, place the filled sample containers on ice immediately.
- In most cases, no specific decontamination procedures are required because of the use of disposable tubing. However, site-specific sample procedures may require additional decontamination and are specified in the project-specific planning documents.
- Complete all chain of custody documents and record information in the field logbook (see the project-specific QAPP for sample custody procedures).
- Mark the sample location and approximate depth, if possible, and note the location on maps and in field logbook. Record applicable data in the field logbook such as color, turbidity, pH, salinity (where applicable), degree of turbulence, and weather conditions.

When medical grade silicon tubing is not available for analytical requirements, the system can be altered as illustrated in Attachment 6, Peristaltic Pump – Modified. In this configuration, the sample volume accumulates in the vacuum flask and does not enter the pump. This system provides excellent sample integrity for most analyses; however, the potential for losing volatile fractions to the reduced pressure of the vacuum flask renders this method unacceptable for sampling of volatiles.

It may sometimes be necessary to sample large bodies of water where a near-surface sample will not sufficiently characterize the body as a whole. In this instance, the above-mentioned pump is appropriate. It is capable of lifting water from slightly deeper than 6 meters. It should be noted that this lift ability decreases with higher density fluids and with increased wear on the silicone pump tubing. Similarly, increases in altitude decrease the ability of the pump to lift from depth. When sampling a liquid stream that exhibits a considerable flow rate, it may be necessary to weight the bottom of the suction line.

5.4.8 Optional Sampling Methods

The above-mentioned methods of surface water sampling are used most often on HGL environmental projects; however, choice of sampling equipment depends on site-specific conditions. The following additional types of samplers are available:

- Isco Teledyne automated sampler,
- Wheaton sampler,
- Bacon Bomb sampler,
- Open tube sampler, and
- Bailer.

Before any fieldwork, the project manager or designee reviews the available sampling equipment and chooses the sampling device that best suits the project requirements. The sampling device to be used is specified in the project-specific planning documents. If using an automated sampler, ensure that the tubing/sample containers selected are appropriate for the site COCs, that the manufacturer's instructions are followed, and that new sample tubing, collection vessels, and sample bottles are used to collect individual samples as specified for the other sampling methods discussed above.

6.0 RECORDS

Documentation generated by this procedure is collected and maintained in accordance with requirements specified in the project-specific planning documents.

- Document all daily field activities in a daily field activity report.
- The Surface Water Sampling Data Form (Attachment 7) must be filled out for each surface water sample collected.
- Complete the field logbook in accordance with the procedures listed in SOP 300.04: *Field Logbook Use and Maintenance*.

7.0 REFERENCES

U.S. Army Corps of Engineers, 2001. *Requirements for the Preparation of Sampling and Analysis Plans* (EM 200-1-3). Appendix C.3.

8.0 REVISION HISTORY

Revision 0	December 2010	Initial Release
Revision 1	July 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	March 25, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 2.16 to 404.01.

ATTACHMENTS

Attachment 1 – Weighted Bottle Sampler
Attachment 2 – Kemmerer Sampler
Attachment 3 – Pond Sampler
Attachment 4 – Manual Hand Pump
Attachment 5 – Peristaltic Pump
Attachment 6 – Peristaltic Pump - Modified
Attachment 7 – Surface Water Sampling Data Form

ATTACHMENT 1

WEIGHTED BOTTLE SAMPLER

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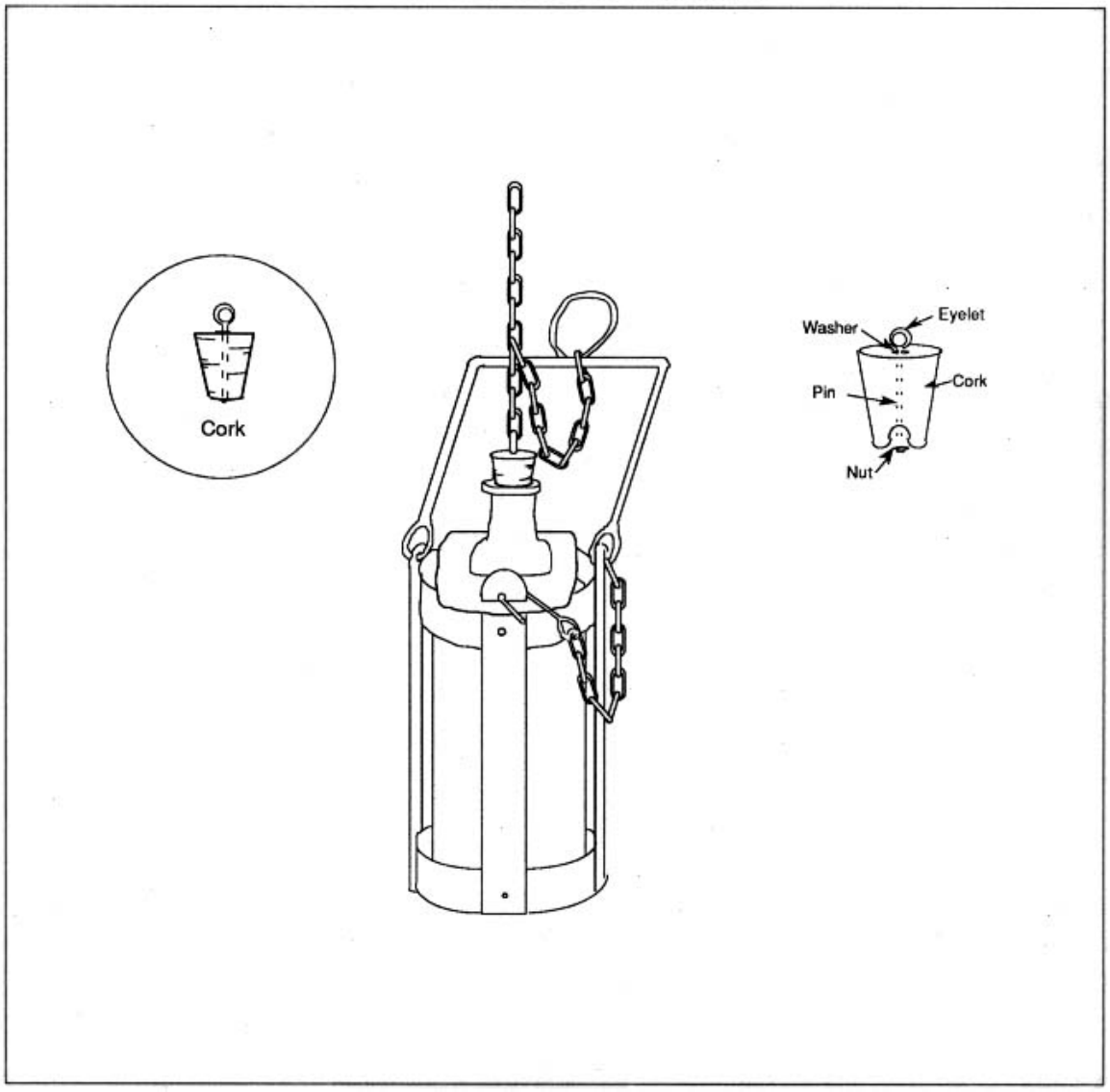
WEIGHTED-BOTTLE SAMPLER



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ATTACHMENT 2

KEMMERER SAMPLER

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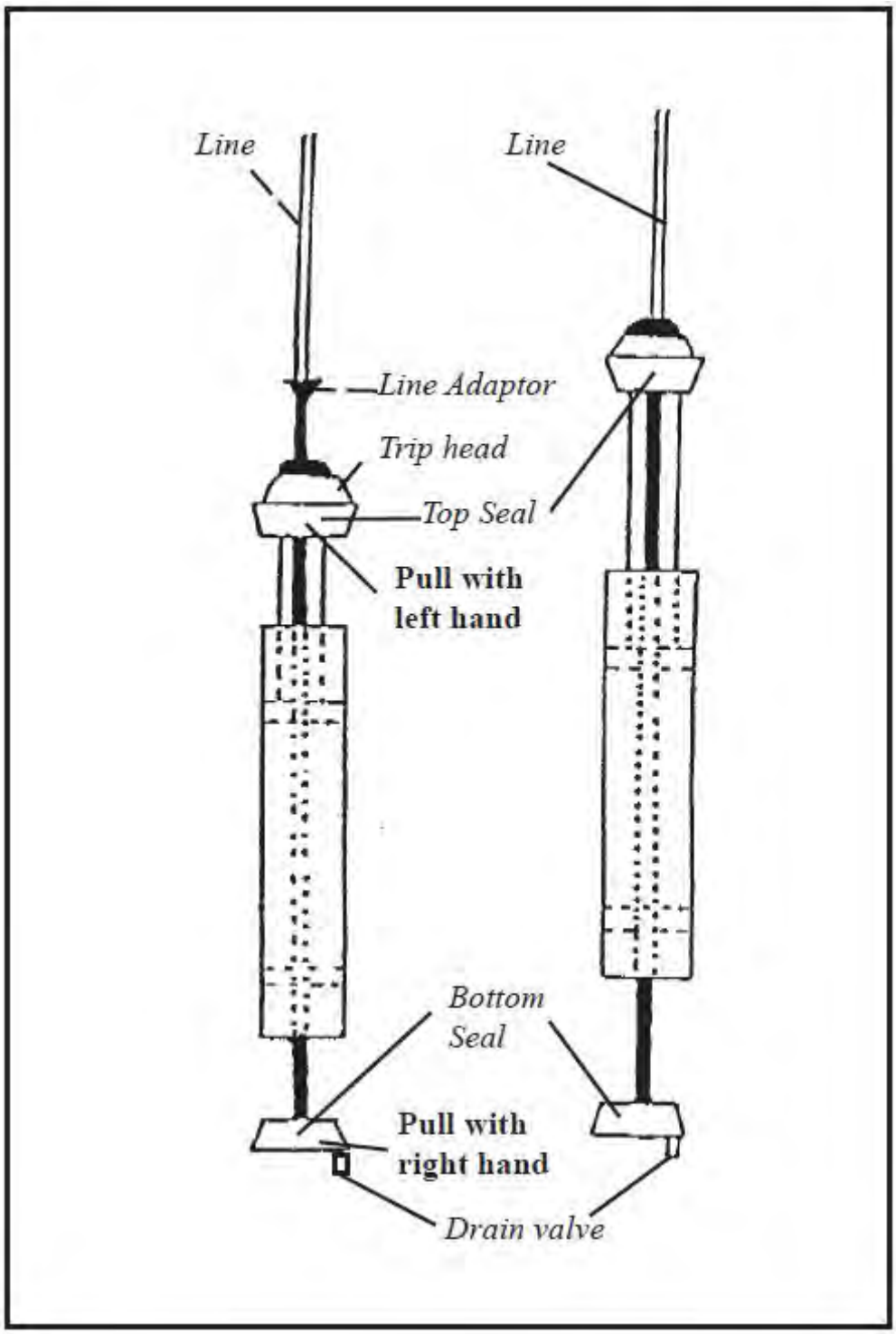
KEMMERER SAMPLER



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ATTACHMENT 3

POND SAMPLER

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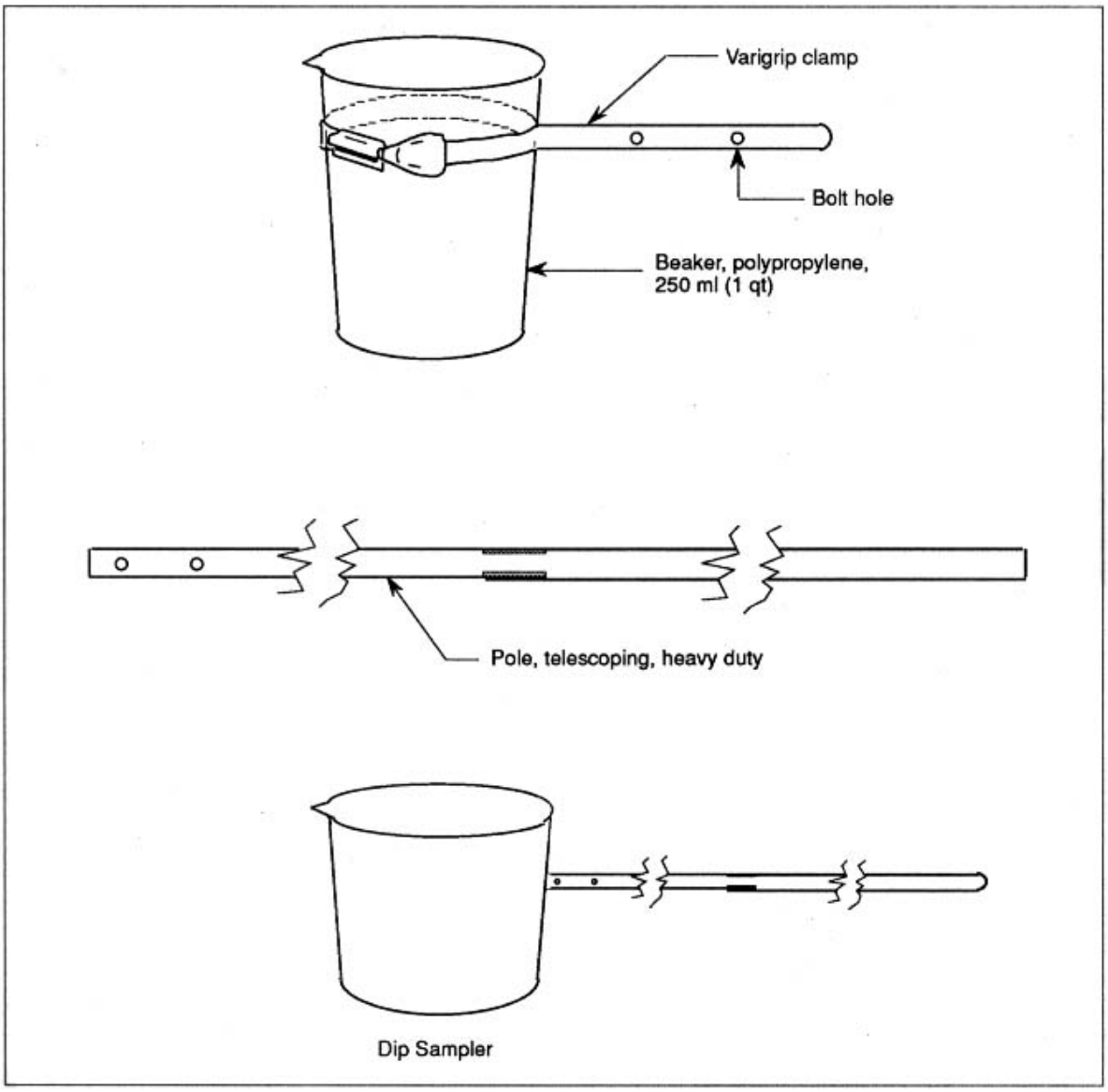
POND SAMPLER



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ATTACHMENT 4

MANUAL HAND PUMP

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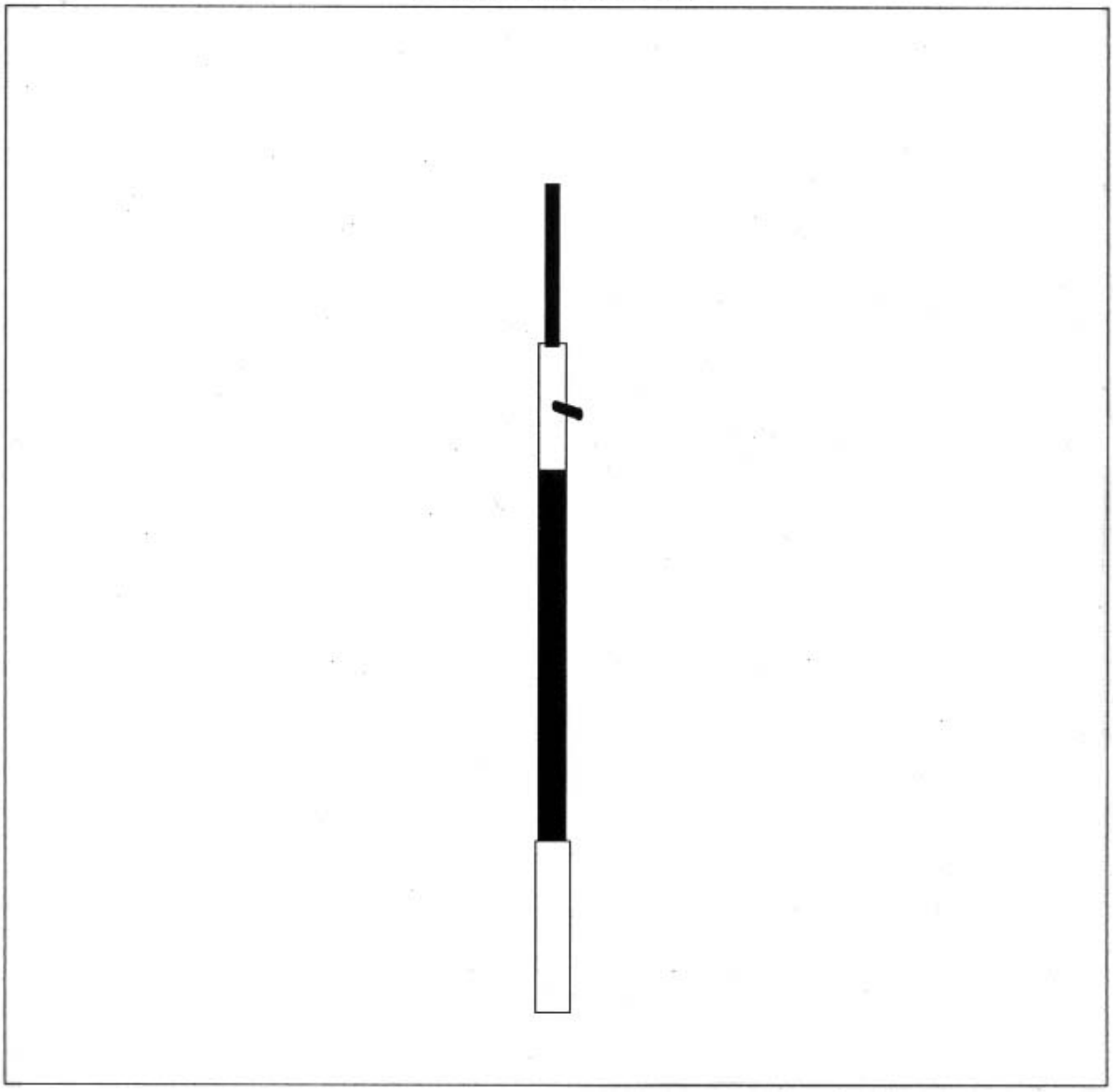
MANUAL HAND PUMP



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ATTACHMENT 5

PERISTALTIC PUMP

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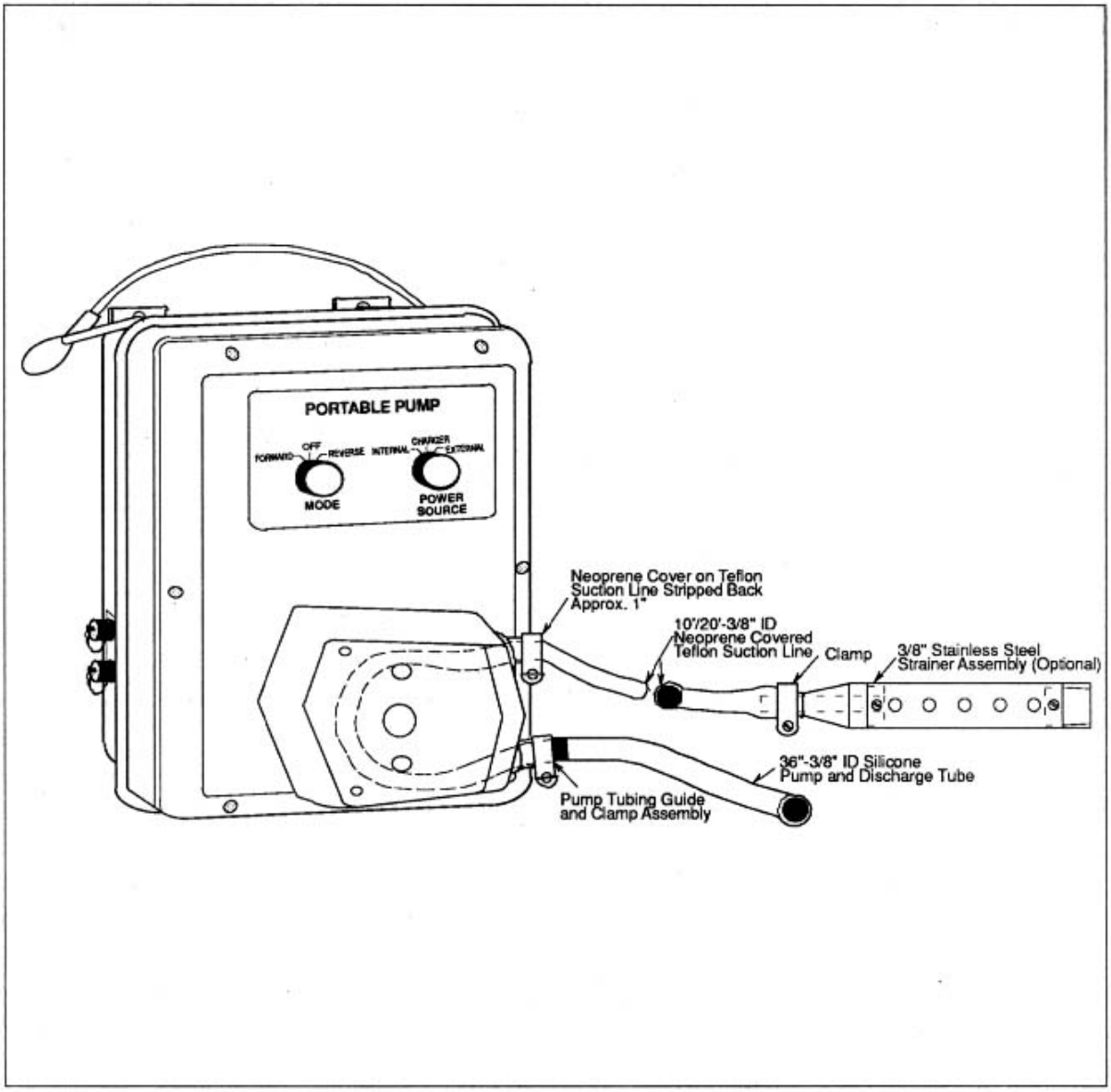
PERISTALTIC PUMP



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ATTACHMENT 6

PERISTALTIC PUMP - MODIFIED

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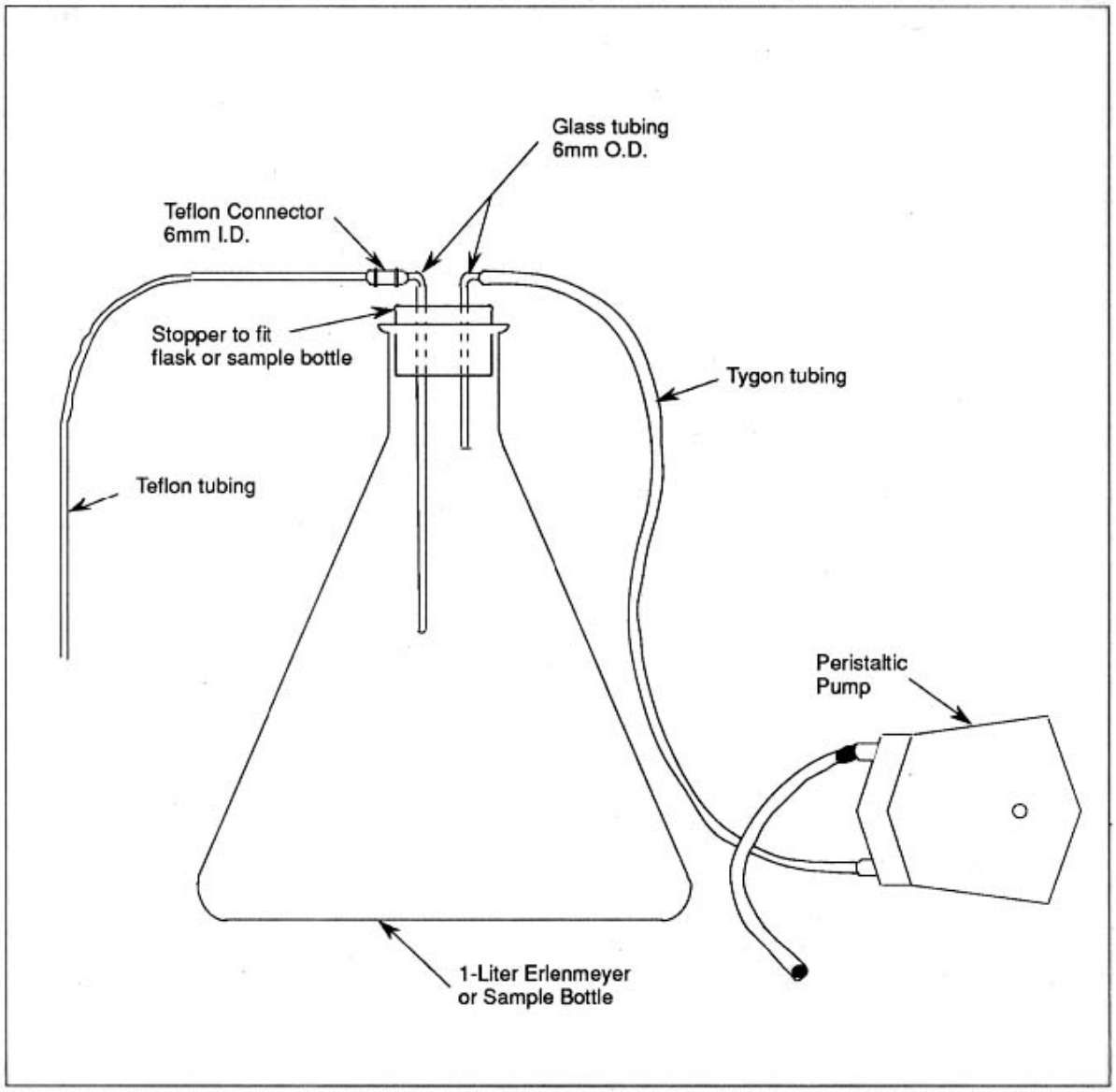
PERISTALTIC PUMP - MODIFIED



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ATTACHMENT 7

SURFACE WATER SAMPLING DATA FORM

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HGL HydroGeoLogic, Inc. Exceeding Expectations

Surface Water Sampling Data

Records Management Data

Project Number	Project Name	Page
		____ of ____

Time/Date: _____ Elevation: _____

Sample No.: _____ Weather: _____

Location: _____ Amb. Temp (°F): _____

Sampling Method: _____

WATER SAMPLE DATA

Water Temp: _____ °C Method of Measurement: _____

Specific Conductance: _____micromhos Method of Measurement: _____

pH: _____ Method of Measurement: _____

Containers Used (VOA Vial, 1 liter jar, etc.): _____

Physical Appearance: _____

Contamination Observed: _____

Remarks: _____

Location Sketch

Recorded By:	Date	Checked By:	Date:

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HGL HydroGeoLogic, Inc Exceeding Expectations	**CORPORATE TECHNICAL PROCEDURE**	
	Approved for issue by:	
	Process Owner	Jodie Johnson
	Corporate Quality Director	Rojas, Theresa Digitally signed by Rojas, Theresa Date: 2021.06.22 12:56:36 -04'00'
Data Validation, U.S. EPA/DoD Stage 2A and Stage 2B		**Document No.: HGL SOP 412.501 (formerly 4.09)**
		Process Category: Services
		Revision No.: 3
		Effective Date: June 15, 2021
		Last Review Date: June 15, 2021
		Next Review Date: June 2023

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) provides information on the methodology and protocols required to review and validate analytical data generated from the laboratory analysis of environmental media. This SOP is intended to provide general guidance for the evaluation of the quality control (QC) elements associated with analytical data. Project-specific criteria for data validation are presented in each project's Quality Assurance Project Plan (QAPP), as are the project-specific QC acceptance criteria. Users of this SOP are authors of QAPPs, preparers of electronic QAPPs (eQAPPs) supporting automated data review (ADR), data validators, and data users.

2.0 SCOPE AND APPLICATION

The U.S. Environmental Protection Agency document *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (EPA, 2009) and Department of Defense *General Data Validation Guidelines* (DoD EDQW, 2019) define five stages of data validation: Stage 1, Stage 2A, Stage 2B, Stage 3, and Stage 4. Each stage increases the level of complexity and detail in the validation process and incorporates all relevant requirements of each preceding stage. Stage 2A and Stage 2B are the two most common stages of data validation performed in support of HydroGeoLogic, Inc.'s (HGL's) environmental projects. Stage 2A validation consists of a review of sample receipt, condition, and documentation (these Stage 1 elements correspond to "data verification"); holding times; and sample-specific and batch-specific QC elements. Stage 2B validation consists of all the elements of a Stage 2A validation, with additional review of instrument and analytical system QC elements. An individual laboratory's data report format may not include a summary form for a required QC element; such cases require the examination of raw data to provide information on the affected QC element.

The appropriate stage of data validation to be performed on analytical results is determined by HGL's project scope of work (SOW) and is presented in the project QAPP. Depending on the objectives for the project dataset, the actual validation performed on any given set of results is determined on a sample- and analytical method-specific basis. Generally, Stage 2B data validation is performed on analytical results that must be considered definitive and usable for supporting final decision-making and for performing quantitative risk assessments. Stage 2A data validation is performed to provide a general assessment of sampling and laboratory performance and does not

result in data that are usable for final decision-making or risk assessment. Stage 2A validation is typically performed on data generated for natural attenuation parameters and on data generated by long-term monitoring, operations and maintenance sampling, and compliance monitoring.

Stage 3 and Stage 4 data validation involve a greater level of effort and build on the Stage 1, 2A, and 2B data validation procedures. Stage 3 validation involves recalculating sample, calibration standard, and QC analysis results; comparing instrument response to minimum response requirements; and verifying that target analytes are quantified with an appropriate internal standard. Stage 4 validation includes verifying transcription of raw data to summary forms and examination of raw instrument results, including standard preparation logs, quantitation reports, chromatograms, and mass spectra for completeness, accuracy, and technical acceptability. Performing the review components associated with Stage 3 and Stage 4 validation relies almost entirely on the validator's professional judgment and experience, and these components are not covered by this SOP. No Stage 3 or Stage 4 data validation tasks can be assigned to HGL personnel without the approval of an HGL senior chemist.

Data generated for waste characterization and data associated with QC samples generally require no validation or only a Stage 1 data verification plus evaluation of holding times unless anomalous results are noted. Federal, state, or program requirements may include performing a higher stage of validation than is normally performed on any given sample or set of samples.

The QC elements that make up data validation Stages 2A and 2B, including the Stage 1 elements on which these stages build, are provided in Attachment A. The components of Stage 3 and Stage 4 data validation are also provided for reference.

3.0 GENERAL REQUIREMENTS

3.1 PRE-REVIEW ITEMS

Prior to beginning validation of laboratory data reports, the data validator must obtain the following items and information from the project manager (or designee):

1. The correct billing code for data validation tasks;
2. The most recent version of all relevant QAPPs (including any basewide QAPP and QAPP addenda);
3. The stage of data validation to be performed on the data (multiple stages are possible depending on end use of individual samples or the results from specific analytical methods);
4. The schedule and anticipated level of effort to complete validation tasks;

5. The identity of any field duplicate or triplicate samples and the associated parent samples; and
6. The identity of any field blanks (equipment, trip, ambient, and material blanks) and the correct association protocol for each blank.

3.2 LABORATORY DATA REPORTS

The data reports produced by each laboratory typically have substantial differences in presentation, bookmarking, structure, and formatting when compared to a data report produced by another laboratory, although some similarities will be present. Each project laboratory is required to provide data packages that support the stage of review that the associated data will undergo. Summary pages that provide all the validation stage-specific information listed in Attachment A are preferred, although in some cases summary pages may need to be supplemented with information only available on instrument printouts or raw data due to limitations in laboratory report-generation software.

Before data validation, the validator should examine the laboratory data reports to ensure that all required information necessary to perform the required stage of data validation is available and presented in a format that supports the validation effort. Familiarity with the laboratory's reporting conventions improves the efficiency of the data validation process as well as the quality of the validation, as the validator will be better able to identify QC discrepancies in the reported data and judge the effect on the associated sample results.

Control limits for surrogate recoveries, laboratory control sample (LCS) and LCS duplicate (LCSD) recoveries, matrix spike (MS) and matrix spike duplicate (MSD) recoveries, LCS/LCSD precision, MS/MSD precision, and duplicate precision are usually presented in the project QAPP. If the control limits are specified in the QAPP, the validator should verify that the laboratory reports incorporate the required control limits. Failure to verify that the laboratory-reported control limits are those specified by the QAPP can cause QC discrepancies to be misidentified as conforming data points and conforming data points to be misidentified as discrepancies. In both cases, the data are not evaluated against the requirements for precision and accuracy specified in the QAPP. This scenario can result in misqualified data and in additional validation efforts to correct the laboratory-applied qualifiers. It can also result in the laboratory's failing to identify a QC discrepancy and subsequently failing to perform required corrective action. Verifying that the correct control limits are being presented prior to beginning the validation effort is the best way to ensure that the reported results meet the precision and accuracy requirements established for the project as presented in the QAPP. If discrepancies are noted, the laboratory project manager should be notified that the data reporting pages do not present the correct information and that the laboratory should ensure that all future deliverables conform to the requirements of the QAPP.

In some cases, the laboratory's internally derived control limits may be acceptable, either for entire analytical suites or individual analytes for which program limits have not been established. Where

a QAPP indicates that a set of control limits are laboratory-specific, those limits can change over time as laboratories evaluate and update their control limits. Should a laboratory data package report laboratory control limits that differ from those in the QAPP, the validator should consider the current control limits to supersede the QAPP limits and document this decision in the data validation report.

If required QC review elements or individual pages are missing from a laboratory data report, and the missing information is a result of an error in report compilation (such as a missing or illegible page), the validator should contact the laboratory project manager directly and request that the missing information be provided. If the missing information is the result of a laboratory report generation convention (that is, the lack of a required data QC element is due to report design, not to an error in report compilation), the data validator should contact the HGL project chemist. The HGL project chemist must coordinate with the laboratory project manager to ensure that any required information is provided to the data validators in alternative formats so that all QAPP-required QC elements can be reviewed.

3.3 DATA VALIDATION REPORTS

Data validation is documented in a data validation report, and each report contains a subsection for each analytical method reported in a single sample delivery group (SDG).

In cases where individual project requirements conflict with the requirements of this SOP, the project requirements take precedence and should be used throughout the data validation and evaluation process; however, the data validator or HGL senior chemist may deviate from the stated project requirements based on professional judgment. Any deviations from specified requirements must be technically appropriate, and they must be justified in the corresponding data validation report and HGL validation report review memo. Deviations in the assessment of the project dataset must also be documented in any data quality or usability evaluation associated with project report deliverables.

Example data report formats are presented in Attachment B. Note that the qualification conventions used in the example reports are based on the requirements of a specific project. The qualifiers assigned during the validation process should reflect the project's conventions.

3.4 PEER REVIEW

All data validation reports generated by HGL personnel are subject to a secondary review by either a peer or senior chemist assigned by the Chemistry Group leader. The peer reviewer evaluates the data validation report against the contents of the laboratory data report to ensure that the following applies:

1. The data validator has correctly applied the project requirements as presented in the QAPP to evaluate and qualify the reported sample results.
2. The data validator has not overlooked any QC discrepancies present in the data package.
3. The validator has correctly associated any QC discrepancies with the correct analytes and analyses.
4. The assigned data qualifiers are complete and correct.
5. The data validator has not made "boilerplate" errors (that is, the inclusion of extraneous and incorrect information in the data report as a result of using another report as a template without removing or modifying material that does not apply).

A validation report that has not been reviewed cannot be considered final.

3.5 SUBCONTRACTED DATA VALIDATION

The goal of subcontracted data validation is to generate a validated project dataset that is qualified in accordance with QAPP requirements and ready for HGL to upload into the project database. Subcontracted data validation is performed in accordance with the individual firm's internal procedures and policies; however, the overall procedure must include pre-review, validation by qualified personnel, and peer or senior review of all data validation reports (in accordance with Section 3.4) before delivery to HGL. All validation must be performed in accordance with the project QAPP and the SOW provided by HGL. In addition to a validation report, the subcontracted validator may be responsible for providing qualified data electronically in a format that allows upload into HGL's project database (see Section 6.0), usually in the form of an Excel file. The validation firm is responsible, in accordance with the project-specific data validation SOW, for any data entry, data entry QC, and removal of any residual laboratory-applied flags prior to delivery to HGL.

HGL reviews data validation reports provided by third-party contractors in accordance with the procedures presented in Attachment F. The initial data validation reports provided by the contractor must be reviewed in depth by an HGL senior chemist as soon as possible to provide the data validator with timely feedback to guide ongoing validation efforts. The primary purpose of the HGL senior chemist review is to verify that the data validators understand the QAPP and project data quality requirements and are applying these requirements correctly when reviewing each data package. Data validation involves a large amount of professional judgment, and there are multiple conventions that are technically valid. Therefore, a secondary purpose of the HGL senior chemist's review is to ensure that the conventions HGL selected are being used by the contractor to maintain consistency in evaluation and application of qualifiers from SDG to SDG within a project. When it has been established that HGL's expectations are being met, subsequent data validation reviews can be streamlined to verify that the identified QC issues discussed in each validation report led to correct qualification of the associated sample results. It should be kept in

mind, however, that many data validation firms have a pool of staff validators and there can be variability in the quality and completeness of individual data validation reports submitted from a third-party contractor.

4.0 PERSONNEL

Data validation and review must be conducted by appropriately qualified and trained personnel.

4.1 ROLES, RESPONSIBILITIES, AND QUALIFICATIONS

4.1.1 HGL Project Staff

HGL project staff are assigned in accordance with contract requirements and HGL's project management procedures. The following personnel have a wide range of responsibilities associated with their project titles; however, only the responsibilities applicable to the data validation process are discussed. It is possible for the HGL chemistry staff identified below to operate in multiple functions. For example, an HGL senior chemist can act as a project chemist for an individual project and perform the functions of both project chemist and senior chemist for that project.

HGL Project Manager – Provides the data validation team with the information listed in Section 3.1, either directly or through a designee (such as a task manager). Ensures that all required project personnel, including sample collection, laboratory, and data validation subcontractors, are provided with the current project QAPP as well as any QAPP revisions in a timely fashion.

HGL Project Chemist – Provides guidance on analytical method requirements for sampling, preservation, and holding time requirements to field sampling teams. Resolves issues not covered by the QAPP or other guidance documents. Ensures that laboratory performance is in accordance with HGL's project technical requirements. For projects with subcontracted data validation, reviews data validation reports to verify that the data validation contractor is performing in accordance with the contract SOW and the QAPP (see Appendix F). After ensuring that the laboratory and validation contractors, if applicable, have performed in accordance with HGL's project technical requirements, provides approval of invoices for payment.

HGL Senior Chemist – For some projects, this role may be identified as "program chemist" based on client organizational designating conventions. Assists senior program chemist in implementing the data validation program and provides technical input to support the program. Assists the project chemist in resolving issues not covered by the QAPP or other guidance documents. Assists the project chemist in ensuring that laboratory and validation contractor, if applicable, is performing in accordance with HGL's project technical requirements. Assists project manager in communicating data quality issues to the client and addressing client or stakeholder concerns. Assists senior program chemist in identifying and resolving deficiencies in project laboratory or

subcontracted validator performance. Trains junior project staff in data validation and monitors performance.

HGL Senior Program Chemist – Provides overall direction to HGL's data validation program. Works with senior HGL management to resolve deficiencies in project laboratory or subcontracted validator performance.

4.1.2 Data Validation Staff

Data validation staff includes data validators and peer reviewers who are assigned on an as-needed basis. Data validation staff can consist of qualified HGL personnel including chemists, geologists, environmental scientists, or other technical staff who have been trained in data validation by an HGL senior chemist or are judged by an HGL senior chemist to have sufficient experience in data validation. The qualifications and roles of data validation staff are described below.

HGL Data Validator – Must have at least a bachelor's degree in chemistry or other scientific discipline. The HGL data validator performs data validation, communicates with the laboratory to resolve issues, and writes the data validation reports. Data validation reports generated by an HGL validator with less than 1 year of experience must be reviewed by an HGL senior chemist.

HGL Peer Reviewer – Must have at least a bachelor's degree in chemistry or other scientific discipline and at least 2 years of data validation experience. Peer reviewers perform a complete review of the findings of each data validation report against the associated laboratory data deliverable and determine if the validator has (1) addressed all QC issues affecting project data in accordance with the requirements of the project QAPP, (2) assigned the correct qualifiers to the reported data, (3) complied with project validation conventions, and (4) presented a clear description of the data quality issues affecting the reported data. Peer reviewers with less than 1 year of peer review experience are subject to approval by an HGL senior chemist before assignment.

Depending on the size of the project and staffing requirements, multiple data validators and peer reviewers may be assigned to a project; a data validator assigned to one laboratory deliverable may be a peer reviewer for another laboratory deliverable validation report. It is recommended, but not required, that each project's project chemist be one of the HGL personnel assigned to perform data validation and peer review tasks for that project.

4.2 TRAINING REQUIREMENTS

HGL data validation staff must be trained directly by an HGL senior chemist. This training preferably takes place in person to allow for greater efficiency in instruction, evaluation, and feedback. Training includes validation of laboratory data reports followed by feedback and revision.

5.0 PROCEDURES

Data will be reviewed and qualified in accordance with the project QAPP and validator judgment. The qualification guidelines presented in each QAPP are based on the project data quality objectives (DQOs) and must specify the stage of data validation required to meet those DQOs. Stage 2A and Stage 2B are the most common stages of validation specified by project QAPPs. These stages of data validation usually include only the examination of the information presented on laboratory-generated summary forms. This approach is generally sufficient to determine that the laboratory is following analytical method, programmatic, and project-specific requirements.

On occasion, a review of specific raw data elements is necessary to supplement the information presented on the summary reporting forms. Stage 4 data validation, which includes a detailed review of instrument raw data and laboratory records and provides the most rigorous evaluation of data quality, is occasionally specified by a project contract. Where required, Stage 3 or Stage 4 validation is commonly performed on a specified subset of project data, such as 10 percent. Unless otherwise specified in the project QAPP, the checks and recalculations associated with Stage 3 and Stage 4 validation should be performed at the frequencies presented in Section 4.7 of the *General Data Validation Guidelines* (DoD EDQW, 2019b). Stage 4 validation is highly dependent on the professional expertise and experience of the validator and is specific to individual analytical methods and instrumentation. Consequently, the procedures required to complete this stage of data validation are not included in this SOP.

The specific procedures required to perform data validation vary greatly among data reports. The sources of variation include method QC requirements, client and regulatory requirements, laboratory-specific reporting conventions, and sample matrix. General guidelines for the evaluation of Stage 2A QC elements and method-specific Stage 2B QC elements are presented in Attachment C.

Stage 2A validation can be supported by ADR, such as the web-based ADR functionalities provided by Environmental Synectics, Inc. (Synectics) and the FUDSChem ADR program developed by the Department of Defense, as part of its scope of data management services. A description of the ADR process and its integration into the data validation process is presented in Attachment D. When ADR is incorporated into a project that requires Stage 2B validation, the data are validated to Stage 2A by ADR followed by manual verification of the ADR results and additional manual validation to complete the Stage 2B validation.

6.0 DATABASE QUALIFICATION

After the method-specific data validation reports for an SDG have been generated in accordance with Section 3.3 and reviewed in accordance with Section 3.4, the data qualifiers assigned by the validator are applied to electronic database output files. The procedures for data entry, review, and

upload are presented in HGL SOP 300.07 (formerly 303.01): *Environmental Data Management*.[1] During what is referred to as the "100 percent QC stage" of this process, all residual laboratory-generated information flags not retained as the final qualification must be removed from each result. The only laboratory-generated flags that are retained are those that have been accepted as the final qualifier by the data validator. When data validation has been subcontracted, the contractor is responsible for removing residual laboratory flags before delivering the qualified data files to HGL.

In some cases, projects require the application of a reason code as well as a qualifier to validated results. In such cases, the HGL project chemist develops a list of reason codes, and the HGL database manager uploads these reason codes to the database. Common reason codes are included in Attachment E. If HGL has not mandated a specific reason code protocol for a project, data validation subcontractors may use their internally developed reason codes.

7.0 SENIOR DATA RE-EVALUATION

When severe QC discrepancies are encountered, it may become necessary to reject associated data points. Rejected data points cause data gaps in the resulting dataset and can prevent that dataset from being used to achieve project DQOs; however, not all data gaps attributable to rejected results have an equal impact. Of special concern are (1) rejected results that affect a contaminant that could be present at the subject site or (2) rejection of a large number of analytes in individual samples because of sample-specific or batch-specific QC issues.

If results are rejected in the initial data validation, the issue must be evaluated for referral to an HGL senior chemist for supplemental senior review. This review includes discussions with laboratory quality assurance personnel, examination of raw data, and evaluation of the end use of the affected data. The review evaluates the feasibility of applying a less severe qualifier. In some cases, a less severe qualifier will not be technically justified, and an R qualifier will be applied to the affected results. In others, it may be determined that the affected results can be used to support decision-making, and the application of a less severe qualifier is technically appropriate. In all cases where HGL determines that rejection is not required, in contradiction to the requirements of the QAPP, an HGL senior chemist documents this judgment. This documentation must be made available to the client for review and approval, either in the form of technical memoranda or discussion in the associated project report (see Section 3.3).

[1] When updated, SOP 300.07 will be renumbered as HGL SOP 411.501.

8.0 REFERENCES

U.S. Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the U.S. Department of Energy (DOE) Consolidated Audit Program (DOECAP) Data Quality Workgroup (DOE-DQW), 2019. *General Data Validation Guidelines*. November.

U.S. Environmental Protection Agency (EPA), 2009. *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use.* OSWER 9200.1-85; EPA-540-R-08-005. January.

9.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
0	November 2012	Initial Release
1	April 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	February 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
3	June 15, 2021	Updated to incorporate lessons learned on the process and changes in DoD programmatic requirements and to reflect changes in SOP formatting, which included changing the SOP number from 4.09 to HGL SOP 412.501.

ATTACHMENTS

Attachment A Components of the Stages of Data Review
Attachment B Example Data Validation Reports
Attachment C General Validation Guidelines
Attachment D Automated Data Review
Attachment E HGL Data Qualification Reason Codes
Attachment F Review of Subcontracted Data Validation Reports

ATTACHMENT A
Components of the Stages of Data Review

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ATTACHMENT A
Components of the Stages of Data Review

All Analytical Fractions	Stage 1	Stage 2A	Stage 2B	Stage 3	Stage 4
Case narrative	X	X	X	X	X
Chain of custody	X	X	X	X	X
Sample receipt and log-in forms	X	X	X	X	X
Sample identification (ID) cross reference (HydroGeoLogic, Inc. sample ID to laboratory sample ID)	X	X	X	X	X
Sample discrepancy reports, corrective action, and client communications	X	X	X	X	X
Holding times (preparation and analysis)		X	X	X	X
LCS/LCSD(1) recoveries and precision		X	X	X	X
MS/MSD(2) recoveries and precision		X	X	X	X
Method blanks		X	X	X	X
Field blanks (trip, ambient, equipment, and material blanks)		X	X	X	X
Field duplicate precision		X	X	X	X
GC/MS, LC/MS, and LC/MS/MS Organic Analytical Fractions	**Stage 1**	**Stage 2A**	**Stage 2B**	**Stage 3**	**Stage 4**
Surrogate recoveries		X	X	X	X
Instrument tuning			X	X	X
Instrument initial calibration (including minimum relative response factors [RRFs])			X	X	X
Second source calibration verification			X	X	X
Instrument continuing calibration verification (including minimum RRFs)			X	X	X
Internal standards or labeled standards			X	X	X
Calculations				X	X
Chromatograms					X
Quantitation reports					X
Mass spectra					X
Transcription					X
GC and HPLC Organic Fractions(3)	**Stage 1**	**Stage 2A**	**Stage 2B**	**Stage 3**	**Stage 4**
Surrogate recoveries		X	X	X	X
Instrument initial calibration			X	X	X
Second source calibration verification			X	X	X
Instrument continuing calibration verification			X	X	X
Degradation summary (organochlorine pesticides only)			X	X	X
Retention times			X	X	X
Confirmation			X	X	X
Calculations				X	X
Chromatograms					X
Quantitation reports					X
Transcription					X

ATTACHMENT A (continued)
Components of the Stages of Data Review

Metals Fractions	Stage 1	Stage 2A	Stage 2B	Stage 3	Stage 4
Laboratory duplicate(2) precision		X	X	X	X
Serial dilution results		X	X	X	X
Post-digestion spike recoveries		X	X	X	X
Initial and continuing calibration blanks			X	X	X
Instrument tuning (ICP-MS methods only)			X	X	X
Internal standards (ICP-MS methods only)			X	X	X
Initial multipoint calibration(4)			X	X	X
Low-level calibration verification			X	X	X
High-level calibration verification			X	X	X
Initial and continuing calibration verification			X	X	X
Interference check sample results			X	X	X
Recovery test recoveries (GFAA methods only)			X	X	X
Method of standard addition results			X	X	X
Calculations				X	X
Interelement correction factors					X
Instrument raw data					X
General Chemistry Fractions	**Stage 1**	**Stage 2A**	**Stage 2B**	**Stage 3**	**Stage 4**
Laboratory duplicate(2) precision		X	X	X	X
Method-specific QC checks(5)		X	X	X	X
Initial and continuing calibration blanks			X	X	X
Initial multipoint calibration			X	X	X
Initial and continuing calibration verification			X	X	X
Method-specific instrument QC			X	X	X
Calculations				X	X
Instrument raw data					X

(1) LCSDs are not a requirement for any method or project; however, they are often provided by the laboratory. They are reviewed when available.
(2) The analytical methods allow for metals and general chemistry precision to be evaluated either using MS/MSDs or laboratory duplicates at the laboratory's discretion. Often laboratories provide both. The data validator reviews all available QC data provided by the laboratory.
(3) These methods use a second column or detector to confirm detected results. QC elements for both columns/detectors should be reviewed during the validation process.
(4) Initial multipoint calibration is optional for ICP methods; if performed, the validator reviews the associated results.
(5) An example of method-specific QC checks is distillation checks for cyanide analysis.

Notes:
GC/MS = gas chromatography/mass spectrometry
GFAA = graphite furnace atomic absorption
HPLC = high-performance liquid chromatography
ICP = inductively coupled plasma
ICP-MS = inductively coupled plasma-mass spectrometry
LC/MS = liquid chromatography/mass spectrometry
LC/MS/MS = liquid chromatography/tandem mass spectrometry
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
MS = matrix spike
MSD = matrix spike duplicate
QC = quality control

ATTACHMENT B
Example Data Validation Report

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B.1
Example Data Validation Report

USEPA Stage 2B Validation Report

Section 1 – General Information

Site: Hero Air Force Base	SDG #: ABC-1234
Laboratory: TestGood Labs	Date: 08/31/2020
HydroGeoLogic, Inc. Reviewer: Justin Hersh HGL Senior Reviewer: Denise Rivers (09/09/20)	Project: AF0055.001.02.03

Client Sample ID	Laboratory Sample ID	Laboratory Receipt Date	Sampling Date and Time	Matrix
HAFB-MW01	ABC-1234-01	08/01/2020	07/31/20 10:10	Water
HAFB-DUP01	ABC-1234-02	08/01/2020	07/31/20 10:10	Water
TB-08122020	ABC-1234-03	08/01/2020	07/31/20 08:00	Water QC
HAFB-MW02	ABC-1234-04	08/01/2020	07/31/20 12:05	Water
HAFB-EB01	ABC-1234-05	08/01/2020	07/31/20 14:00	Water QC

1a. Narrative and Completeness Review – The case narrative and data package were checked for completeness. It was noted that the laboratory reported its internally derived control limits instead of the QAPP control limits for PCBs and TRPH. The QAPP control limits were used to evaluate the data. No other discrepancies were noted.

Qualification: None required.

1b. Sample Delivery and Condition – All samples arrived intact at the laboratory in acceptable condition and temperature and were properly preserved, as applicable. Proper custody was documented, with one exception. Field duplicate HAFB-DUP01 was incorrectly associated with sample HAFB-MW02 while in the field; the correct parent sample is HAFB-MW01, which will be amended in all field paperwork and the data validation report for this SDG.

Qualification: None required.

1c. Equipment Blanks – One equipment blank, identified as HAFB-EB01, was associated with all samples analyzed for PCBs in this SDG and was free from contamination.

Qualification: None required.

1d. Field Duplicate – Sample HAFB-DUP01 is a field duplicate of sample HAFB-MW01. Detections for the duplicate pair and the calculated RPD or absolute difference, as applicable, are listed in the table below.

ANALYTE	HAFB-MW01		HAFB-DUP01		RPD or |Diff|
	Conc.	LOQ	Conc.	LOQ	
VOCs					
Isopropylbenzene	11	1.0	13	1.0	16.7%
Total Metals					
Antimony	0.5	1.0	0.75	1.0	0.25

ANALYTE	HAFB-MW01		HAFB-DUP01		RPD or |Diff|
	Conc.	LOQ	Conc.	LOQ	
Pesticides					
Dieldrin	23	1.0	24	1.0	4.3%
Wet Chemistry					
Sulfate	5.87	0.5	5.93	0.5	1.0%

Qualification: None required.

Section 2 – Volatile Organic Compounds (SW-846 Method 8260B)

Client Sample ID	Laboratory Sample ID	Analysis Batch
HAFB-MW01	ABC-1234-01	690453
HAFB-DUP01	ABC-1234-02	690453
TB-08122020	ABC-1234-03	690193

2a. Holding Times – All samples were analyzed within the 14-day holding time required by the QAPP for preserved aqueous samples.

Qualification: None required.

2b. Initial Calibration – One initial calibration (ICAL) was associated with all samples in this SDG. The ICAL performed for instrument MSV11 on 08/14/20 (associated with batches 690193 and 690453) had acceptable mean RRFs for all SPCCs and acceptable %RSDs for all CCCs. All target analytes had acceptable RRFs and %RSDs. The second source ICV associated with this initial calibration met the control criteria established by the QAPP for all target analytes.

Qualification: None required.

2c. Continuing Calibration – Two continuing calibration verification (CCV) and two closing CCV standards were associated with the samples in this SDG. The CCV and closing CCV standards analyzed on 08/17/20 for batch 690193 had acceptable CCRFs for all SPCCs and acceptable %Ds for all CCCs. The %Ds for all target analytes met the control limits established by the QAPP.

The CCV and closing CCV standards analyzed on 08/20/20 for batch 690453 had acceptable CCRFs for all SPCCs and acceptable %Ds for all CCCs. The %Ds for all target analytes met the control limits established by the QAPP.

Qualification: None required.

2d. GC/MS Tuning – The sample analytical sequences were all performed within 12 hours of an acceptable GC/MS tune.

Qualification: None required.

2e. Internal Standards – All internal standards met the peak area and retention time criteria.

Qualification: None required.

2f. Surrogates – All surrogate recoveries were within the control limits specified in the QAPP for aqueous samples.

Qualification: None required.

2g. Laboratory Control Sample – Two LCS/LCSD pairs were associated with the samples in this SDG. Both LCS/LCSDs for batches 690193 and 690453 met all %R and RPD control limits established by the QAPP.

Qualification: None required.

2h. MS/MSD – MS/MSD analyses were performed for all target analytes on sample HAFB-MW01 from this SDG. The %R and RPD results were within the QAPP control limits with the exception of 1 high recovery (135%) for the MS. The isopropylbenzene result for parent sample HAFB-MW01 was a detection above the LOQ and should be qualified J.

***Qualification:* The isopropylbenzene result for sample HAFB-MW01 was qualified J.**

2i. Method Blank – Two method blanks were associated with the samples in this SDG. The blanks analyzed on 08/17/20 and 08/20/20 for batches 690193 and 690453, respectively, were free from contamination.

Qualification: None required.

2j. Trip Blanks – One trip blank, identified as TB-08122020, was submitted with the samples in this SDG and was free from contamination.

Qualification: None required.

Section 3 – Total Metals (ICP-MS; SW-846 Method 6020B)

Client Sample ID	Laboratory Sample ID	Preparation Batch	Analysis Batch(1)
HAFB-MW01	ABC-1234-01	695011	695628
HAFB-DUP01	ABC-1234-02	695010	695628
HAFB-MW02	ABC-1234-04	695011	695628

(1) Samples analyzed for total antimony, iron, and lead only.

3a. Holding Times – All samples were analyzed within the 6-month holding time required by the QAPP for preserved aqueous samples.

Qualification: None required.

3b. Calibration – All %R results for the ICV, bracketing CCV, and LDR standards, met the 90-110% recovery criterion for both target metals. The %R results for the low-level CCV standards met the 80-120% QAPP criteria.

Qualification: None required.

3c. Calibration Blanks – The ICBs and CCBs associated with the sample analyses were free from contamination, with one exception. The CCB analyzed on 11/06/20 at 1347 for analysis batch

695628 was contaminated with total antimony (0.73 µg/L), yielding an action level of 3.65 µg/L. The dissolved antimony result for sample HAFB-MW02 was a detection below the action level and should be qualified U.

Qualification: **The total antimony result for sample HAFB-MW02 was qualified U.**

3d. Interference Check Samples – Two ICSA and ICSAB sets were analyzed with the samples in this SDG. All non-spiked target metals results were less than the LOD in the ICSAs. All spiked metals met the 80-120% QAPP control criteria for the ICSAB standards.

Qualification: None Required.

3e. ICP Serial Dilutions/Post Digestion Spike Samples – A serial dilution and/or post digestion spike (PDS) were performed for total metals antimony, iron, and lead on sample HAFB-MW01 from this SDG. All PDS %R results were within the QAPP control limits. All metals were less than 50x the respective LOD, and the serial dilution %D results were not calculated or applicable.

Qualification: None Required.

3f. Laboratory Control Sample – Two LCS standards were associated with the samples in this SDG. The LCS standards for preparation batches 695011 and 695010 met all %R control limits established by the QAPP.

Qualification: None required.

3g. MS/MSD – MS/MSD analyses were performed for total metals antimony, iron, and lead on sample HAFB-MW01 from this SDG. All %R and RPD results were within the QAPP control criteria.

Qualification: none required.

3h. Laboratory Duplicate Sample – A laboratory duplicate analysis was not performed on a sample from this SDG.

Qualification: None required.

3i. Method Blank – Two method blanks were associated with the samples in this SDG. The method blanks for preparation batches 695011 and 695010 were free from contamination.

Qualification: None required.

Section 4 – Polychlorinated Biphenyls (SW-846 Method 8082A)

Client Sample ID	Laboratory Sample ID	Preparation Batch	Analysis Batch
HAFB-MW01	ABC-1234-01	232943	232958
HAFB-DUP01	ABC-1234-02	232943	232958
HAFB-MW02	ABC-1234-04	232943	232958

4a. Holding Times – All samples were extracted and analyzed within the 1 year holding time specified in the QAPP for aqueous samples.

Qualification: None required.

4b. Initial Calibration – All target analytes in the primary and secondary column ICALs had %RSDs less than the method maximum of 20% or r^2 values greater than 0.99. All second source ICV %Ds were less than the method maximum of 20%.

Qualification: None required.

4c. Continuing Calibration – In the instance of PCBs, single peaks are not qualified if the average %D was within the QAPP control limit. All %Ds for CCVs bracketing the samples were less than the 20% method maximum stated in the QAPP.

Qualification: None required.

4d. Internal Standards – All internal standards met the peak area and retention time criteria.

Qualification: None required.

4e. Surrogates – All surrogate recoveries were within the QAPP acceptance limits.

Qualification: None required.

4f. Laboratory Control Sample – One LCS was associated with all samples in this SDG. The LCS for preparation batch 232943 met the %R control limits established in the QAPP.

Qualification: None required.

4g. MS/MSD – Matrix spike/matrix spike duplicate analyses were not requested or performed on a sample from this SDG.

Qualification: None required.

4h. Method Blank – One method blank was associated with all samples in this SDG. The method blank prepared on 01/12/21 for batch 232943 was free from contamination.

Qualification: None required.

4i. Detection Confirmation – All results for the samples in this SDG were non-detect.

Qualification: None required.

Section 5 – Petroleum Range Organics (TRPH; Method FL-PRO)

Client Sample ID	Laboratory Sample ID	Preparation Batch	Analysis Batch
HAFB-MW01	ABC-1234-01	231795	231789
HAFB-DUP01	ABC-1234-02	231795	231789
HAFB-MW02	ABC-1234-04	231795	231789

5a. Holding Times – All samples were extracted within the 7-day holding period required for aqueous samples and analyzed within 40-days of preparation.

Qualification: None required.

5b. Initial Calibration – One initial calibration was associated with the samples in this SDG. The target analyte in the ICAL had a %RSD less than the method maximum of 20% or an r^2 value greater than 0.99. No second source ICV was presented.

Qualification: None required.

5c. Continuing Calibration – Two continuing calibration verification (CCV) standards were associated with the samples in this SDG. All CCV %Ds were less than the 25% method maximum stated in the QAPP.

Qualification: None required.

5d. Surrogates – All surrogate recoveries were within the QAPP acceptance limits.

Qualification: None required.

5e. Retention Times – All retention times met the QAPP criteria.

Qualification: None required.

5f. Laboratory Control Sample – One LCS was associated with the samples in this SDG. The LCS for preparation batch 231795 met the %R control limit established in the QAPP.

Qualification: None required.

5g. MS/MSD – Matrix spike/matrix spike duplicate analyses were performed for TRPH on sample HAFB-MW01 from this SDG. All %R and RPD results met the QAPP control criteria.

Qualification: None required.

5h. Method Blank – One method blank was associated with the samples in this SDG. The method blank prepared on 12/11/20 for batch 231795 was free from contamination.

Qualification: None required.

Section 6 – Polynuclear Aromatic Hydrocarbons (SW-846 Method 8270D-SIM)

Client Sample ID	Laboratory Sample ID	Preparation Batch	Analysis Batch
HAFB-MW01	ABC-1234-01	340410	340438
HAFB-DUP01	ABC-1234-02	340410	340438
HAFB-MW02	ABC-1234-04	340410	340438

6a. Holding Times – All samples were prepared within the 7-day holding time required by the QAPP for aqueous samples and analyzed within 40-days of extraction.

Qualification: None required.

6b. Surrogates – The surrogate recoveries were within the control limits specified in the QAPP for

aqueous samples, with two exceptions. The recoveries for surrogate 2-methylnaphthalene-d10 were below the lower QAPP criteria for samples HAFB-MW01 (38%) and HAFB-DUP01 (24%). All results for both samples were non-detections and should be qualified UJ.

Qualification: **All results for samples HAFB-MW01 and HAFB-DUP01 were qualified UJ.**

6c. Initial Calibration – One initial calibration was associated with the samples in this SDG. For the initial calibration run on 05/13/20, all target analytes had %RSDs less than the method maximum of 20% or r^2 values greater than 0.99. All second source ICV %Ds were within the 80%-120% criteria.

Qualification: None required.

6d. Continuing Calibration – One continuing calibration verification (CCV) and one closing CCV standards were associated with the samples in this SDG. The CCV standards that were associated with the samples in this SDG had %Ds within the QAPP acceptance limits.

Qualification: None required.

6e. GC/MS Tuning – The sample analytical sequences were all performed within 12 hours of an acceptable GC/MS tune.

Qualification: None required.

6f. Internal Standards – All internal standards met the peak area and retention time criteria.

Qualification: None required.

6g. Laboratory Control Sample – One LCS/LCSD pair was associated with the samples in this SDG. The LCS/LCSD for preparation batch 340410 met all %R and RPD control limits established in the QAPP.

Qualification: None required.

6h. MS/MSD – Matrix spike and matrix spike duplicate analyses were performed for all target PAHs on sample HAFB-MW01 from this SDG. The table below lists all MS/MSD recoveries and RPDs that were outside of the QAPP control limits and the appropriate qualification, as necessary.

Parent Sample	Prep Batch	Compound	%R / %R / RPD	Qualifier	Affected Samples
HAFB-MW01	340410	1-Methylnaphthalene	34% / OK / 52%	UJ	1
		2-Methylnaphthalene	29% / OK / 49%	UJ	1
		Naphthalene	18% / 37% / 69%	UJ	1

Qualification: **Please refer to the table above.**

6i. Method Blank – One method blank was associated with the samples in this SDG. The blank prepared on 10/08/20 for batch 340410 was free from contamination.

Qualification: None required.

Section 7 – Organochlorine Pesticides (SW-846 Method 8081B)

Client Sample ID	Laboratory Sample ID	Prep Batch	Analysis Batch
HAFB-MW01	ABC-1234-01	592177	592626
HAFB-DUP01	ABC-1234-02	592177	592664
HAFB-MW02	ABC-1234-04	592177	592626
HAFB-EB01	ABC-1234-05	592177	592626

7a. Holding Times – All samples were prepared within the required 7-day holding period for aqueous samples and analyzed within 40-days of extraction.

Qualification: None required.

7b. Surrogates – All surrogate recoveries were within the QAPP acceptance limits.

Qualification: None required.

7c. Second-Column Confirmation – Pesticide detections require secondary column confirmation. The RPD calculated from corresponding primary and secondary column heptachlor epoxide results for sample HAFB-MW02 was less than the 40% QAPP criteria.

Qualification: None required.

7d. Initial Calibration – One initial calibration was associated with the samples in this SDG. The target analyte had a %RSD less than the method maximum of 20% or an r^2 value greater than 0.99 for both standards. The second source ICV %Ds were less than the method maximum of 20%.

Qualification: None required.

7e. Continuing Calibration – Two continuing calibration verification (CCV) standards were associated with the samples in this SDG. All CCV %Ds for the target analyte were less than the 20% method maximum stated in the QAPP.

Qualification: None required.

7f. Breakdown Check – The degradation of endrin and 4,4'-DDT was ≤15% as specified in the QAPP.

Qualification: None required.

7g. Retention Time Window – All target analytes met the retention time criteria established in the QAPP.

Qualification: None required.

7h. Laboratory Control Sample – One LCS/LCSD pair was associated with all samples in this SDG. The LCS/LCSD for preparation batch 592177 met all %R and RPD control limits established in the QAPP.

Qualification: None required.

7i. MS/MSD – Matrix spike and matrix spike duplicate analyses were performed for all target analytes on sample HAFB-MW01 from this SDG. All %R and RPD results met the criteria established by the QAPP.

Qualification: None required.

7j. Method Blank – One method blank was associated with the samples in this SDG. The method blank prepared on 08/08/16 for batch 592177 was free from contamination.

Qualification: None required.

Section 8 – Sulfate (SW-846 Method 9056A)

Client Sample ID	Laboratory Sample ID	Analysis Batch
HAFB-MW01	ABC-1234-01	654604
HAFB-DUP01	ABC-1234-02	654604
HAFB-MW02	ABC-1234-04	654604

8a. Holding Times – All samples were analyzed within the 28-day holding time required by the QAPP for aqueous samples.

Qualification: None required.

8b. Calibrations – The initial calibration performed on 07/11/20 met the criteria established by the QAPP. All %R results for the bracketing CCV standards met the 90-110% recovery criterion for sulfate.

Qualification: None required.

8c. Calibration Blanks – All CCBs associated with the sample analyses were free from contamination.

Qualification: None required.

8d. Method Blanks – One method blank was associated with all samples in this SDG. The method blank analyzed on 08/23/20 for batch 654604 was free from contamination.

Qualification: None Required.

8e. Laboratory Control Sample – One LCS sample was associated with all samples in this SDG. The LCS result for batch 654604 met the %R requirements established by the QAPP.

Qualification: None required.

8f. MS/MSD – Matrix spike and matrix spike duplicate analyses were performed for sulfate on sample HAFB-MW01 from this SDG. All %R and RPD results met the QAPP criteria.

Qualification: None required.

8g. Laboratory Duplicate Sample – A laboratory duplicate analysis was not performed on a sample from this SDG.

Qualification: none required.

Section 9 – Compound Quantitation

Analyte non-detections are reported as the LOD and qualified U. These U qualifiers are retained unless superseded by a more severe qualifier. Analytes detected between the LOQ and DL are reported as either J- or I-qualified results by the laboratory. The I-qualifiers are changed to J flags per the QAPP requirements and these J qualifiers are retained unless superseded by a more severe qualifier. The non-standard M-qualifiers applied by the laboratory to indicate the manual integration of results should be removed from all samples.

Qualification: **All non-standard I-qualifiers applied by the laboratory were changed to J flags. The non-standard M-qualifiers applied by the laboratory were removed from all samples.**

Qualification Summary Table (all concentrations in mg/L or µg/L depending on the method):

Sample	Analyte	Lab Value	Lab Qualifier	HGL Value	HGL Qualifier
HAFB-MW01	Isopropylbenzene	21.4	--	21.4	J
	All PAH results	Varies	U / UM / UJ1 / UMJ1	Varies	UJ
HAFB-DUP01	All PAH results	Varies	U / UM	Varies	UJ
HAFB-MW02	Antimony, total	0.73	I	0.73	U
	Iron, total	83.7	I	83.7	J
	All PAH results	Varies	UM	Varies	U

Only environmental samples and field duplicates are included in the above table. Field blanks are used to evaluate the sample data but are not qualified during the review process.

PFAS Stage 2A Data Validation Checklist

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3
Project Name: Off-Base Drinking Water Site Inspection, USAF Installations, Multiple Sites
Sample Delivery Group: FA82510

Laboratory ID	Sample ID	Received	Collected		Matrix	Sample Type
ABC-1234-01	HAFB-MW01	1/21/2021	1/20/2021	13:45	Water	Normal
ABC-1234-02	HAFB-DUP01	1/21/2021	1/20/2021	13:22	Water	Field Duplicate
ABC-1234-04	HAFB-MW02	1/21/2021	1/20/2021	13:23	Water	Normal

	Yes	No	NA	Comments
I. Case Narrative/Sample Receipt/Holding Times				
Were all samples listed on the COC reported with the correct sample IDs?	●	○	○	
Did the case narrative include any issues that impact the data validation?	○	●	○	
Were samples received in proper containers and properly preserved?	●	○	○	
Were there any discrepancies noted at sample receipt?	○	●	○	
Were all samples listed on the COC analyzed?	●	○	○	
Were all holding times met?	●	○	○	
II. DoD QSM Specified Ion Transitions				
Were the ion transitions those specified in QSM Table B-15 (below)? PFOA: 413 → 369 PFOS: 499 → 80 PFHxS: 399 → 80 PFBS: 299 → 80 4:2 FTS: 327 → 307 6:2 FTS: 427 → 407 8:2 FTS: 527 → 507 NEtFOSAA: 584 → 419 NMeFOSAA: 570 → 419	●	○	○	
III. Extracted Internal Standard (EIS) Recoveries				
Were EIS recoveries within the control limits specified in the QAPP or 50-150%, if QSM limits used)?	●	○	○	
Were EIS retention times within 0.40 minutes of retention time of midpoint std in ICAL or initial CCV?	●	○	○	
IV. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	●	○	○	
Were the laboratory blanks free of contamination?	○	●	○	The MB was contaminated with 2.4 ng/L PFOS. All three PFOS detections were greater than the action level, and no qualification was required.
V. Field blanks				
Were field blanks included in this SDG?	●	○	○	
Were target compounds detected in the field blanks?	○	●	○	

VI. Equipment blanks				
Were equipment blanks included in this SDG?	○	◉	○	
Were target compounds detected in the equipment blanks?	○	○	◉	
VII. Matrix spike/Matrix spike duplicates				
Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG?	○	◉	○	An MS and laboratory duplicate from another site were reported with the samples in this SDG.
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	○	○	◉	All recoveries were within control for the batch MS. For the laboratory duplicate, the absolute difference of the PFHxS results met the criteria.
VIII. Laboratory control samples				
Was an LCS/LCSD analyzed per extraction batch for this SDG?	◉	○	○	No LCSD.
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	◉	○	○	
IX. Field duplicates				
Were field duplicate pairs identified in this SDG?	◉	○	○	
Did the field duplicate meet the criteria specified in the QAPP?	◉	○	○	
X. Compound quantitation				
Did the reported list of analytes include all those specified in the QAPP?	◉	○	○	
Did the laboratory reporting limits (i.e. DL, LOD, LOQ) meet the QAPP?	◉	○	○	
Did reported results include both branched and linear isomers?	◉	○	○	
XI. Overall assessment of Data				
Overall assessment of data was found to be acceptable.	◉	○	○	

Reviewer: John Powell	Date: 02-07-2021	Second Reviewer: Denise Rivers	Date: 02-08-2021

Table 1: Qualification Summary (all concentrations in ng/L):

Sample ID	Analyte	Lab Concentration	Lab Qualifier	HGL Concentration	HGL Qualifier

The following provides a brief explanation of the data validation qualifiers assigned to results during the data review process by the data validator.

Qualifier	Definition
U	The analyte was not detected and was reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
J	The reported result was an estimated value with an unknown bias.
J+	The result was an estimated quantity, but the result may be biased high.
J-	The result was an estimated quantity, but the result may be biased low.
N	The analysis indicates the presence of an analyte for which there was presumptive evidence to make a "tentative identification."
NJ	The analyte has been "tentatively identified" or "presumptively" as present and the associated numerical value was the estimated concentration in the sample.
UJ	The analyte was not detected and was reported as less than the LOD or as defined by the customer. However, the associated numerical value is approximate.

X	The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Acceptance or rejection of the data should be decided by the project team (which should include a project chemist), but exclusion of the data is recommended.

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ATTACHMENT C
General Data Validation Conventions

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ATTACHMENT C
General Data Validation Conventions

1.0 INTRODUCTION

The general conventions presented below describe the evaluation and qualification process applied to project data undergoing a Stage 2A or Stage 2B data validation. The data validator should always use the Quality Assurance Project Plan (QAPP) as the primary source for project-specific validation requirements. Where the general conventions presented below conflict with the requirements presented in the QAPP, the QAPP requirements should take precedence. Situations that are not covered by the project QAPP or by the general conventions should be referred to a HydroGeoLogic, Inc. (HGL) senior chemist for resolution.

Note that the guidance presented in this attachment assumes that the project QAPP presents validation and qualification criteria based on the quality control (QC) requirements of the U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM), version 5.3. Laboratory certification under the DoD Environmental Laboratory Accreditation Program is performed under the requirements of the QSM version current at the time of certification. This recertification process is on an approximately 18-month cycle. As a result, some project QAPPs will cite the version of the QSM that was in effect at the time of the project laboratory's accreditation; also, there are still QAPPs in use that have data qualification protocols based on the QC requirements of older versions of the QSM. If the guidance presented in this attachment conflicts with the project QAPP qualification protocols, the requirements of the project QAPP should take precedence unless alternative direction is received from the client project manager. As additional versions of the DoD QSM are issued, new project QAPPs will incorporate the most up-to-date DoD requirements consistent with project laboratory certification status.

2.0 SENSITIVITY LIMITS

The principal reasons for assigning data qualifiers are the magnitude of detected results relative to the associated sensitivity limits and the conventions for reporting nondetected results. There are two principal conventions for establishing sensitivity limits, the conventions originally established by the U.S. Environmental Protection Agency (EPA) to support the Contract Laboratory Program (CLP) and the conventions established by DoD. Both are in common use and are described below. Table C.1 presents the DoD terms, their definitions, and the corresponding EPA terms that are also in common usage.

Table C.1
Sensitivity Limit Definitions[(1)]

Sensitivity Limit Term	Definition	Corresponding EPA Terms
Detection limit (DL)	The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.	Method detection limit (MDL)
Limit of detection (LOD)	The smallest amount or concentration of a substance that must be present in a sample to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. An LOD may be used as the lowest concentration for reliably reporting a nondetect of a specific analyte in a specific matrix with a specific method at 99% confidence.	--
Limit of quantitation (LOQ)	The lowest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ is set at or above the concentration of the lowest initial calibration standard and within the calibrated range.	Reporting limit Quantitation limit Practical quantitation limit Method quantitation limit Contract-required detection limit Contract-required quantitation limit

(1) Terms and definitions are from Section 3.1 of the QSM, version 5.3 (May 2019).

2.1 EPA SENSITIVITY LIMIT CONVENTIONS

The EPA convention involves setting a concentration limit above which analytical results are considered to be of sufficient quantitative significance to be reported without qualification (unless affected by QC issues). In practice, this limit is established at or above the low point on the calibration curve for each target analyte. A variety of terms has been applied to this limit, including reporting limit (RL), practical quantitation limit, and method quantitation limit. EPA's CLP uses the term contract-required quantitation limit, although historical data may include the term contract required detection limit (CRDL) applied to inorganic results. Results between the MDL and RL are reported as detections qualified as estimated due to being below the calibrated range. Results below the MDL are considered nondetected results and are reported as the numerical value of the MDL or the RL (depending on project-specific requirements) qualified U.

For many of HGL's DoD projects, the EPA sensitivity limit conventions have been superseded by the DoD conventions described in Section 2.2; however, most projects performed for non-DoD clients will still use the EPA conventions. Older DoD projects with existing basewide QAPPs also may retain the use of EPA conventions to maintain comparability with the existing project dataset or to comply with state or permit data reporting requirements.

2.2 DOD SENSITIVITY LIMIT CONVENTIONS

The current DoD sensitivity limit conventions were introduced in version 4 of the QSM in April 2009 and have remained in use in subsequent versions of the QSM. QSM version 4 established a three-tiered system of DL, LOD, and LOQ. The QSM provides definitions for all these terms; however, in practical applications, the DL and LOQ are used in an analogous fashion as the MDL and RL, respectively, are used in the EPA sensitivity conventions. Results between the DL and LOQ are reported as detections qualified as estimated due to being below the calibrated range. The LOD term was introduced in QSM version 4 and corresponds to the lowest level that can be present in a sample and have a 99 percent probability of being detected in that sample. In the DoD conventions, results below the DL are considered nondetected results and are reported as the numerical value of the LOD qualified U.

3.0 DATA QUALIFIERS

Each validated result consists of three components: (1) a numerical value that corresponds to a concentration, (2) a data qualifier, and (3) the concentration units. The concentration can correspond to a detected value or to a proxy value used for nondetected results in that is assigned accordance with the conventions presented in the project QAPP. The data validation process generally focuses on the application of the appropriate data qualifier on each result. Some projects will require a change to the numerical concentration presented under specific circumstances (see Section 3.2.4).

Data qualification indicates that an analytical result falls into one of three broad categories: (1) usable; (2) usable but estimated; and (3) unusable. The validation conventions presented below do not present specific qualification requirements. The qualifiers to be used for a project will be defined in that project's QAPP. The allowed final data qualifiers will be defined depending on the client and the regulatory body that will be the final recipients of the data. Descriptions of commonly applied data qualifiers are presented below, but the data validator must use the qualification requirements specified in the QAPP for each project.

The most used data qualification conventions for DoD projects will be based on those qualifiers listed and defined in the DoD General Data Validation Guidelines.

3.1 LABORATORY-APPLIED FLAGS

In some cases, data points may be reported by the laboratory with one or more informational flags, such as an alphanumeric code or a symbol. These flags are not considered valid qualifiers and should be automatically removed from all affected data points, with the exceptions noted in Sections 3.2.2, 3.2.4, and 3.3.1 below. In some cases, the laboratory-applied informational flag will mimic a valid final qualifier but may or may not be applicable as the final qualifier. In such cases, the validator's discussion of the effect of a QC discrepancy on the associated results should

also include a discussion of whether laboratory-applied flags that mimic a valid qualifier should be retained, deleted, or altered. All residual laboratory-applied flags that are not accepted as the final qualifier by the data validator must be removed from the electronic data at what is referred to as the "100 percent QC stage" of data upload and incorporation into the project database (see Section 6.0 of the standard operating procedure [SOP]).

3.2 QUALIFICATION OF DETECTED RESULTS

3.2.1 Detected Results Not Requiring Qualification

Results that are detected within the calibrated range of the instrument and that are not associated with a QC discrepancy will be accepted by the validation process as the numerical value of the concentration (with appropriate units) and without any data qualifier.

3.2.2 Detected Results below the Calibrated Range

Detected results with concentrations equal to or greater than the DL but below the LOQ (corresponding to the lower limit of the calibrated range of the instrument) are considered to be estimated results by default. Laboratories report such results with an informational flag to indicate that the result is below the calibrated range. This informational flag is most often a "J," "B" (CLP convention for inorganic results), or "I" (Florida Department of Environmental Protection convention). In some cases, these flags correspond to commonly used final qualifiers that are applied to such results. When the laboratory assigns a flag that corresponds to the project qualification convention, the assigned flag can be accepted as the final qualifier by the validator if no other qualification is required for a QC issue. In other cases, the validator will need to specify that, absent any other qualification on specific results, the laboratory's default flag for a detected result below the LOQ is globally changed to the project-specific qualifier.

3.2.3 Detected Results Requiring Qualification as Estimates

Detected results affected by QC issues will be qualified as estimated values as required by the project validation guidelines. The most common qualifier used to indicate an estimated result is "J," although it is common for projects to use alternative qualifiers if the overall direction of bias can be determined. These alternative qualifiers can include the DoD qualifiers "J+" if the bias is high, or "J–" if the bias is low.

3.2.4 Detected Results Requiring Qualification as Artifacts

One of the goals of data validation is to determine if detected concentrations of analytes reported in samples are representative of site conditions. Detected concentrations reported by the laboratory that are artifacts of the sampling, shipping, storage, preparation, and analytical processes that the sample undergoes are not representative of the site and must be identified by the validator. The most common procedure to identify results as artifacts is to apply the qualification of "U."

In addition to being used to identify artifacts under some conventions, the U qualifier is almost universally used to identify nondetected results (see Section 3.3.1). When the U qualifier is used both as a laboratory qualifier for identifying nondetects and as a validator qualifier for identifying artifacts, the final qualifier will not allow the data user to determine whether the analyte in question is a nondetection or was determined to be an artifact. However, artifacts are treated in the same fashion as nondetections for most end uses of analytical data, so in practice this convention does not introduce unacceptable ambiguity into interpreting the qualified result. The quantitated value associated with the U qualifier assigned to an artifact can be the originally reported detected value, the LOD, or the LOQ (or equivalent), depending on the data reporting conventions presented in the project QAPP. For projects using the DoD sensitivity limit conventions, results qualified U as artifacts that have a concentration that exceeds the DL but are lower than the associated LOD will have the reported concentration changed at a minimum to the value of the LOD or to a higher value as directed by the data validation protocols.

3.3 QUALIFICATION OF NONDETECTED RESULTS

3.3.1 Nondetected Results Not Requiring Qualification

Nondetected results receive a final qualifier of U in almost every data qualification convention. Depending on the requirements of the QAPP, the quantitated value associated with the U qualifier can either be the DL (or equivalent), the LOD, or the LOQ (or equivalent). The reporting conventions to be used for each project should be included in the project QAPP and should be confirmed with the laboratory prior to generating project results. For most projects, a large majority of the reported laboratory results will be nondetections. Ensuring that the laboratory will report nondetected data flagged U using the same protocols as are required for the final U qualification will allow the data validator to retain the laboratory flags unchanged.

Some laboratories report nondetected results as "ND" or as "<#," where # represents a number that can be the DL (or equivalent), LOD, or LOQ (or equivalent). The data validation report should indicate that such results are considered to be the equivalent of results qualified U according to the project conventions, unless superseded by a more severe qualifier.

3.3.2 Nondetected Results Requiring Qualification as Estimated

Nondetected results affected by QC issues will be qualified as estimated values as required by the project validation guidelines. The most common qualifier used to indicate an estimated result is the combination qualifier "UJ." Nondetected results are not considered to be affected by high bias or precision discrepancies (except when reported as part of a duplicate or triplicate set of analyses that also includes detections of the affected analyte). As with nondetected results not requiring qualification, the quantitated value associated with the qualified result can be the DL (or

equivalent), the LOD, or the LOQ (or equivalent), depending on the project conventions for reporting nondetected results.

3.4 REJECTED RESULTS

Data points affected by severe QC discrepancies are potentially unusable for their intended purposes as described in the project data quality objectives. The data qualification guidelines presented in the QAPP establish the circumstances under which data is rejected or otherwise noted as suspect by the validator. Any data rejected or identified as suspect in the data validation process should be evaluated by the HGL project chemist and the project team to determine if a final qualifier of R should be applied or if a less severe qualifier can be justified. If a less severe qualifier is selected for the affected results, the technical rationale must be included in the HGL data validation report (internal data validation) or the HGL data validation report review memo (subcontracted data validation). The technical rationale must also be included in any data quality evaluation provided as part of the project deliverables (see Section 3.3 of the main body of this SOP).

A result that receives a final qualifier of R should have the "Report Usability" field in the associated electronic file populated with Y. The Report Usability field should only be populated with N if the result is superseded by another result (see Section 3.5 below).

3.4.1 Rejection of Detected Results

Most data qualification conventions will not require rejection of detected results unless severe instrumental or systematic deficiencies are identified. Detected results with extreme high or low bias that are compromised by severe discrepancies in sample collection or shipment or that were generated while the analytical system was unacceptably compromised will not be of sufficient quality to be incorporated into a quantitative risk assessment. In some cases, however, data points rejected in accordance with the validation protocols may have limited usability.

> *Example*: A detected result is associated with a severe low bias, but the result is greater than the screening level for the site. Although the validation protocols indicate this result should be rejected, the affected result could be used to determine if that compound were a contaminant of concern at the site if it was above the associated screening value. However, the numerical value could be too compromised to be incorporated into the quantitative determination of risk at the site.

Rejected detected results are qualified R; quantitated values should not be reported in association with a result qualified R.

3.4.2 Rejection of Nondetected Results

Nondetected results are generally rejected under more circumstances than detected results. This is because most projects consider a Type II (false negative) error to be a more severe error than a Type I (false positive) error. Rejected nondetected results are qualified R; quantitated values should not be reported in association with a result qualified R.

3.4.3 DoD Data Rejection Conventions

The most recent DoD data qualification conventions (DoD EMDQ, 2019) include an X flag. The X flag is intended to be used as an interim qualifier that replaces the R qualifier at the data validation stage and is replaced by the R qualifier or a less severe qualifier at the data usability stage. HGL's multiple stages of data validation review and the data usability assessment procedures included in project QAPPs are analogous to the intended use of the DoD X flag. HGL's procedures ensure that data qualified R during the validation process are subject to additional technical evaluation to determine if the R qualifier is an appropriated final qualifier. While many current HGL QAPPs indicate that the data validator should apply R qualifiers pending further review, new QAPPs for DoD clients should incorporate the most recent DoD data qualifiers, including the use of the X flag as an initial qualifier at the validation stage.

3.5 QUALIFICATION OF EXCLUDED RESULTS

In cases where multiple analysis results are reported for a sample due to dilution or reanalysis, all analyses are to be reviewed. Based on the body of QC data, the validator should select one definitive result for each analyte in each sample, and all other results for that analyte in that sample are denoted as superseded by applying an # qualifier.[2] Clearly indicating results that are not to be used with an # assists in managing data for report preparation and database submittal. Results that receive an # qualifier do not need to be further validated or qualified; however, the validation narrative should include the rationale for selecting the definitive result. Results receiving an # qualifier should be included in the data qualification table in each validation report, with the analysis receiving the qualification clearly differentiated from the other analyses performed on the same sample. Where large categories of results in a sample analysis receive an # qualifier, this qualifier may be noted for the class of results (for example, "All nondetections") instead of as an analyte-by-analyte listing. Applying an # qualifier may result in the data for the full analyte list for a particular sample being composed of results from multiple analyses. For example, in an original analysis/diluted analysis pair, all analytes in the original analysis are considered definitive except for those analytes that exceeded the calibrated range, which are reported from the diluted analysis.

[2] HGL previously applied an X qualifier. In the most recent DoD data validation guidance (DoD EMDQ, 2019), X is an interim data flag to be applied instead of R at the validation stage.

3.6 RESULTS WITH MULTIPLE APPLICABLE QUALIFIERS

Some results may be affected by more than one QC discrepancy. In such cases, the final qualifier applied to each result is the highest priority qualifier as defined by the project QAPP.

When "U" is used the qualifier to denote an artifact, the validator should treat the associated result as a detection when evaluating additional qualification for other QC issues.

> *Example*: A result is determined to be an artifact and the conventions call for that result to be qualified U. Another QC issue also affects that result, and the qualification conventions call for a detected result to be qualified J and a nondetected result to be qualified R or X. The validator should apply UJ as the final qualifier instead of R or X to any affected results that were originally reported as detections but have been qualified U as a result of being considered an artifact. However, once the data validation stage is complete, the Detected field in the electronic data deliverable should be populated with N in accordance with Section 3.3.2 above.

4.0 STAGE 2A QC ELEMENTS

The following are general guidelines for reviewing the QC elements identified as Stage 2A QC elements in Attachment A. Final qualification will be applied in accordance with the QAPP. As Stage 2A data validation includes the components of a Stage 1 data review, the Stage 1 components are included in the requirements for Stage 2A validation.

4.1 CASE NARRATIVE

Qualification is usually not required based on the results of the case narrative; however, the validator should review the narrative prior to beginning validating the data package. The narrative can assist in identifying QC issues, describe corrective action or causes for QC discrepancies, describe sample receipt discrepancies, and indicate any special client instructions for the sample analyses. In the data validation report, the validator should include any items of note that were in the narrative, as well as indicate if there were any errors or omissions in the laboratory narrative.

4.2 CHAIN OF CUSTODY

Review the chain of custody (CoC) form and verify that there are no discrepancies. Some general issues can include difficult-to-read sample IDs, crossed-out items, incorrect analyses requested, incorrect or missing time of collection, and missing or incorrect preservative information. The laboratory also may indicate additional information on the CoC form such as special client requests, sample receipt temperature, and samples added or deleted from those requested on the chain. Generally, results are not qualified based on the CoC form alone; however, this information can be useful to the validator.

4.3 SAMPLE RECEIPT AND LOG-IN FORMS

This form should be checked for discrepancies in sample temperature and sample preservation; discrepancies between the sample labels and the CoC forms; missing, broken, or damaged bottles; and bubbles in containers that should have zero headspace. Results may be qualified based on sample receipt and condition.

Some methods, such as metals and volatile organic compounds (VOC), allow for alternatives if preservation requirements are not met. Aqueous VOC samples must be submitted with zero headspace; however, samples may arrive at the laboratory with some headspace. A VOCs sample with headspace is considered to be acceptable if the bubble in the vial is less than "pea-sized" (defined as approximately ¼ inch or 6 millimeters). If larger bubbles or headspace is observed in VOC samples, this may be an indication of a reaction of the acid preservative with the sample matrix causing effervescence. The HGL project manager should be alerted as soon as possible so that corrective action can be implemented, including resampling or eliminating preservative in future VOC samples collected from the affected locations.

Although it is good practice to ship all samples iced, temperature discrepancies are less likely to affect persistent organic compounds like polynuclear aromatic hydrocarbons, pesticides, and polychlorinated biphenyls (PCBs); temperature discrepancies should have minimal to no effect on metals samples. If the samples were delivered to the laboratory by courier on the same day they were collected, the samples may not have had enough time to chill to the acceptance range (0 to 6 degrees Celsius [°C]). In such cases, the sample temperature is considered to be compliant if the samples arrived at the laboratory iced and were refrigerated on arrival.

Current EPA guidance (EPA, 2014) allows for acid-preserved aqueous metals samples to be shipped and stored at ambient temperature. Soil samples collected by incremental sampling methodology are dried at ambient temperatures over a period of days at the laboratory. Although individual QAPPs may specify temperature requirements for these samples, the impact the samples arriving at the laboratory >6°C is negligible and this should be considered by the validators when evaluating the effect on the analytical results.

4.4 SAMPLE ID CROSS REFERENCE

Review the laboratory listing of HGL sample identifications (IDs) against the CoC form. Common errors involving letter/numeral substitutions include "0" and "O" or "D"; "5" and "S"; "6" and "G"; and "8" and "B." Another common error is inconsistencies in incorporating dashes or spaces in sample IDs.

Errors can occur at sample login when the parent sample and the requested matrix spike (MS) and matrix spike duplicate (MSD) samples are submitted in using an ID format that inserts "MS" and "MSD" into a long string of alphanumeric characters: "PARENTSAMPLEID,"

"PARENTMSSAMPLEID," and "PARENTMSDSAMPLEID." When there is no clear indication that a sample is an MS or an MSD sample, the laboratory log-in department may not notice that the sample IDs are indicating an MS or MSD, causing these samples to be logged in as "normal" samples. The result is that instead of results for parent sample and an MS/MSD pair, the samples are analyzed as a sample triplicate. In such cases, the laboratory log-in department should be notified to be alert for such sample IDs, and the HGL project manager should be alerted that more explicit instructions should be provided to the laboratory when submitting MS/MSDs.

4.5 HOLDING TIMES

The holding times for preparation and analysis for each analytical method should be presented in the project QAPP. Holding times expressed in hours are evaluated based on time of collection to time of preparation or analysis, as measured in hours and holding times expressed in days are evaluated based on calendar days elapsed, with the sampling date considered day "0."

The validator should be aware that time zone difference and daylight savings time need to be accounted for when evaluating holding time to the hour. Also, some sampling teams assign a "dummy" sample collection time (such as "1200") to field duplicate samples. Before qualifying field duplicate sample results for a holding time exceedance of less than a day, the validator should verify the actual sample collection time with the field team.

The validator has some discretion to consider a holding time exceedance to be nominal and determine that qualification is not necessary.

4.6 LCS/LCSD RECOVERIES AND PRECISION

As discussed in Section 3.2 of the SOP, the validator should verify that the control limits reported by the laboratory match those required in the project QAPP. Note that laboratory control sample duplicates (LCSD) are not a QC element required by any analytical methods; however, reporting an LCSD in association with a laboratory control sample (LCS) is a common laboratory practice. When LCSDs are reported, the accuracy performance should be evaluated in the same manner as the associated LCS, and discrepancies in either the LCS or LCSD should be considered grounds for qualifying associated data. In some cases, however, the validator can consider acceptable performance in the LCS or LCSD as a mitigating factor and reduce the severity of the data qualifier applied to associated results for a discrepancy in the other member of the LCS/LCSD pair. The decision to reduce the severity of the data qualifier in this instance should be discussed in the data validation report.

LCSs (and LCSDs) should be spiked with the full list of target analytes unless the QAPP specifically allows for the use of a shorter list. The exception is in the analysis of PCBs. Because there are multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs LCSs are spiked with a standard containing only PCB-1016 and PCB-1260. Generally, discrepancies

shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

LCS/LCSD recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) the validator should consider whether an analytical system problem has occurred. If the cause for abnormally high recoveries is not noted in the case narrative, the validator should contact the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

When LCS/LCSD precision (the reported relative percent difference [RPD]) does not meet the requirements for an analyte, detected results for the affected analyte should be qualified in the associated samples. Nondetected results generally do not require qualification for LCS/LCSD precision discrepancies.

4.7 MS/MSD RECOVERIES AND PRECISION

The evaluation of MS/MSDs is generally the same as the evaluation performed on LCSs and (if performed) LCSDs. Given that MS/MSDs are intended as verification that the laboratory can detect target analytes in the project-specific sample matrix, only MS/MSD analyses performed on HGL-collected samples from the same site (or installation) are considered applicable to the associated sample results. Laboratories often report MS/MSD results from a different sample delivery group (SDG) as batch control without the client sample ID. When a batch control MS/MSD is reported, the validator should use the laboratory sample ID to confirm whether the MS/MSD is actually from a site sample reported in a different SDG or from a non-site sample. If the MS/MSD is from a site sample, it will be considered applicable to associated results. If the MS/MSD cannot be associated with a site sample, it is sufficient to indicate that that one or more reported MS/MSDs were performed on non-project samples and were not used to evaluate the data. No qualification should be applied based on discrepancies in non-project MS/MSDs unless the underlying cause of the discrepancy is suspected to be a problem with the analytical system.

MS/MSD recovery discrepancies in samples that have concentrations of the affected target analytes greater than 4 times the spiked concentration are not considered applicable; this is commonly referred to as the "4 times rule." However, in many cases, the RPD for such MS/MSDs can still be evaluated and used to qualify associated results.

Some laboratories compare the concentrations detected in the MS and the MSD to calculate precision rather than compare the percent recoveries. This convention can cause RPDs to be an incorrect representation of the analyte-specific precision if the spiked concentration in the MS

differs substantially from the spiked concentration in the MSD. The validator should examine the MS and MSD spike concentrations to determine if the reported RPD, calculated using a direct comparison of the detected concentrations, is not relevant. The validator should verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is the same as in the MSD. If the RPDs are calculated using noncomparable spike concentrations, the validator should use alternative means, such as comparing the reported MS and MSD percent recoveries, to determine if precision criteria were met.

Dilution should reduce or eliminate matrix effects and MS/MSD discrepancies in cases where the MS and/or MSD were diluted require some interpretation on the part of the reviewer to determine whether there is actually a matrix effect or whether some other factor is contributing to the discrepancy. In cases where MS/MSD recoveries are calculated from spike recoveries that are above the calibrated range, the reviewer should evaluate whether any discrepancies are a result of matrix effects or are a result of the inherent unreliability of such results.

MSs (and MSDs) should be spiked with the full list of target analytes unless the QAPP specifically allows for the use of a shorter list. The exception is in the analysis of PCBs. Because of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs MS/MSDs are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

For some methods, it is permissible to analyze a single MS as a check for accuracy and use a laboratory duplicate as the check for precision. Laboratory duplicate evaluation is discussed under field duplicates (Section 4.11). If the laboratory performs both an MSD and a laboratory duplicate, both should be evaluated and used to qualify associated results. As with MSs and MSDs, laboratory duplicate results may be from a site sample reported in another SDG or from a non-site sample, and the validator should determine the applicability of laboratory duplicate results reported from other SDGs.

The qualification of results for MS/MSD discrepancies is project- and method-specific. Generally, inorganic and wet chemistry MS/MSD results are considered to be associated with all environmental samples in the same preparation batch and organic MS/MSD results are considered to be associated only with the parent sample.

The QAPP should include additional instructions for evaluating and qualifying results based on MS/MSD discrepancies. Nondetected results generally do not require qualification for MS/MSD precision discrepancies. MS/MSD recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) that are not attributable to native analyte concentration or matrix effects, the validator should consider whether an analytical system problem is occurring. If the cause for

abnormally high recoveries is not noted in the case narrative, the validator should contact the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

4.8 SERIAL DILUTIONS AND POST-DIGESTION SPIKES

For DoD projects, serial dilution and post-digestion spike (PDS) analyses are only required for metals analyses and only if the MS/MSD shows discrepancies. Data are not qualified based on serial dilution or PDS results alone; they are used to supplement the overall evaluation of matrix effects if the MS/MSD shows discrepancies or is not applicable due to an elevated target analyte concentration in the parent sample (greater than 4 times the spike concentration). Serial dilution results are applicable to target analytes that are present in the MS/MSD parent sample at or above 50 times the laboratory's default (undiluted) LOQ and PDS results are applicable to target analytes that are present in the MS/MSD parent sample at less than 50 times the laboratory's default LOQ. The evaluation of MS/MSD recoveries, PDS recoveries, and serial dilution percent differences and the qualification conventions will be specified by the project QAPP.

PDS results are subject to the same "4 times rule" that is used for MS/MSDs. There may be some situations where the MS/MSD and PDS results are out of control but are not applicable because of the 4 times rule, but the parent sample is below the 50 times LOQ rule for serial dilution results to be applicable. In such cases, the validator must evaluate the matrix data as a whole and decide whether qualification for matrix effects is required.

Other methods may require PDSs as method-specific QC elements. The evaluation requirements for non-metals PDSs will be included in the project QAPP, and generally these PDSs can be used alone to qualify data.

4.9 METHOD BLANKS

HGL's QAPPs list acceptance criteria for method blanks. These acceptance criteria are the levels above which blank contamination necessitates that the laboratory performs corrective action. However, *all* method blank concentrations that are greater than the associated DL or have a negative concentration with absolute value greater than the associated DL should be used to qualify the associated sample results. The data validator should note any concentrations of target analytes detected in method blanks that are greater than the associated acceptance limits, including metals method blanks showing negative concentrations with absolute value greater than the acceptance limits.

Target analyte concentrations detected in method blanks should be multiplied by 5; this calculated value is called the artifact threshold.[3] Concentrations of these analytes in associated samples that are less than the artifact threshold are considered artifacts and are qualified in accordance with the QAPP.

Concentrations of common laboratory contaminants are multiplied by 10 instead of 5 to determine the artifact threshold. Common laboratory contaminants for VOCs include methylene chloride, acetone, and 2-butanone (methyl ethyl ketone). Common laboratory contaminants for semivolatile organic compounds (SVOCs) are the phthalate esters.

When comparing method blank action levels to sample concentrations, the artifact threshold should be adjusted to account for sample-specific information, including percent moisture, subsample size, and dilution factor. Often, the easiest way to determine a sample-specific adjustment is to compare the LOQ of a target compound in the sample to the LOQ for that compound in the method blank.

> *Example*: Toluene is detected in a method blank at 4.3 micrograms per kilogram (µg/kg). The toluene LOQ is 5 µg/kg in the method blank and 7.4 µg/kg in sample ABC123. The sample-specific artifact threshold for toluene is 4.3 x (7.4/5) x 5 µg/kg = 32 µg/kg.

In most cases, it will be readily apparent that a result is above or below an artifact threshold and this sample-specific adjustment is necessary for only a minority of comparisons.

4.10 FIELD BLANKS

Field blanks are evaluated in a similar manner as method blanks (Section 4.8). Two main differences are (1) the artifact threshold calculated from concentrations in field blanks is *not* adjusted for sample-specific factors; and (2) most field blanks are aqueous and conversion to equivalent solid units is not straightforward for some analytical methods.

When evaluating the effect of aqueous field blank results on associated aqueous field samples, the artifact threshold associated with field blank contamination is 5 times the concentration detected in the blank (10 times the concentration in the case of common laboratory contaminants). When evaluating the effect of aqueous field blank results on associated solid matrix field samples, the field blank results must first be converted to the equivalent solid concentration.

[3] Note that the term "action level" was previously used to describe this value; the use of the term action level is discouraged because that term is also used in site characterization and has a different meaning when used in that context.

4.10.1 Water-to-Soil Conversion for Organic Extraction Methods

Aqueous field blank results for organic extraction methods can generally be converted to solid units by comparing the ratio of the aqueous LOQs to the LOQs reported in the solid matrix method blanks.

> *Example*: A rinse blank has a detected result of 7.8 micrograms per liter (µg/L) for diethyl phthalate. The aqueous LOQ is 10 µg/L and the solid LOQ is 330 µg/kg. The diethyl phthalate result in the rinse blank is the equivalent of a result of 257.4 µg/kg (7.8 x 330/10). Because diethyl phthalate is a common laboratory contaminant, the artifact threshold is 2,574 µg/kg.

4.10.2 Water-to-Soil Conversion for VOCs

For VOCs, the formula for converting a water result to a soil result is not straightforward; the laboratory should be consulted before the convention used for organic extraction methods can be used to evaluate VOCs field blank results. In some cases, the raw data will show an "on-column" result reporting the concentration in the extract not converted to the final units used for the matrix of the samples. In these cases, the on-column results for field blanks can be multiplied by 5 (or 10) and compared directly to the on-column results reported for the associated field samples. It is more likely; however, that the laboratory software will show the raw data results already converted to the matrix units and this method of comparison will be usable only in a limited number of cases.

4.10.3 Water-to-Soil Conversion for Metals

For metals, the conversion equation is as follows:

$$C_S = (C_W \times V_F)/M_E$$

Where:

C_S = the calculated equivalent solid concentration (in milligrams per kilogram [mg/kg])
C_W = the reported aqueous concentration in µg/L
V_F = The final volume of soil digestate extracts in liters (L)
M_E = The nominal mass extracted for solid samples in grams (g) (use the mass of a solid method blank)

> *Example*: A rinse blank has a detected zinc concentration of 5.3 µg/L. The laboratory's preparation forms show that the final volume of soil extracts is 50 milliliters (= 0.05 L) and the soil method blank was extracted using 1.00 g. The rinse blank result is the equivalent of 0.265 µg/g = 0.265 mg/kg, which leads to an artifact threshold of 1.325 mg/kg. Note that the laboratory may report an actual mass for the method blank that is not a "round" number. If it can be determined that that the nominal method blank mass is a round number

like 1.00 g or 0.50 g, use that value even if an individual method blank may be slightly off (for example, 1.02 g instead of 1.00 g or 0.49 g instead of 0.5 g).

4.11 FIELD DUPLICATE PRECISION

The evaluation of field duplicate precision depends on the concentration of each target analyte detected in the duplicate pair relative to the LOQ. Concentrations can be considered "low-level" or "high-level." The QAPP will specify the criteria for making this determination, and this determination should be made for every detected analyte before any further duplicate evaluation. One of the most common criteria for determining if a pair of results is high-level is if both results are greater than 5 times the associated LOQ.

General rules for evaluating field duplicate results include the following elements in the sequential order they are presented:

1. Two nondetected results are considered to be in control.
2. Two results detected below the LOQ, or one result below the LOQ and one nondetected result, are considered to be in control.
3. Two low level results or one low level-result and one high-level result are considered to be in control if the absolute difference of the two results is less than the value of the LOQ.
4. Two high-level results are considered to be in control if the RPD of the two results meets the RPD acceptance criterion listed in the QAPP.

The evaluation criteria presented in this section are also applicable to laboratory duplicate analyses that are performed for metals and other inorganic methods.

4.12 SURROGATE RECOVERIES

As discussed in Section 3.2 of the SOP, the validator should verify that the surrogate control limits reported by the laboratory match those required in the project QAPP. Although some data validation conventions assign individual surrogate compounds to lists of target compounds, HGL discourages this practice and the preferred approach is to assume that all surrogate discrepancies are associated with all target analytes. An exception to this is the evaluation of SVOCs surrogate results. When evaluating surrogate recoveries for this method, the acid extractible fraction surrogates should be associated with the acid extractible fraction target compounds (phenols and benzoic acid), and the base/neutral extractible surrogates should be associated with the base/neutral extractible fraction target compounds (all other analytes).

Surrogate recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) the validator should consider whether an analytical system problem has occurred. If the cause for

abnormally high recoveries is not noted in the case narrative, the validator should contact the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking, and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

Dilution of samples can affect surrogate recovery performance. For methods that have surrogate compounds added to a sample before any dilution steps, surrogate discrepancies can occur that are not caused by matrix or analytical effects but rather are caused by dilution effects. The validator should examine surrogate discrepancies in diluted analyses. In most cases, surrogate discrepancies reported in samples diluted greater than 5 times should be considered to be a dilution effect and qualification should not be applied to the affected sample results. Some methods, such as VOCs, can have surrogates added after dilution; in this case, dilution effects will not occur and the surrogate recoveries can be evaluated regardless of the dilution level.

4.13 METHOD-SPECIFIC QC CHECKS

Method-specific QC elements include such checks as pH buffer checks, cyanide distillation standards, synthetic precipitation leaching procedure extraction blanks, and replicate precision for total organic carbon. If these checks are reported in a Stage 2A data package, the validator should review these items as appropriate to the assigned level of validation. If the review guidelines are not included in the QAPP, the validator should consult with the project chemist to develop a review and qualification approach.

4.14 ANALYTE QUANTITATION

The validator should discuss any dilutions performed. In some cases, multiple analyses will be performed on a sample because of a required dilution or to verify results affected by a QC discrepancy. Some laboratories will report the entire analytical dataset for all analyses performed on a sample, while others will report only the "best" result for each analyte. If the laboratory reported multiple results for an analyte or set of analytes in a sample, the validator should select the best result for each analyte in each sample and indicate which result was chosen and why in the validation narrative. All results not selected for use are excluded from the dataset, and this is indicated by applying a # qualifier to the laboratory applied qualifiers (see Section 3.5).

Samples that are nominally solid samples may have very high percent moisture content. This is especially true of sediment samples that are very "soupy." Calculation of concentration on a dry weight basis for solid samples composed of less than 50 percent solids is complicated by the added nonhomogeneity of the samples. The validator should evaluate results from solid samples with high liquid content and apply qualification in accordance with professional judgment if qualification protocols are not specified in the QAPP.

5.0 STAGE 2B QC ELEMENTS

The Stage 2A validation guidelines presented in Section 4.0 are applicable to QC elements that are common to many analytical methods. Stage 2B validation guidelines build on the Stage 2A requirements and address QC elements that are more specific to individual extraction and analytical principles.

5.1 GC/MS ORGANICS

Gas chromatography (GC)/mass spectrometer (MS) organics include analyses for VOCs and for SVOCs, most commonly by SW-846 methods 8260B or C and 8270C or D, respectively, and the associated selected ion monitoring (SIM) modifications to these methods. Air sample analyses performed by Method TO-15 and TO-15-SIM are also performed by GC/MS; however, in most cases, method-specific requirements that apply to TO-15 analysis will differ from the general GC/MS requirements discussed in this section.

5.1.1 Instrument Tuning

SW-846 GC/MS methods require that the MS be tuned at the beginning of each 12-hour analytical sequence. MS tuning is a critical QC component, and no analyses may proceed without an acceptable MS tuning. Each GC/MS method document prescribes the ions of interest and the required relative abundances. If MS tuning data show discrepancies and sample analyses proceeded without corrective action, the project chemist should be contacted immediately to resolve this issue.

In some cases, laboratories report tuning criteria for CLP analysis methods for SW-846 analyses. Although this approach is permissible, it is not in accordance with the QAPP. When the validator observes incorrect MS tuning criteria applied to tuning results, they should immediately contact the project chemist to determine if the affected results are usable and to initiate corrective action at the laboratory.

In some cases, analytical samples and the closing calibration verification standard (CCV) of an analytical batch will be analyzed outside the 12-hour window that begins with an instrument tune. The validator should examine the magnitude of the exceedance to determine if the discrepancy is nominal. For larger discrepancies, the closing CCV results and other information should be reviewed to determine if any additional qualification is required.

5.1.2 Instrument Initial Calibration

Most GC/MS analytes will be calibrated to a mean relative response factor (RRF), which quantitatively relates the concentration of each target analyte to the associated internal standard.

There should be at least 5 calibration points for an initial calibration to a mean RRF to be valid. If the calibration relationship for a compound is linear or quadratic, a minimum of 6 and 7 points, respectively, is required.

5.1.2.1 Instrument Performance Criteria

For an initial calibration to be valid for GC/MS methods 8260B and 8270C, system performance check compounds (SPCCs) and calibration check compounds (CCCs) are critical QC elements and must meet acceptance criteria, even if these method-specified compounds are not target analytes for the associated samples. One exception to this statement is if SVOCs analyses are only requested for base/neutral-extractable compounds or acid extractable compounds, only the SPCCs and CCCs associated with the requested fraction need be reported and evaluated. Each SPCC must meet minimum mean RRF requirements, even if an individual SPCC is calibrated to a linear or quadratic relationship. Each CCC must meet maximum percent relative standard deviation (%RSD) requirements, even if an individual SPCC is calibrated to a linear or quadratic relationship. Failure of these compounds to meet acceptance criteria can indicate instrumental problems such as dirty injector ports, carrier gas flow problems, or reactive sites on the chromatography column. Consequently, analyses performed in association with failed SPCCs and CCCs are potentially compromised by instrument performance. Methods 8260C and D and 8270D and E do not have requirements for SPCCs and CCCs; SPCC and CCC performance is also not evaluated for the SIM modifications to Method 8260B and 8270C (see Section 5.1.2.2).

If SPCC or CCC discrepancies are noted, this information must be referred to the HGL senior chemist and project manager for immediate follow-up with the laboratory. SPCC and CCC discrepancies are serious QC deficiencies and can potentially result in the rejection of all data produced in association with that initial calibration. The HGL senior chemist, the HGL project manager, and the laboratory project manager and QC manager will determine (1) if the associated results can be used, (2) the appropriate instrument maintenance and recalibration procedures, and (3) the notification measures to ensure that SPCC and CCC deficiencies are appropriately addressed at the laboratory as soon as they are noted by the analyst.

Note that an SPCC or a CCC that is also a target compound will be evaluated against both the SPCC or CCC acceptance criteria and against the target analyte criteria presented in Section 5.1.2.2 below. These two evaluations are independent of each other.

> *Example*: VOCs CCC vinyl chloride is reported calibrated to a mean RRF with %RSD of 17.5 percent. The requirement for VOCs CCCs is that each have a %RSD of no greater than 30 percent. Vinyl chloride shows acceptable performance as a CCC; however, the target analyte criterion is for %RSD to be no greater than 15 percent. Vinyl chloride does not meet the acceptance criterion for target analytes. The effects, if any, of this discrepancy would be considered to affect vinyl chloride alone and not to be indicative of an instrument performance issue.

Laboratory initial calibration summary form formats will vary. If SPCCs are reported as calibrated to a linear or quadratic relationship, some laboratories' summary reporting forms may present the m1 term associated with the curve instead of the mean RRF. Other laboratories' summary forms may present both. If the summary forms do not include the mean RRF for one or more SPCCs, the validator should examine the associated continuing calibration verification forms; on occasion, the initial calibration mean RRF is reported there in addition to the continuing calibration RRF. The mean RRF also may be discussed in the case narrative if HGL has requested the laboratory to do so. If the mean RRF is not available in other locations in the data package, the data validator should contact the laboratory project manager and have this information transmitted.

As with SPCCs, laboratory summary forms may not present the CCC %RSDs for those CCCs calibrated to linear or quadratic relationships. This information is generally not presented elsewhere in the data package unless HGL has arranged with the project laboratory to present this information in the case narrative. Otherwise, the data validator should contact the laboratory project manager and have this information transmitted.

5.1.2.2 Target Analyte Performance Criteria

The linearity criterion for GC/MS initial calibration is %RSD no greater than 15 percent. The correlation (r^2) of linear or quadratic relationships should be no less than 0.99.

Although many laboratories are still using Method 8260B for VOCs analysis, some projects require the use of Method 8260C. Most laboratories have discontinued the use of Method 8270C and have updated the SVOCs method to 8270D. Methods 8260C and 8270D have replaced the mean RRF requirements for SPCCs with analyte-specific minimum mean RRFs and have discontinued the use of CCCs. The analyte-specific mean RRF requirements also apply to the SIM modifications to these methods. The mean RRF only needs to be checked for target analytes. The laboratory's summary forms may not present this information for target analytes calibrated to linear or quadratic relationships. If so, the validator should review the continuing calibration forms and case narrative to determine if this information is available from other sources, as described in Section 5.1.2.1 above. While some laboratories now have DoD accreditation for methods 8260D or 8270E, these methods not currently widely used although they are expected to become more common in the future.

Methods 8260B and 8270C do not have a requirement for minimum mean RRF for target analytes; however, some historical project QAPPs may include a requirement for all target analytes to show a mean RRF of no less than 0.050. This requirement comes from the requirements of the CLP scope of work and associated data validation protocols.

5.1.3 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. SPCC and CCC performance evaluation or minimum mean RRF performance are not required for second source calibration verification standards.

5.1.4 Instrument Continuing Calibration

Continuing calibration standards must be analyzed immediately after an acceptable MS tuning has been performed. Continuing calibration standards are reviewed for SPCC, CCC, and target analyte performance in a manner similar to the evaluation performed for initial calibrations. SPCCs must meet method-specified continuing calibration RRF criteria and CCCs must meet method-specified percent difference (%D) criteria for methods 8260B and 8270C. Target analyte RRFs must meet criteria for methods 8260C and 8270D and for the SIM modifications to this method. Target analytes are evaluated against the target analyte criterion of no greater than 20 percent, and some QAPPs may also require that target compounds also meet minimum continuing calibration RRF criteria.

Some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias.

QSM version 5.0 introduced the requirement that GC/MS analyses to be bracketed by an end-of-sequence CCV, also known as a closing CCV. The first CCV standard analyzed after project sample analyses in a sequence is considered the ending CCV associated with those samples, even if there are additional CCVs analyzed later in the sequence. If samples are analyzed in a continuous sequence extending over more than 12 hours and involving multiple tunes and opening CCV standards, it is acceptable to consider each opening CCV to be the closing CCV for the preceding samples. Closing CCVs are required to have a %D requirement less than 50% for each target analyte. SPCC, CCC, and minimum target analyte RRFs do not need to be reviewed for closing CCVs.

5.1.5 Internal Standards

Internal standard compounds must be spiked into every sample, standard, and blank analyzed by GC/MS methods. Internal standards must meet the method area and retention time criteria for peak area and retention time. Older versions of the DoD QSM required that the peak area for each internal standard compound must be no less than 50 percent and no greater than 200 percent of the peak area for that internal standard compound in the midpoint standard in the associated initial calibration sequence. The retention time for each internal standard must be within 10 seconds of

the retention time of the midpoint standard in the associated initial calibration sequence. While this requirement was retained in DoD QSM version 5.1, this version of the QSM (and subsequent versions) expanded the internal standard acceptance criteria to allow for the daily initial CCV to be used for peak area and retention time comparison on days when initial calibration is not performed.

Discrepancies in internal standard performance are generally associated with the matrix characteristics of individual samples. Although internal standard discrepancies are not usually indicative of an instrument issue, the QSM presents a requirement for the laboratory to include an evaluation of the analytical system when assessing the potential causes and corrective action for internal standard discrepancies, as there are potential systematic issues that can also lead to poor internal standard performance. Internal standard discrepancies should always be associated with a corrective action by the laboratory, which will usually consist of re-extraction and reanalysis of the affected samples or perform instrument maintenance and recalibration if the internal standard discrepancies are attributable to an issue with the analytical system and not sample specific. The only exception is if the internal standards that exhibit discrepancies are not associated with any target analytes.

Each internal standard is associated with a specific set of analytes. When internal standards are out of control, only the associated target analytes are qualified in the affected sample. Many formats of initial calibration summary forms are organized to show the internal standard associations. If the internal standard associations are not shown on the initial calibration summary or other form, the validator should contact the laboratory to have the required information transmitted.

5.2 GC AND HPLC ORGANICS

GC and high-performance liquid chromatography (HPLC) organics include analyses for pesticides (organochlorine and organophosphorus), PCBs, explosives, herbicides, and petroleum products. GC and HPLC analyses use dual columns or dual detectors to identify target analytes. Some laboratories assign the same quantitative significance to both columns/detectors, while others specify a dedicated primary and secondary column/detector. If presented, the QC data for both the primary and secondary column/detector should be evaluated. In cases where instrument QC discrepancies affect one column/detector and not the other, some degree of interpretation by the validator is required to determine the effect on the associated samples. If the detector or column used to report the result for each analyte in a sample can be determined, discrepancies reported from other columns or detectors that were not used to report the results should not be used to qualify results.

5.2.1 Instrument Initial Calibration

As with GC/MS methods, initial calibrations must include at least five calibration points for calibration to response factor. Six calibration points are required for linear calibration and seven

calibration data points are required for quadratic calibration. Initial calibration to response factor is required to meet the method-specific requirement, which is usually a %RSD no greater than 15 percent or 20 percent.

The analysis of PCBs only requires multipoint calibration for PCB-1016 and PCB-1260, with single point calibration for all other reported PCB congeners. PCBs are quantified using five characteristic peaks. The *mean* %RSD of the PCB-1016 peaks and the mean %RSD of the PCB-1260 peaks are compared to the acceptance criteria. Individual characteristic peaks may exceed the %RSD criterion so long as the mean %RSD for each congener is acceptable. Discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260. If PCBs other than 1016 or 1260 are identified in any associated sample, the laboratory should perform a multipoint calibration for all identified congeners and reanalyze the samples to quantify the detected congeners. These reanalyses should be accompanied by all other QC elements spiked with the specific detected PCBs and not with the representative PCB-1016/1260 mixture.

5.2.2 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP.

Because of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs second source calibration verifications are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

5.2.3 Instrument Continuing Calibration

GC and HPLC methods require a continuing calibration standard to be analyzed at the beginning of each analytical sequence, at regular intervals after a specified number of sample analyses (generally 10), and at the end of the end of the analytical sequence. Each continuing calibration standard is associated with all samples analyzed after the previous continuing calibration standard analysis and before the following continuing calibration standard analysis. Discrepancies in continuing calibration standard analyses will require evaluation of the affected analytes in the associated samples.

As a result of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs continuing calibration verification standards are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs

1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias.

5.2.4 Degradation Summary

Analysis for organochlorine pesticides requires that a 4,4′-dichlorodiphenyltrichloroethane (DDT) and endrin degradation standard be measured before samples are analyzed and at the beginning of each 12-hour shift. These compounds are easily degraded at the injection port. Generally, the acceptance criterion is that neither DDT nor endrin should have a breakdown of greater than 15 percent. Unacceptable DDT breakdown will cause the qualification of all associated DDT, 4,4′-dichlorodiphenyldichloroethene, and 4,4′-dichlorodiphenyldichloroethane results. Unacceptable endrin breakdown will cause the qualification of all associated endrin, endrin aldehyde, and endrin ketone results. However, this test should be performed as a test of the inertness of the analytical system even when DDT and endrin are not target analytes for a given project, unless otherwise specified in the QAPP.

5.2.5 Retention Times

There are no standardized summary forms for reporting chromatographic retention times, and each laboratory's forms will vary greatly in both format and content. In general, the validator should review all available retention time data. Retention time shifts, either in calibration standards or in sample results, must be accompanied by analyst documentation for the associated results to be accepted.

5.2.6 Confirmation

GC and HPLC methods require confirmation (except for petroleum hydrocarbon analysis) to differentiate target analytes from matrix interferences. Detected results are confirmed either by a second detector or by retention time on a second column that has different chemical properties than the primary column. Target analytes detected on one column/detector that are not confirmed are potentially interferences rather than a true detection. Such results should not be reported as detections by the laboratory unless the analyst and section leader provide documentation as to why the analytes should be considered detected in the absence of confirmation. Results that are detected and confirmed should have approximately the same quantitation on both columns/detectors; results that do not meet RPD criteria should be qualified as estimated.

5.3 METALS

Metals analyses are performed using SW-846 methods 6010C or D (inductively coupled plasma-atomic emission spectroscopy [ICP-AES]) and 6020A or B (inductively coupled plasma-mass spectrometry [ICP-MS]) for "full list" metals; cold vapor atomic absorption (CVAA) methods 7470A and 7471B for mercury in water and soil, respectively. Graphite furnace atomic absorption (GFAA) method 7010 can be used for select metals that can be affected by spectral interferences that prevent definitive analysis by ICP-AES; however, with improvements to ICP-AES and the emergence of ICP-MS as the metals method of choice, GFAA analysis is now rarely used.

5.3.1 Instrument Tuning

Methods 6020A and B use a mass spectrometer to identify target elements; the mass spectrometer must be tuned prior to use. Instrument tuning data is not always available on summary forms. If the required data is not available for review on summary forms, the data validator should contact the laboratory to request the required information. If the information is not available on summary forms, the raw data must be examined.

The QSM requires that tuning peaks show a resolution of no greater than 0.9 atomic mass units (amu) at 10 percent peak height. Some instrumental systems report the peak resolution at 5 percent of total peak height; this is more stringent than the QSM requirement and should not be considered a discrepancy provided that the resolution criterion of ≤0.9 amu is met.

5.3.2 Internal Standards

Methods 6020A and B use internal standards in the quantification of target elements. If an internal standard does not meet acceptance criteria and corrective action was not performed or was not successful, the target analytes associated with that internal standard should be qualified in the affected sample.

In some cases (especially with short analyte lists), there may be internal standards that do not meet acceptance limits but are not associated with target metals. Some laboratories also will choose a secondary internal standard to quantify a metal if the primary internal standard does not meet acceptance criteria.

5.3.3 Initial Multipoint Calibration

Initial multipoint calibration is required for CVAA and GFAA methods. It is not required for ICP-AES or ICP-MS analyses and there are QC elements described below that are intended to be performed instead of initial multipoint calibration; however, if a multipoint initial calibration is performed, it must meet the acceptance criteria in the QAPP. If the alternative QC checks are acceptable but the multipoint initial calibration was out of control, the associated results must be considered for qualification. The laboratory should not present such a situation as being in control.

5.3.4 Low-Level Calibration Verification

Low-level calibration verification standards at or below each target compound LOQ are required under projects with QC requirements from the QSM. This QC check should be performed for ICP-AES and ICP-MS methods regardless of whether an initial multipoint calibration is performed. Note that the DoD QSM requires that this check meet control limits of 80 to 120 percent even though the methods allow a window of 70 to 130 percent.

Some laboratories also perform what is called a CRDL check standard. This CRDL check standard is generally spiked at 2 times the LOQ. If the low-level calibration verification standard does not meet acceptance criteria, the usual response is to qualify detections with concentrations up to 10 times the LOQ and nondetections. However, if a low-level calibration verification does not meet acceptance criteria and an associated CRDL check standard is performed and is in control, stability at 2 times the LOQ has been demonstrated and only detected results up to 2 times the LOQ and nondetections require qualification.

5.3.5 High-Level Calibration Verification

High-level calibration verification standards are used to determine the upper end of the working range of the instrument. If the high-level calibration verification standard does not meet acceptance criteria, the validator should determine if a multipoint initial calibration has been performed. If so, and the high point on the calibrated curve has a concentration below that of the high-level calibration verification standard, only results above the high point on the curve (adjusted for matrix as necessary) require qualification.

Detected results above the high-level calibration verification should be qualified unless the laboratory performed appropriate dilutions so that the effective concentration measured by the instrument is less than the high-level calibration verification standard concentration.

5.3.6 Initial and Continuing Calibration Verification

Most laboratories use initial calibration verification (ICV) standard analyses as a second source verification check. HGL's preferred convention is to associate ICV results with all sample results in an analytical sequence and to the associated continuing CCV results only with sample results "bracketed" by a given CCV. A result is considered bracketed by a CCV if that CCV is the last CCV analyzed before that result was generated or is the first CCV analyzed after that result is generated.

More recent versions of Methods 6010 and 6020 include the analysis of low-level ICVs and CCVs. The QSM does not provide control limits for these low-level standards and HGL uses general acceptance criteria of 70-130 percent. If the project laboratory uses the low-level ICV as the DoD-

required low-level calibration verification standard (see Section 5.3.5), then the low-level ICV is required to meet the DoD acceptance criteria of 80-120 percent.

It is allowable to evaluate ICV/CCV results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable if the ICV or CCVs are from the same source as the initial calibration; however, if the ICV and/or CCVs are from a second source, the associated results should be considered for qualification.

5.3.7 Continuing Calibration Blanks

Continuing calibration blanks (CCBs), including initial calibration blanks (ICBs), are performed for inorganic methods. CCBs are evaluated like method blanks (Section 4.9). HGL's preferred convention is to associate ICB results with all sample results in an analytical sequence and to associated CCB results only with sample results bracketed by a given CCB. A result is considered bracketed by a CCB if that CCB is the last CCB analyzed before that result was generated or is the first CCB analyzed after that result is generated.

CCBs are aqueous but can be associated with both aqueous and solid matrix analyses. When determining the potential effect of CCB contamination on the associated solid matrix sample results, convert the CCB result to an equivalent soil concentration using the procedure presented for field blanks (Section 4.10.3).

The artifact threshold associated with field blank contamination is 5 times the concentration detected in the blank (10 times the concentration in the case of common laboratory contaminants). As with action levels associated with method blank contamination, both aqueous and solid-equivalent artifact levels should be adjusted on a sample-specific basis to account for sample-specific variables. In most cases, it will be clear that a result is above or below an action level and in practice this sample-specific adjustment is necessary for a minority of comparisons.

5.3.8 Interference Check Sample Results

Interference check samples (ICSs) are analyzed in pairs. ICS A (ICSA) is a blank spiked with high concentrations of aluminum, calcium, iron, and magnesium; in some cases, ICSAs will also be spiked with lower concentrations of other elements that are also potentially interfering. ICS AB (ICSAB) is spiked with the same levels of aluminum, calcium, iron, and magnesium as is the ICSA and contains lower spiked levels of the elements of concern. The purpose of analyzing ICSAs is to determine if interelement correction factors from naturally occurring elements that are often present at high concentrations cause false positive or false negative results due to over- or under-correction. The purpose of analyzing ICSABs is to determine if interelement correction factors for all elements, including those that occur at high concentrations naturally, are being applied correctly and provide correct quantitation. Generally, QAPPs will require a single ICSA and ICSAB be analyzed before sample analyses as a minimum requirement; however, if the laboratory reports

multiple ICSA/ICSAB results in an analytical sequence, the reviewer should evaluate the bracketing ICSA/ICSAB results both before and after the sample analyses and assign both sets equal significance.

According to QSM version 5.1, the ICSA acceptance criteria are a concentration with absolute value less than one-half the LOQ; however, note that QAPPs written in accordance with earlier versions of the QSM (through version 5.0) will present acceptance criteria of less than the LOD for target metals instead. ICSA discrepancies can be an indicator of problems with interelement correction. HGL has had experiences with false positive results ultimately traced to failure of the analytical system to take advantage of all mathematical tools available to correct for interferences. In cases where ICSA discrepancies are attributable to known contamination in the stock solution, this situation should be noted by the laboratory in the case narrative. In other cases, ICSA discrepancies can be attributed to instrument drift or system contamination. Indicators of this kind of issue will include positive or negative results in associated CCBs or method blanks. If ICSA discrepancies are potentially attributable to sources other than interelement interference, the reviewer should consider not qualifying the associated results or reducing the severity of qualification.

Most data validation conventions consider ICSA results with absolute value greater than the LOQ to constitute a severe discrepancy. If severe ICSA discrepancies are noted, the data reviewer should contact the HGL senior chemist before rejecting the associated results. ICSAs often contain higher levels of interfering element concentrations than are present in environmental samples, and alternatives to rejection may be available.

It is rare for ICSAB results to fail to meet control criteria, and often this is an indication of a spiking error rather than a problem with the analytical sequence.

5.3.9 Recovery Test Results

GFAA methods use recovery tests to determine if the sample matrix has affected reported results. The method requires a recovery test to be performed on a representative sample in each preparation batch, but in practice, laboratories perform recovery tests on a sample-specific basis.

5.3.10 Method of Standard Addition Results

The method of standard additions (MSA) is associated with GFAA analyses; this procedure is rarely performed as virtually all laboratories perform sample-specific recovery tests rather than batch-specific recovery tests. If MSA results are reported in a data package, the data validator should consult with the HGL senior chemist.

5.4 GENERAL CHEMISTRY

General chemistry parameters include a variety of analytical parameters and methodologies, including colorimetry, ion chromatography, GC, and infrared spectrometry. Usually, these parameters are secondary data that are used to determine the potential for a site to undergo monitored natural attenuation or the progress of monitored natural attenuation. Often, these tests will only require a Stage 2A data review; however, some parameters, such as cyanide, perchlorate, anions, or total organic carbon will, on occasion, require Stage 2B validation.

In many cases, the review of general chemistry QC parameters is similar to the review of the corresponding parameters for metals. Method-specific QC parameters should be discussed in the QAPP along with the acceptance criteria and qualification requirements. Some laboratories do not have summary forms for Stage 2B QC elements and the raw data will need to be examined by the validator to evaluate performance.

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ATTACHMENT D
Automated Data Review

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ATTACHMENT D
Automated Data Review

1.0 INTRODUCTION

The most common programs used to perform automated data review (ADR) are the web-based data validation functions provided by Environmental Synectics, Inc. (Synectics) of Sacramento, California, and the FUDSChem data validation and evaluation program developed by U.S. Department of Defense with Synectics. ADR programs identify quality control (QC) issues by comparing QC results in the laboratory-generated electronic data deliverable (EDD) against a data library generated in accordance with the requirements of the project Quality Assurance Project Plan (QAPP). This data library is often referred to as an electronic QAPP (eQAPP). ADR programs can streamline the data validation process by identifying QC issues and providing a listing of preliminary data qualification to be applied to the associated results; the extent of chemist review post-ADR will depend on project-specific requirements and objectives and on the EDD-generating capabilities of the laboratory.

2.0 ADR USES AND LIMITATIONS

ADR can reduce the amount of time spent reviewing laboratory data reports by generating a comprehensive list of QC discrepancies in a data package and identifying the associated affected results. ADR can be the primary data validation tool used for a project, integrated with only minimal "sanity check" review by a staff chemist, or it can be used as a tool to support manual data validation, relieving the validator from the task of reviewing each page of the laboratory data report and documenting all observed QC discrepancies.

ADR can support Stage 2A validation (as defined in Attachment A).

2.1 STAGE 2A REVIEW LIMITATIONS

ADR is not capable of evaluating the information in several critical areas of Stage 2A data review. In some cases, the QC element is not included in ADR. In other cases, ADR can perform an initial check of a QC element against the performance criteria but is not capable of incorporating additional sample- or method-specific information that is used to modify the initial evaluation. Following ADR, the ADR result should be reviewed by a staff chemist to ensure that all qualification applied by ADR is appropriate based on additional information not able to be evaluated by ADR.

2.1.1 Case Narrative

ADR cannot review any issues identified in the case narrative that may not be reflected in the associated QC data results. The case narrative should be examined by a chemist to ensure that

there are no additional issues that require corrective action, resolution, or qualification of the associated data.

2.1.2 Sample Delivery and Condition

ADR is capable of qualification based on sample temperature at receipt; however, it cannot evaluate other issues associated with sample delivery and condition, including broken bottles, misidentified samples, improper preservation, and bubbles greater than 6 millimeters noted in volatile organic compound sample vials. The staff chemist should review the chain of custody, the laboratory sample chronicle, and sample receipt documentation to verify that the samples were delivered to the laboratory in good condition, and properly identified.

2.1.3 Holding Times

Holding time can be evaluated by ADR. However, the holding time calculated from the time of collection on the chain of custody to the time of preparation or analysis at the laboratory can differ from the true holding time. This can be due to time zone differences between the sample location and the laboratory or a switch to or from daylight savings time occurring between the time of sampling and the time of preparation or analysis. The staff chemist should review the holding time calculations and ensure that these differences are accounted for.

Additionally, some projects require that the field teams assign "dummy" sample times to field duplicate samples to obscure the parent sample identity. The staff chemist should ensure that holding times for field duplicate samples have been calculated using the actual collection time and not an arbitrary collection time entered by the field sampling team.

In general, holding times longer than 72 hours are expressed in "days" and are evaluated to the nearest calendar day. The staff chemist should review any holding time discrepancies identified by ADR to determine if the affected analyses meet the holding time when evaluated against calendar days instead of the number of elapsed 24-hour periods. The Synectics ADR program is known to qualify samples based on 24-hour periods. This qualification may need to be corrected manually for those analyses with holding times expressed in days.

2.1.4 Surrogate Recoveries

Sample dilution can cause surrogate recovery discrepancies that are not associated with matrix interferences or analytical problems. When ADR identifies surrogate discrepancies in diluted samples, the staff chemist should review the affected data. Generally, data from sample analyses performed at dilution greater than fivefold should not be qualified for surrogate discrepancies unless a matrix effect is noted to have affected the sample even when analyzed under dilution. Most ADR programs can incorporate a dilution factor above which results will not be qualified for

surrogate discrepancies, and this maximum dilution factor should be identified on a method-specific basis in the eQAPP.

2.1.5 Matrix Spike/Matrix Spike Duplicate Recoveries

Matrix spike (MS)/matrix spike duplicate (MSD) recovery discrepancies are not considered to have significance if the native concentration of the affected analyte in the parent sample is more than four times the concentration resulting from the spike (see Section 4.7 of Attachment C). In some cases, the native concentration of one or more target analytes is so high that the MS/MSD will be analyzed under dilution. Discrepancies in diluted MS/MSDs are likely to be a result of dilution effects rather than matrix effects, as the majority of material in a diluted sample will consist of material not representative of the site (that is, it will be analyte-free laboratory water or solvent) and unlikely to contain interferences. In some cases, MS/MSDs are analyzed without dilution but with one or more spiked compounds quantitated above the calibrated range. Quantification of results above the calibrated range is inherently less reliable, and MS/MSD discrepancies can be caused by quantification errors.

Some ADR programs cannot take into account the "four times" rule, the effects of dilution, or the effects of results quantitated above the calibrated range when assigning qualifiers for MS/MSD discrepancies. The staff chemist should evaluate the MS/MSD percent recovery discrepancies identified by ADR and determine if these results are truly indicative of a matrix effect or are caused by other factors that eliminate the need for qualification of the associated results.

In some cases, the laboratory will report MS/MSD results from a different sample delivery group (SDG) as batch control; such batch control MS/MSDs are often presented without the client sample identification (ID). When a batch control MS/MSD is reported, the staff chemist should use the laboratory sample ID to confirm whether the MS/MSD is actually from a site sample reported in a different SDG or from a nonsite sample. If the MS/MSD is from a site sample, it will be considered applicable to associated results and any data qualification selected by ADR will be considered applicable. If the MS/MSD cannot be associated with a site sample, the results should be noted but no qualification should be applied unless the underlying cause of the discrepancy is suspected to be a problem with the analytical system.

Serial dilution and post-digestion spike (PDS) results are considered part of Stage 2A evaluation. These QC checks can be used to modify the qualifiers applied due to MS/MSD percent recovery (%R) discrepancies; however, these elements are not usually provided in laboratory EDDs. Where ADR applies qualifiers to metals results based on MS/MSD %R discrepancies, the validator should examine the serial dilution or PDS results in accordance with the QAPP validation guidelines to determine if those qualifiers should be eliminated or reduced in severity.

2.1.6 Matrix Spike/Matrix Spike Duplicate Precision

As described in Section 4.7 of Attachment C, some laboratories compare the concentrations detected in the MS and the MSD to calculate precision rather than comparing the percent recoveries. This convention can lead to the resulting relative percent differences (RPD) being an incorrect representation of the analyte-specific precision. If the expected concentration in the MS is different than the expected concentration in the MSD, calculation of the RPD using a direct comparison of the detected concentrations is not relevant. The staff chemist should verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is the same or reasonably similar to the expected concentration in the MSD. If the RPDs are calculated using noncomparable results, the validator should contact the laboratory and request that the calculations be performed using percent recoveries. If this information cannot be produced by the laboratory, the validator will have to perform these calculations.

2.1.7 Field and Laboratory Duplicate Precision

ADR evaluates the performance of field and laboratory duplicates based on the calculation of the RPD of the results for the parent sample and duplicate. However, some ADR programs will not evaluate duplicate performance considering the commonly used convention for "low-level" results, usually defined as results that are less than 5 times the quantitation limit. Under most data validation protocols, low-level results are evaluated by comparing the absolute difference between the parent and duplicate result to the associated quantitation limits (see Section 4.11 of Attachment C). If ADR is used without supplemental manual review, there is a potential for data to be over-qualified for field or laboratory duplicate discrepancies.

2.1.8 PCB Discrepancy Associations

As described in Sections 4.6 and 4.7 of Attachment C, laboratory control samples (LCS) and MS/MSDs for polychlorinated biphenyls (PCBs) analysis are spiked with only two representative PCB congeners. Discrepancies affecting PCB-1016 are also considered to affect results for PCBs 1221 and 1232, and discrepancies affecting PCB-1260 are also considered to affect results for PCBs 1242, 1248, and 1254. If the ADR program is not able to extend the association of a QC issue reported for one compound to other compounds in accordance with the QAPP, this situation will have to be addressed by the staff chemist.

2.1.9 Selection of Final Result

In cases where multiple analysis results are reported for a sample because of dilution or reanalysis, all analyses are reviewed by ADR. Based on the body of QC data, the staff chemist should select one definitive result for each analyte in each sample in accordance with Section 3.5 of Attachment C. All other results for that analyte in that sample should be denoted as superseded by applying an # qualifier to the qualifiers applied by ADR.

2.2 STAGE 2B REVIEW LIMITATIONS

The QC elements included in a Stage 2B data validation are limited by the specific capabilities of the selected ADR program and the laboratory's ability to supply an EDD that addresses these QC elements. When an ADR program is used to perform Stage 2B validation, the data validator must be aware of the limitations of the laboratory EDD and the ability of ADR to address situations where the data is not reported in the standard format (e.g., the evaluation of system performance check compounds that have been calibrated to a curve and do not have the associated mean relative response factor reported.

3.0 ELECTRONIC QAPP AND DATA LIBRARY

All ADR functions require reference to the project-specific data library that is assembled into an eQAPP. It is critical that the eQAPP be prepared and the associated data library transmitted to the laboratory before project sampling activities. If the data library has not been constructed at the time of sample analysis, the required information may not be captured in the laboratory EDD, resulting in the need to regenerate EDDs that conform to the data library requirements or late EDD delivery, causing delays and potentially increased laboratory costs.

The eQAPP should encompass the sensitivity limits, control limits, validation protocols, qualification conventions, and qualifier priorities that have been established in the project QAPP. The data library requires the input from a HydroGeoLogic, Inc. (HGL) project chemist and the laboratory database manager at a minimum. After the draft eQAPP has been prepared, all information contained in it must undergo a QC review against the requirements of the QAPP by an HGL chemist. Any discrepancies between the eQAPP and the QAPP must be resolved before the eQAPP can be used to support ADR.

3.1 SENSITIVITY LIMITS

There are two principal conventions for establishing sensitivity limits. Both are in common use and are described in Attachment C, Table C.1. ADR file formats can support either sensitivity limit convention, as specified in the project QAPP.

3.2 CONTROL LIMITS

The method- and matrix-specific control limits listed in the QAPP should be incorporated into the eQAPP. Control limits can be differentiated by QC element (such as LCS/LCS duplicates and MS/MSDs).

3.3 VALIDATION PROTOCOLS

The project-specific validation protocols are entered into the eQAPP using the Qualification Scheme application of the ADR program. The Qualification Scheme for a project must match the

procedures presented in the project QAPP. The Qualification Scheme allows for qualifiers to be assigned based on whether each affected result is a detection or a nondetection. The Qualification Scheme also allows for discriminating between minor discrepancies and major discrepancies that require results to be rejected, i.e., several QC elements allow the entry of both an estimation limit and a rejection limit for that element.

3.4 QUALIFICATION CONVENTIONS

The Qualification Scheme includes the project-specific qualifiers that will be applied to analytical results either as a result of quantification (for example, results below the quantitation limit) or as a result of a QC discrepancy. The eQAPP can specify on a method-specific basis whether some QC elements, such as MS/MSD results, affect the parent sample only or all samples in the associated preparation batch.

3.5 QUALIFIER PRIORITY

ADR includes a Qualifier Hierarchy matrix that allows for the determination of the final qualifier applied to each data point. The Qualifier Hierarchy matrix for some ADR programs only allows for the simultaneous evaluation of two qualifiers; if more than two qualifiers are potentially applicable to a sample result, ADR will evaluate only the two highest priority qualifiers as defined in the QAPP.

4.0 ADR LABORATORY DELIVERABLES

The primary ADR programs can process a staged EDD-formatted EDD. The specifications for providing data for FUDSChem are provided on the FUDSChem website: http://fudschem.com/public/framework/bannerhtml.aspx?dsn=systm&idhtml=10642&themesuffix=default&banner=banner_fudschem.jpg&idMenu=78296&ddlDSN=SYSTM&Title=HOME.

5.0 ADR PROCEDURES

At a minimum, each ADR EDD delivered by the laboratory will undergo a QC review upon receipt and QC sample associations will be added to the file. If additional manual review is required after the QC and association step, the procedures described in Sections 5.1 and 5.2 must be followed.

5.1 ADR FILE QC

On receipt from the laboratory, each set of EDD files should be reviewed to ensure that all required fields have been populated correctly and that all information is complete and correct. Following this QC check, the field QC sample results in the laboratory data package must be associated with the field sample results. This step includes associating trip blanks and equipment blanks with the

corresponding field samples and associating designated field duplicate samples and MS/MSDs with the corresponding parent samples.

5.2 SUPPLEMENTAL MANUAL REVIEW – STAGE 2A

Manual chemist review of Stage 2A QC elements should include the following elements, in accordance with the referenced guidance presented in Section 2.1 of Attachment D and the referenced sections of Attachment C:

- Case narrative (Section 4.1), including any associated sample discrepancy reports;
- Chain of custody (Section 4.2);
- Sample receipt and log-in forms (Section 4.3);
- Sample ID cross reference (Section 4.4);
- Association of Aroclors 1016 and 1260 QC discrepancies with additional Aroclors (Sections 4.6 and 4.7);
- Evaluation of any MS/MSD results potentially not relevant to sample results (Section 4.7); and
- Evaluation of any low-level field duplicate and laboratory duplicate comparisons (Section 4.11).

Any changes made to the ADR results based on manual review must be documented and undergo a peer review.

5.3 SUPPLEMENTAL MANUAL REVIEW – STAGE 2B

A manual chemist review of Stage 2B QC elements should verify that all required QC elements were validated by the ADR program with manual review and validation to address any identified gaps or special circumstances outside the capabilities of the ADR program.

Any changes made to the ADR results based on manual review must be documented and undergo a peer review.

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ATTACHMENT E
Data Qualification Reason Codes

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ATTACHMENT E
Data Qualification Reason Codes

QC Element	Reason Code	Definition
Ambient Blank	ABH	Ambient blank result ≥ limit of quantitation (LOQ)
Ambient Blank	ABHB	Result is judged to be biased high based on associated ambient blank result
Ambient Blank	ABL	Ambient blank result <LOQ
Analyte Quantitation	ACR	Result above the upper end of the calibrated range
Analyte Quantitation	EXC	Result excluded; another data point for this analyte was selected for use (use with X-qualified results)
Analyte Quantitation	RTW	Target analyte outside retention time window
Analyte Quantitation	PSL	Solid matrix sample with percent solids less than 50%
Analyte Quantitation	PSLX	Solid matrix sample with percent solids less than 10%
Analyte Quantitation	TR	Result between the detection limit and LOQ
Calibration Blank	CBH	Initial or continuing calibration blank result ≥LOQ
Calibration Blank	CBHB	Result is judged to be biased high based on associated continuing calibration blank result
Calibration Blank	CBL	Initial or continuing calibration blank result <LOQ
Calibration Blank	CBN	Negative initial or continuing calibration blank result with absolute value <LOQ
Calibration Blank	CBNH	Negative initial or continuing calibration blank result with absolute value ≥LOQ
Continuing Calibration	CCCC	Calibration check compound did not meet percent difference (%D) criterion in continuing calibration standard
Continuing Calibration	CCVD	Continuing calibration standard did not meet %D criterion
Continuing Calibration	CRFL	Continuing calibration RRF below acceptance criterion
Continuing Calibration	CSPC	System performance check compound did not meet minimum RRF criterion in continuing calibration
Continuing Calibration	CVDX	Continuing calibration standard did not meet %D criterion, extreme discrepancy
Confirmation	CF	Confirmation precision exceeded acceptance criterion
Cyanide Method	DSH	High-level distillation standard did not meet %D criterion
Cyanide Method	DSL	Low-level distillation standard did not meet %D criterion
Equipment Blank	EBH	Equipment blank result ≥LOQ
Equipment Blank	EBHB	Result is judged to be biased high based on associated equipment blank result
Equipment Blank	EBL	Equipment blank result <LOQ
Field Duplicate	FDPA	Field duplicate results did not meet absolute difference criterion
Field Duplicate	FDPR	Field duplicate results did not meet RPD criterion
Holding Time	HTA	Analytical holding time exceeded
Holding Time	HTAX	Analytical holding time exceeded, extreme discrepancy
Holding Time	HTP	Preparation holding time exceeded
Holding Time	HTPX	Preparation holding time exceeded, extreme discrepancy
Initial Calibration	ICCC	Calibration check compound did not meet percent relative standard deviation (%RSD) criterion in initial calibration

ATTACHMENT E (continued)
Data Qualification Reason Codes

QC Element	Reason Code	Definition
Initial Calibration	ICLS	Initial calibration low-level standard >LOQ
Initial Calibration	ICR2	Initial calibration r^2 below acceptance criterion
Initial Calibration	ICRD	Initial calibration %RSD above acceptance criterion
Initial Calibration	ICRX	Initial calibration %RSD above acceptance criterion, extreme discrepancy
Initial Calibration	IRFL	Initial calibration RRF below acceptance criterion
Initial Calibration	ISPC	System performance check compound did not meet minimum mean RRF criterion in initial calibration
Initial Calibration	LQSH	LOQ check standard above acceptance criteria
Initial Calibration	LQSL	LOQ check standard below acceptance criteria
Initial Calibration	SSVD	Second-source standard did not meet %D criterion
Initial Calibration Verification	ICVD	Continuing calibration standard did not meet %D criterion
Initial Calibration Verification	ICVX	Continuing calibration standard did not meet %D criterion, extreme discrepancy
Interference Check Standard	ICAH	Non-spiked concentration above acceptance criterion in ICSA
Interference Check Standard	ICAN	Negative concentration with absolute value above acceptance criterion in ICSA
Interference Check Standard	ICHX	Non-spiked concentration above acceptance criterion in ICSA, extreme discrepancy
Interference Check Standard	ICNX	Negative concentration with absolute value above acceptance criterion in ICSA, extreme discrepancy
Interference Check Standard	ICSH	ICSA or ICSAB spiked analyte with high percent recovery (%R)
Interference Check Standard	ICSL	ICSA or ICSAB spiked analyte with low %R
Internal Standards	IRH	Internal standard peak area above upper limit
Internal Standards	IRL	Internal standard peak area below lower limit
Internal Standards	IRLX	Internal standard peak area below lower limit, extreme discrepancy
Internal Standards	ISRT	Internal standard retention time outside window
Labeled Standards	LSH	Labeled standard %R above acceptance criterion
Labeled Standards	LSL	Labeled standard %R below acceptance criterion
Labeled Standards	LSLX	Labeled standard %R below acceptance criterion, extreme discrepancy
Laboratory Control Sample	LCLX	LCS and/or LCSD %R below acceptance criterion, extreme discrepancy
Laboratory Control Sample	LCSH	LCS and/or LCSD %R above acceptance criterion
Laboratory Control Sample	LCSL	LCS and/or LCSD %R below acceptance criterion
Laboratory Control Sample	LCSP	LCS/LCSD RPD above acceptance criterion
Laboratory Duplicate	LDPA	Laboratory duplicate results did not meet absolute difference criterion
Laboratory Duplicate	LDPR	Laboratory duplicate results did not meet RPD criterion

QC Element	Reason Code	Definition
Low-Level Calibration Check	LLCH	Low-level calibration check above the upper limit
Low-Level Calibration Check	LLCL	Low-level calibration check below the lower limit
Low-Level Calibration Check	LLXL	Low-level calibration check below the lower limit, extreme discrepancy
Method Blank	MBH	Method blank result ≥LOQ
Method Blank	MBHB	Result is judged to be biased high based on associated method blank result
Method Blank	MBL	Method blank result <LOQ
Matrix Spike	MSH	MS and/or MSD %R above acceptance criterion
Matrix Spike	MSL	MS and/or MSD %R below acceptance criterion
Matrix Spike	MSLX	MS and/or MSD %R below acceptance criterion, extreme discrepancy
Matrix Spike	MSP	MS/MSD RPD above acceptance criterion
Post-Digestion Spike	PDH	Post-digestion spike recovery high
Post-Digestion Spike	PDL	Post-digestion spike recovery low
Post-Digestion Spike	PDLX	Post-digestion spike recovery low, extreme discrepancy
Post-Digestion Spike	PDN	Post-digestion spike not performed or not applicable and serial dilution result not performed or not applicable
Sample Delivery and Condition	BUB	Bubbles >5 millimeters in volatile organic compounds vial
Sample Delivery and Condition	DAM	Sample container damaged
Sample Delivery and Condition	PRE	Sample not properly preserved
Sample Delivery and Condition	TEMP	Sample received at elevated temperature
Sample Delivery and Condition	TMPX	Sample received at elevated temperature, extreme discrepancy
Serial Dilution	SDIL	Serial dilution did not meet %D criterion
Serial Dilution	SDN	Serial dilution not performed
Surrogate	SSH	Surrogate %R high
Surrogate	SSL	Surrogate %R low
Surrogate	SSLX	Surrogate %R low, extreme discrepancy
Surrogate	SSN	Surrogate compound not spiked into sample
Trip Blank	TBH	Trip blank result ≥LOQ
Trip Blank	TBL	Trip blank result <LOQ
Validator Judgment	VJ	Validator judgment (see validation narrative)

ICS = interference check sample
MS = matrix spike
MSD = matrix spike duplicate
QC = quality control
RPD = relative percent difference
RRF = relative response factor

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ATTACHMENT F
Review of Subcontracted Data Validation Reports

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ATTACHMENT F
Review of Subcontracted Data Validation Reports

1.0 INTRODUCTION

The goal of subcontracted data validation is to generate a validated project dataset that is qualified in accordance with Quality Assurance Project Plan (QAPP) requirements and ready for HydroGeoLogic, Inc. (HGL) to upload into the project database, and to do so at a cost savings to HGL's projects. Subcontracted data validation will be performed in accordance with the individual firm's internal procedures and policies; however, the overall procedure must include prereview, validation by qualified personnel, and peer or senior review of all data validation reports before delivery to HGL. All validation should be performed in accordance with the project QAPP and the scope of work provided by HGL.

Note that the guidance presented in this Attachment assumes that the project QAPP presents validation and qualification criteria based on the quality control (QC) requirements of the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) version 5.3. Although a majority of project QAPPs will reference QSM version 5.3 or the similar requirements of QSM versions 5.1 or 5.2, there are still older QAPPs in use that have the data qualification protocols based on the QC requirements of DoD QSM version 4.2 or 5.0. If the guidance presented in this Attachment conflicts with the project QAPP qualification protocols, the requirements of the project QAPP should always take precedence.

2.0 DELIVERABLES

2.1 SUBCONTRACTED DATA VALIDATOR

Subcontracted data validators will deliver data validation reports to HGL. These reports may be in the validation firm's internally derived format; however, HGL prefers that an individual report be prepared for each sample delivery group (SDG) and analytical method within that SDG (although "bundling" methods for metals and wet chemistry parameters is acceptable, in the same fashion as HGL's internally produced data validation reports). Each report should include a summary of every QC element evaluated by the data validator, an identification of discrepancies, the qualification required by this discrepancy, and an identification of the associated samples. Subcontracted data validation reports are required to include a summary of all qualified data. This summary can be provided as a table of qualified results, as a listing of qualifiers assigned by QC element, or as copies of data reporting forms with validation qualifiers applied by hand.

In most cases, the subcontracted validator will also be responsible for providing qualified data electronically in a format that allows upload into HGL's project database (see Section 6.0 of the standard operating procedure [SOP]), usually in the form of an Excel file. The validation firm will

be responsible for data entry, data entry QC, and removal of any residual laboratory-applied flags before delivery to HGL.

2.2 HGL REVIEWER

The HGL reviewer should prepare a review report to document the findings of the review of each subcontracted data validation report. This review should include a discussion of any discrepancies noted in the data validation report, any follow-up communications with the data validator or the laboratory, and any changes to the final data qualifiers assigned by the validator (including qualifiers applied by the laboratory and accepted as the final qualifier by the laboratory). The HGL reviewer is also responsible for ensuring that any HGL modifications to the validator's data qualifiers and other fields applicable to the validation process (including the HGL Value, HGL Qual, Detected, Report Usability, and HGLReason Code fields) are correctly incorporated into the 100 percent QC Excel file generated by the project database and transmitted to the project's database administrator. The HGL reviewer should at a minimum indicate any changes made to the 100 percent QC Excel file by color coding any affected cells. An example of an HGL data validation review report is presented as Attachment F.1.

3.0 INITIAL HGL REVIEW

The initial data validation reports provided by the contractor should be reviewed in-depth by an HGL senior chemist as soon as possible to provide the data validator with timely feedback to guide ongoing validation efforts. Promptly alerting the data validators to any discrepancies allows for data validator to issue correct reports rather than reissuing revised reports. Performing and in-depth review will assist in identifying areas where the data validation contractor's interpretation of QC elements differs from the requirements of the QAPP.

This review should mimic HGL's peer review of an internally generated data validation report (see Section 3.4 of the SOP), including a re-examination of the laboratory data package to verify that no QC discrepancies have been overlooked by the validator. The most common cause for a QC element being overlooked or misinterpreted by the data validator is unfamiliarity with the specific requirements of the project QAPP, which should supersede any corporate validation conventions in place at the validation firm.

4.0 GENERAL HGL REVIEW GUIDELINES

The following are the general guidelines for reviewing data validation reports from subcontracted validators.

4.1 REPORT DETAIL

When conducting data validation, HGL's practice is to identify and discuss all QC discrepancies associated with an analytical fraction, whether those QC discrepancies cause data to be qualified or not. Data validation subcontractors and individual validators vary in the amount of detail that is provided in the report narrative, especially if no corresponding results require qualification. The HGL reviewer should be alert to cases where the validator has indicated no discrepancies for a QC element when, in fact, there were discrepancies, but no qualification is required or no project sample results are associated with that specific discrepancy. Many validation firms provide a checklist with the text of the validation report. If such a checklist is available for review, it should be compared to the report text to check if there are QC discrepancies noted that are not discussed in the report because no qualification was required. This comparison can also assist in verifying that the validation report does not contain any "template" errors.

4.2 APPLICATION OF FINAL QUALIFIERS

In all cases, the final qualifier applied by the data validator must be an allowable project qualifier. When more than one qualifier is applicable to a result, the final qualifier must have been assigned in accordance with the priority of qualifiers presented in the QAPP.

The HGL reviewer should examine the qualified electronic file to ensure that all the validator-applied qualifiers are allowable under the project QAPP and that there are no changes to laboratory qualifiers that do not make sense. For instance, if a laboratory qualifier is U and the final qualifier is B, the HGL reviewer should suspect that the B qualifier is in error and determine the correct final qualifier that should be applied.

5.0 REVIEW OF STAGE 2A DATA VALIDATION ELEMENTS

The HGL reviewer should examine the following elements of each data validation report. The common discrepancies associated with each QC element are also discussed in the following subsections.

5.1 SAMPLE RECEIPT AND DELIVERY

The HGL reviewer should review the validation report and verify that any qualification is performed in accordance with the QAPP.

5.2 HOLDING TIMES

The holding times for preparation and analysis for each analytical method should be presented in the project QAPP. The validator should have used the QAPP conventions for evaluating holding times or provide justification (such as nominal exceedance) for not qualifying results that are associated with holding time exceedances. The validator should have considered any time zone

differences, daylight savings time changes, or "dummy" sample collection times (such as on field duplicates) when evaluating short (≤72 hour) holding times.

5.3 LCS/LCSD RECOVERIES AND PRECISION

Laboratory control sample (LCS) (and laboratory control sample duplicate [LCSD]) recoveries greater than the control limits should not cause qualification of nondetected results unless there is a gross exceedance that is evidence of a problem with the analytical system.

LCS/LCSD relative percent difference (RPD) exceedances should not cause qualification of nondetected results.

Discrepancies shown by polychlorinated biphenyl (PCB)-1016 should be considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 should be considered to affect PCBs 1242, 1248, 1254, and 1260. The validator should have taken this convention into account when applying qualifiers.

Some QAPP data validation protocols establish a two-tiered approach for evaluating LCSs. The HGL reviewer should verify that the validator distinguished between routine and extremely low percent recoveries (%Rs) when applying qualifiers to the associated results.

5.4 MS/MSD RECOVERIES AND PRECISION

The issues applying to LCS (and LCSD) performance also apply to matrix spike (MS)/matrix spike duplicates (MSDs). There are additional issues that affect the evaluation of MS/MSDs.

The association of MS/MSD results to project samples varies by method and by project. Ensure that any identified MS/MSD discrepancies are associated correctly.

Ensure that no qualification of project samples is performed based on discrepancies found in nonsite samples unless the validator has provided an appropriate rationale.

Ensure that no qualification has been performed based on MS/MSD %R discrepancies identified for analytes that are present in the parent sample at greater than 4 times the spiked concentration.

Ensure that project samples from other SDGs that were reported as batch control MS/MSDs were properly identified as project samples and used to qualify project data.

Verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is comparable to the expected concentration in the MSD. If the RPDs are calculated using non-comparable results (different spiked concentrations in the MS and MSD), the validator should have noted this in the evaluation of the RPDs. Note that it may

be justifiable to assign qualifiers based on MS/MSD RPD discrepancies even if MS/MSD recoveries are affected by the "4 times" rule.

Where there are MS/MSD %R discrepancies affecting metals results from methods 6010 or 6020, the laboratory should perform a serial dilution or post-digestion spike (PDS) using the same parent sample, whether the "4x rule" applies to the discrepancy (see Section 5.5).

On occasion, the laboratory will select a member of a field duplicate pair to perform MS/MSD analyses. For organics, the general convention is to qualify only the MS/MSD parent sample for when MS/MSD discrepancies are noted. If an MS/MSD is performed on one of the members of a duplicate pair, however, the MS/MSD results are applicable to both members of the pair, and the HGL reviewer should verify that both samples were qualified.

5.5 SERIAL DILUTIONS AND POST-DIGESTION SPIKES

The use of serial dilution and post-digestion spike results varies depending on when the QAPP was written. The current guidance used in HGL QAPPs follows, but the specific QAPP requirements should be used to evaluate these QC elements.

When a metals MS/MSD analysis shows %R discrepancies, the laboratory should perform a serial dilution and PDS on the MS/MSD parent sample. Serial dilution and PDS results should only be used to modify the qualifiers applied due to MS/MSD %R discrepancies in accordance with the qualification protocols presented in the project QAPP. If the MS/MSD %R is in control for a metal; qualification should not be applied for serial dilution or PDS discrepancies associated with acceptable MS/MSD %R results.

Serial dilution results are applicable to analytes that are present at ≥50 times the limit of quantitation (LOQ) in the MS/MSD parent sample, and PDS results are applicable to analytes that are presented at <50 times the LOQ in the MS/MSD parent sample. The "4x rule" that is used for MS/MSD results is also applicable to PDS results, so there may be situations where a parent sample concentration for a metal is high enough that MS/MSD and PDS results cannot be used to qualify the associated samples, but the concentration below the threshold for using serial dilution results. In these cases, the validators should use judgment to evaluate whether matrix effects are suspected. If the serial dilution results are in control and the parent sample concentration is greater than the LOQ, the serial dilution results can be used as corroborating evidence that there is no matrix effect, even if the concentration is below the ≥50 times the LOQ threshold.

The HGL reviewer should evaluate the validation narrative and verify that serial dilutions and PDSs were evaluated in accordance with QAPP criteria.

If the laboratory performed neither a serial dilution nor a PDS using a project sample, then matrix effects cannot be ruled out. The validator should have reviewed available MS/MSD data, site

results reported from other data packages, and the case narrative and determine whether qualification is necessary.

5.6 METHOD BLANKS

The evaluation of laboratory blank results is one of the few QC elements where the results can meet acceptance requirements for reporting data (instead of performing corrective action), but the associated results will still be qualified. HGL often sets acceptance criteria for laboratory blanks using the QSM criteria, which are "No analytes detected > ½ LOQ (>LOQ for common laboratory contaminants) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater." These acceptance criteria are the thresholds above which the laboratory should take corrective action and evaluate the need to reanalyze any affected samples. However, HGL's convention is that any contamination detected in laboratory blanks at or above the associated detection limit (DL) must be used to establish an artifact threshold and qualify associated results below that threshold. This qualification must be applied whether the associated blank result is above the acceptance criterion or below it.

This division between acceptance criteria and qualification criteria is a common source of error in subcontracted evaluation of laboratory blanks. The HGL review must ensure that the validator has evaluated all blank results at or above the DL and applied qualification in accordance with the validation conventions. For metals, this will also include the evaluation of blanks with negative concentrations that have an absolute value greater than the DL.

5.7 FIELD BLANKS

Field blanks are evaluated in a similar manner as method blanks (Section 5.5). Two main differences are (1) the artifact threshold calculated from concentrations in field blanks is *not* adjusted for sample-specific factors; and (2) most field blanks are aqueous and conversion to equivalent solid units is not straightforward for some analytical methods.

Ensure that the data validator correctly calculated the artifact threshold and made any corrections for conversion from water to soil units.

5.8 FIELD DUPLICATE PRECISION

Ensure that the appropriate criterion, absolute difference for low-level results of RPD for high-level results, was used to evaluate each set of duplicate results, as specified in the QAPP.

The association of field duplicate results to project samples beyond the parent sample varies by method and by project. Ensure that any identified field duplicate discrepancies are associated correctly.

5.9 SURROGATE RECOVERIES

The HGL reviewer should examine any results qualified as a result of surrogate discrepancies noted in diluted samples. Generally, qualification should not be applied for surrogate discrepancies if the sample dilution factor was greater than 5 and the surrogates were added prior to dilution.

5.10 METHOD-SPECIFIC QC CHECKS

Method-specific QC elements include such checks as pH buffer checks, cyanide distillation standards, synthetic precipitation leaching procedure extraction blanks, and replicate precision for total organic carbon. If these checks are reported in a Stage 2A data package, the validator should review these items. If the review guidelines are not included in the QAPP, the validator should consult with the project chemist to develop a review and qualification approach.

6.0 REVIEW OF STAGE 2B DATA VALIDATION ELEMENTS

Stage 2B QC elements are specific to individual analytical methods.

6.1 GC/MS ORGANICS

Gas chromatography (GC)/mass spectrometry (MS) organics include analyses for volatile organic compounds (VOCs) and for semivolatile organic compounds (SVOCs), most commonly by SW-846 methods 8260B or 8260C and 8270D, respectively.

6.1.1 Instrument Tuning

It is rare for a laboratory data package to include mass spectrometer tuning discrepancies. Data validation reports for this QC element will rarely include more than a statement that tuning frequencies and results were acceptable.

6.1.2 Instrument Initial Calibration

A common source of error in subcontracted data validation reports is the confusion between instrument performance criteria for Method 8260B (and SVOCs method 8270C, which is now infrequently performed) and target compound performance criteria in the evaluation of initial calibration data. Subcontracted data validation reports should note that the following QC elements were reviewed, along with any noted discrepancies:

- System performance check compounds (SPCCs) evaluated against analyte-specific mean relative response factor (RRF)
- Calibration check compound (CCCs) evaluated against percent relative standard deviation (%RSD) of 30 percent

- Target analytes (including CCCs that are also target analytes) evaluated against %RSD of 15 percent (20% for analysis by 8270-SIM) or r^2 of 0.99

The failure of an SPCC or CCC to meet *the SPCC- or CCC-specific criteria* constitutes a failure of the entire calibration and can cause rejection of all associated results; whereas the failure of a target compound to meet the linearity criterion constitutes a failure for only that target compound and causes less severe qualification. In some cases, a CCC can pass the CCC criterion but fail the target analyte criterion. The reverse can also be true.

> *Example*: Method 8260B CCC vinyl chloride is reported calibrated to a mean RRF with %RSD of 17.5 percent. The requirement for VOCs CCCs is that each has a %RSD of no greater than 30 percent. Vinyl chloride shows acceptable performance as a CCC; however, the target analyte criterion is for %RSD to be no greater than 15 percent. Vinyl chloride does not meet the acceptance criterion for target analytes. The effects, if any, of this discrepancy would be considered to affect vinyl chloride alone and not to be indicative of an instrument performance issue.

> *Example*: Method 8270C CCC di-n-octyl phthalate is reported calibrated to a mean RRF with %RSD of 31.2 percent, but the laboratory elected to fit the calibration sequence to a curve with an r^2 of 0.996. The requirement for SVOCs CCCs is that each has a %RSD of no greater than 30 percent. Even though a r^2 of 0.996 meets the acceptance criterion for a target analyte, this CCC does not meet the acceptance criterion of %RSD $\leq$30 percent. Although mean RRF is not used as the calibration relationship for this compound, the laboratory should have performed corrective action in this case.

Some QAPPs include a requirement that target analytes also be evaluated against analyte-specific mean RRF requirements. This should only be done if included as a QAPP requirement, such as for Methods 8260C and 8270D and the selected ion monitoring (SIM) modifications to these methods; if the data validator has qualified data based on target compound mean RRF when not required by the QAPP, the data validation reports should be revised to remove this extraneous qualification.

6.1.3 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. SPCC and CCC performance evaluation is not required for second source calibration verification standards.

6.1.4 Instrument Continuing Calibration

The data validator should have evaluated continuing calibration verification (CCV) standards for SPCC, CCC, and target analyte performance in a manner similar to the evaluation performed for initial calibrations. The data validation report should note that the SPCCs met method-specified continuing calibration RRF criteria and CCCs met method-specified percent difference (%D)

criteria. For GC/MS methods, CCV standards performed at the end of the analytical sequence are only required to meet the %D requirement for target analytes; SPCC, CCC, and minimum target analyte RRF performance evaluation is not required for ending CCVs.

Target analytes are evaluated against the target analyte criterion of no greater than 20 percent. Some QAPPs may also require that target compounds also meet minimum continuing calibration RRF criteria in the opening CCV standards, such as for Methods 8260C and 8270D and the SIM modifications to these methods. If the QAPP does not require the evaluation of target compound RRFs, the data validation report should not use this QC element to assign qualifiers to target analyte data.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias. The data validation report should not use the direction of bias when evaluating continuing calibration results.

6.1.5 GC/MS Internal Standards

Internal standard compounds must be spiked into every sample, standard, and blank analyzed by GC/MS methods. Internal standards must meet the method area and retention time criteria for peak area and retention time. Older versions of the DoD QSM required that the peak area for each internal standard compound must be no less than 50 percent and no greater than 200 percent of the peak area for that internal standard compound in the midpoint standard in the associated initial calibration sequence. The retention time for each internal standard must be within 10 seconds of the retention time of the midpoint standard in the associated initial calibration sequence. While this requirement was retained in DoD QSM version 5.1 and subsequent versions, internal standard acceptance criteria were expanded to allow for the daily initial CCV to be used for this comparison on days when initial calibration is not performed.

6.2 GC AND HPLC ORGANICS

GC and high-performance liquid chromatography (HPLC) organics include analyses for pesticides (organochlorine and organophosphorus), PCBs, explosives, herbicides, and petroleum products. GC and HPLC analyses use dual columns or dual detectors to identify target analytes. Some laboratories assign the same quantitative significance to both columns/detectors, while others specify a dedicated primary and secondary column/detector. If presented, the QC data for both the primary and secondary column/detector should have been evaluated. In cases where instrument QC discrepancies affect one column/detector and not the other, some degree of interpretation by the validator is required to determine the effect on the associated samples.

6.2.1 Instrument Initial Calibration

The interpretation of GC initial calibration is generally straightforward. If any discrepancies are identified in the initial calibrations associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

6.2.2 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. If any discrepancies are identified in the second source calibration verifications associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

6.2.3 Instrument Continuing Calibration

If any discrepancies are identified in the continuing calibration verifications associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias. The data validation report should not use the direction of bias when evaluating continuing calibration results.

6.2.4 Degradation Summary

The evaluation of this QC element is straightforward and should not be a source of error in the validation report.

6.2.5 Retention Times

Verify that retention time shifts were evaluated in the data validation report.

6.2.6 Confirmation

Verify that confirmation for detected results was evaluated and that confirmed results were qualified if confirmation agreement criterion (RPD ≤40%) was not met.

Most GC and HPLC methods use a second column or second detector to confirm detected results, and the QSM requires that QC results for the confirmation column/detector meet the same QC criteria as the primary column/detector. HGL's preferred convention for qualifying results is by the detector used to report the results for each analyte. This reporting can vary on a sample-specific basis to address sample matrix characteristics that affect one column/detector more than the other.

> *Example*: The laboratory has designated column X as the primary column for reporting herbicide results by Method 8151A. The initial calibration associated with all sample analyses has an acceptable %RSD for dinoseb in column X but a high %RSD for dinoseb in column Y. All reported dinoseb results are nondetections; however, of the nine samples associated with this initial calibration, six have dinoseb reported from column X and three have dinoseb reported from column Y. The three dinoseb results reported from column Y should be qualified UJ; the six dinoseb results reported from column X would not require qualification for an initial calibration discrepancy.

6.3 METALS

Metals analyses often contain discrepancies between the validation criteria applied by the validator and the QAPP criteria. The HGL reviewer should be especially alert to errors in evaluating continuing calibration blanks (CCBs) (Section 6.3.7), and interference check samples (ICSs) (Section 6.3.8).

6.3.1 Instrument Tuning

Instrument tuning data is not always available on summary forms. Verify that the validators were able to evaluate instrument tuning data, including mass windows, peak widths, and %RSD of scans.

6.3.2 Internal Standards

Verify that the validators reviewed internal standard results. In some cases (especially with short analyte lists), there may be internal standards that do not meet acceptance limits but are not associated with target metals. Some laboratories will also choose a secondary internal standard to quantify a metal if the primary internal standard does not meet acceptance criteria.

6.3.3 Initial Multipoint Calibration

Initial multipoint calibration is required for cold vapor atomic absorption and graphite furnace atomic absorption (GFAA) methods. It is not required for inductively coupled plasma (ICP) atomic emission spectroscopy or ICP-MS analyses; however, if a multipoint initial calibration is performed, it must meet the acceptance criteria in the QAPP. If the supplemental calibration checks

described in Section 6.3.4 or 6.3.5 are acceptable but the multipoint initial calibration was out of control, the associated results should have been qualified by the validator.

6.3.4 Low-Level Calibration Verification

The integration of the results for initial calibration, low-level calibration standards, and contract required detection limit standards is a common source of validator error. The HGL validation reviewer should ensure that the validator understands how to evaluate these three QC elements in totality and apply the correct final qualifier to any results affected by discrepancies associated with the initial calibration QC checks.

6.3.5 High-Level Calibration Verification

Verify that the validator evaluated high-level calibration standards and qualified any results reported from above the calibrated range.

6.3.6 Initial and Continuing Calibration Verification

Most laboratories use initial calibration verification standard (ICV) analyses as a second source verification check. HGL's preferred convention is to associate ICV results with all sample results in an analytical sequence and to associate CCV standard results only with sample results "bracketed" by a given CCV. A result is considered bracketed by a CCV if that CCV is the last CCV analyzed before that result was generated or is the first CCV analyzed after that result is generated.

Note that some laboratories evaluate ICV/CCV results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. For metals methods, HGL considers it to be acceptable to evaluate the direction of the bias when qualifying associated results. The HGL validation reviewer should ensure that the data validator correctly identified ICV/CCV results that did not meet acceptance criteria and that any discrepancies were associated in accordance with the QAPP conventions.

6.3.7 Continuing Calibration Blanks

CCBs present the same common source of error as do method blanks: the confusion caused by the qualification criteria differing from acceptance criteria (see Section 5.5). The HGL reviewer should ensure that all CCB contamination at or above the DL was evaluated for the potential effect on associated sample results, not just the CCB contamination that was present above the acceptance criteria.

CCBs are always aqueous; the concentrations should be converted to the equivalent soil concentration when comparing the blank results to the concentrations found in any associated soil

samples. The HGL reviewer should verify that the appropriate conversion was made by the validator.

HGL's preferred convention is to associate initial calibration blank (ICB) results with all sample results in an analytical sequence and to associate CCB results only with sample results bracketed by a given CCB. A result is considered bracketed by a CCB if that CCB is the last CCB analyzed before that result was generated or is the first CCB analyzed after that result is generated. The HGL reviewer should verify that the association conventions used by the data validator are those in the QAPP.

The HGL validation reviewer should ensure that the data validator correctly identified ICB/CCB results that did not meet acceptance criteria and that any discrepancies were associated in accordance with the QAPP conventions. The HGL reviewer should also verify that any blank contamination with concentrations or absolute values of concentrations greater than the acceptance levels were noted by the validator with a discussion of any laboratory corrective action.

6.3.8 Interference Check Sample Results

The evaluation of ICS data is another common source of error in data validation reports. One of the primary reasons for this is that laboratory data summary reporting forms generally provide inadequate information for the data validator to be able to evaluate the results that are presented. The HGL reviewer should evaluate whether the data validator evaluated ICS A (ICSA) results in accordance with the QAPP and applied the correct qualifiers. Common errors are:

- Failure to evaluate ICSA results at all (some firms consider this a Stage 4 item);
- Failure to identify severe discrepancies (results greater than the LOQ or converted water-to-soil LOQ); and
- Failure to interpret discrepancies and apply qualification in accordance with the QAPP.

Note that QAPPs written to include QSM version 5.1 (or later) requirements will require the absolute value of each unspiked analyte in the ICSA to be less than one-half the LOQ; QAPPs written in accordance with older versions of the QSM will include a requirement that the absolute value of each unspiked analyte to be less than the limit of detection.

The evaluation of ICS AB results is generally straightforward, and this QC element rarely shows discrepancies.

6.3.9 Recovery Test Recoveries

GFAA methods use recovery tests to determine if the sample matrix has affected reported results. The method requires a recovery test to be performed on a representative sample in each preparation

batch, but in practice, laboratories perform recovery tests on a sample-specific basis. The HGL reviewer should verify that this QC element was evaluated in accordance with QAPP requirements.

6.3.10 Method of Standard Addition Results

The method of standard additions (MSA) is associated with GFAA analyses; this procedure is rarely performed as virtually all laboratories perform sample-specific recovery tests rather than batch-specific recovery tests. If MSA results are reported in a data package, the HGL reviewer should consult with the HGL Senior Chemist.

6.4 GENERAL CHEMISTRY

General chemistry parameters include a wide variety of analytical parameters and methodologies, including colorimetry, ion chromatography, GC, and infrared spectrometry. Usually, these parameters are secondary data that are used to determine the potential for a site to undergo monitored natural attenuation or the progress of monitored natural attenuation. Often, these tests will only require a Stage 2A data review; however, some parameters, such as cyanide, perchlorate, anions, or total organic carbon, will on occasion require Stage 2B validation.

In many cases, the review of general chemistry QC parameters is similar to the review of the corresponding parameters for metals. Method-specific QC parameters should be discussed in the QAPP along with the acceptance criteria and qualification requirements. Some laboratories do not have summary forms for Stage 2B QC elements and the raw data will need to be examined by the validator to evaluate performance.

The HGL reviewer should ensure that each general chemistry parameter was validated to the appropriate stage, and that all appropriate QC elements were validated. If it is found that the subcontracted data validator is not applying the correct stage of validation to one or more general chemistry parameters, this should be brought to the attention of the HGL project manager and the project chemist.

HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE
	Approved by: Corporate Quality Manager

Sampling Equipment Cleaning and Decontamination	SOP No.: 411.02 (formerly 2.01)
	SOP Category: Environmental Services
	Revision No.: 5
	Revision Date: June 18, 2020
	Review Date: June 2022

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe field methods to be used for cleaning and decontaminating sampling equipment.

This procedure is specifically applicable to sampling equipment that has been used to collect environmental samples or could have been exposed to contamination that could affect worker safety and/or the integrity of the analytical results of the media sampled.

Other decontamination procedures may apply to a specific project; refer to the project-specific planning documents for project-specific decontamination methods and schedules.

Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager and discussed in the approved project plans. Deviations from requirements are documented sufficiently to re-create the modified process.

2.0 SUMMARY OF THE METHOD

This SOP describes the procedures to be followed to achieve effective decontamination as follows: (1) remove contaminants from contaminated surfaces, (2) minimize the spread of contamination to uncontaminated surfaces, (3) avoid any cross-contamination of samples, and (4) minimize personnel exposures. The intent is to accomplish the required level of decontamination while minimizing the generation of additional solid and liquid waste.

3.0 DEFINITIONS

ASTM Type II Water: This is the type of deionized reagent grade water, as defined by ASTM International, used in the final rinse of surfaces of contaminated equipment.

Equipment: Equipment comprises those items (variously referred to as "field equipment" or "sampling equipment") that are necessary to conduct sampling activities but that do not directly contact the samples.

Laboratory Detergent: This is a standard brand of phosphate-free laboratory detergent such as Liquinox® ~~or Luminox®~~. Liquinox® is a traditional anionic laboratory detergent used for general cleaning and when there is concern that harsher cleaners could affect the stability of the sampling equipment. ~~Luminox® is a specialized detergent that can remove oils and organic contamination. It may be used in lieu of a solvent rinse step in cleaning equipment for trace contaminant sampling~~.

~~Where not specified in these procedures, either detergent is acceptable. The project-specific plans should indicate if Luminox® use is acceptable~~.

Organic-free Water: This is tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water, and it should contain no detectable pesticides, herbicides, or extractable organic compounds and no volatile organic compounds above minimum detectable levels for a given set of analyses. Organic-free water obtained by other methods is acceptable as long as it meets the above analytical criteria.

Potable/Tap Water: Potable/tap water is provided by local city sources and is safe for consumption. Chemical analysis of the water source is not required before it is used. Deionized water or organic-free water may be substituted for tap water.

Sampling Devices: This is equipment used to acquire samples.

4.0 GENERAL REQUIREMENTS

All work is performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan for relevant health and safety requirements. Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager. Deviations from requirements are documented sufficiently to re-create the modified process.

5.0 EQUIPMENT AND SUPPLIES

The following equipment is specific to decontamination requirements and does not include required safety equipment and field documentation described in the site-specific plans. Project-specific plans should be consulted for any additional equipment or deviations from the list below:

- Laboratory detergent,
- Brushes (not wire wound),
- Paper towels/rags,
- Squirt bottles (one for each decontamination fluid),
- 5-gallon buckets or decontamination pad/kiddie pool to contain decontamination fluids,
- Potable water,
- Deionized water,
- Drums or containers for decontamination fluids/solids,
- Drum/container waste labels,
- Sampling containers for decontamination fluid/solid sampling,
- Aluminum foil,
- Steam cleaner, and
- Generator and fuel.

6.0 PROCEDURAL STEPS

Decontamination of sampling devices is performed in a designated decontamination area, removed from any sampling or dedicated office location. This designated area must be in a location free of direct exposure to airborne and radiological surface contaminants and upwind of any field activities that could jeopardize the decontamination procedures or cross contaminate the cleaned equipment.

6.1 GENERAL

The following general rules are followed for decontamination operations:

- Contaminated or dirty sampling devices/equipment should not be stored with or above clean (decontaminated) sampling devices/equipment.
- Clean, decontaminated sampling devices should be segregated from all other equipment and supplies.
- Paint or any other coatings must be removed from any part of a sampling device that may either contact a sample or may otherwise affect sample integrity. After such coatings are removed, the sampling device must be decontaminated using the appropriate method.
- For any of the specific decontamination methods that may be used, the substitution of higher-grade water is permitted (for example, using deionized water in place of tap water). However, deionized water is less effective than tap water in rinsing away detergent during the initial rinse.
- Decontaminated sampling devices and all filled and empty sample containers are stored in locations protected from exposure to any contaminant.
- The method for decontaminating sampling devices and the exterior of sample containers that have been exposed to radioactive material is based on the material contaminated, the sample medium, the radiation levels, and the specific radionuclides to be removed.
- The release of decontaminated sampling devices and sample containers for unrestricted use is based on site-specific criteria. These site-specific criteria should be detailed in the project-specific plans.
- Rags/paper towels used during decontamination activities may become a hazardous waste and require segregation. Refer to the project-specific plans for hazardous waste disposal requirements.
- Sampling devices must be decontaminated before being used in the field to prevent potential cross-contamination of a sample.
- Sampling devices must be decontaminated between samples to prevent cross-contamination.

- Sampling devices must be decontaminated at the close of the sampling event before being taken off site.
- An acceptable alternative to cleaning and decontaminating sampling devices is using items cleaned or sterilized by the manufacturer that are discarded after one use. Care must be exercised to ensure that such previously cleaned or sterilized items do not retain residues of chemical or radioactive sterilizing agents that might interfere with analytical techniques.
- Whenever visible dirt, droplets of liquid, stains, or other extraneous materials are detected on the exterior of a sample container, the exterior surfaces must be decontaminated. This step should be performed before the container is placed in a sample cooler or shipping container.
- For sample containers used in controlled access areas, more rigorous cleaning and/or radiation monitoring may be required before removal from the site. Refer to the project-specific planning documents for details.
- Decontamination fluids/solids as well as other used cleaning supplies, such as paper towels and rags, should be treated as investigation-derived waste and managed in accordance with the project-specific planning documents.

6.2 DECONTAMINATION METHODS

The following decontamination methods are examples of some of those most commonly used in field investigations. Note that the decontamination methods described in this section are for guidance only; the project-specific planning documents and the SOPs referenced in them provide the actual procedures that must be followed. The field operations manager may need to adjust decontamination practices to fit the sampling situation and applicable requirements. All variances from the project-specific planning documents must be approved by the project manager in advance and documented. Procedures for packaging and disposing of all waste generated during decontamination are described in the project-specific planning documents.

6.2.1 Water Level Indicators

The following steps are taken to decontaminate water level indicators. Unless conditions warrant, it is only necessary to decontaminate the wetted portion of the measuring tape. It may be more practical to decontaminate the tape as it is being rewound, but with the reel several feet away from the wellhead (see project-specific planning documents):

1. Wash with detergent and tap water.
2. Rinse with tap water.
3. Rinse with deionized water.

6.2.2 Submersible Groundwater Pumps

The following procedures are taken to decontaminate submersible pumps used to collect groundwater samples. This is the general procedure for non-dedicated pumps, unless the dedicated pump is being removed from the well.

1. Disconnect and discard the previously used tubing from the pump. Wash the pump exterior with detergent and water.
2. Prepare and fill three containers with decontamination solutions consisting of Container 1, tap water and detergent solution; Container 2, a tap water rinsing solution; and Container 3, a deionized water final rinsing solution. The containers should be large enough to hold the pump and 1 to 2 liters of solution. An array of 2-foot-long 2-inch PVC pipes with bottom caps is a common arrangement. Buckets can also be used as long as the water covers the intake screen of the pump. The containers should be labeled to ensure that decontamination is completed in the correct steps. The solutions should be changed at least daily.
3. Place the pump in Container 1. Turn the pump on and circulate the detergent and water solution through the pump and then turn the pump off.
4. Place the pump in Container 2. Turn the pump on and circulate the tap water through the pump and then turn the pump off.
5. Place the pump in container 3. Turn the pump on and circulate the deionized water through the pump and then turn the pump off.
6. Disconnect the power and remove the pump from Container 3.
7. Decontaminate the power lead by washing it with detergent and water, followed by tap water and a deionized water rinse. This step may be performed before washing the pump, if desired.
8. Wind the power lead back on a reel, and place the pump and reel in a clean plastic bag.

6.2.3 Bladder Pumps

The following procedures are used to decontaminate bladder pumps that use disposable bladders. If the bladder pump being used does not have a disposable bladder, the decontamination procedures outlined in Section 6.2.2 should be used.

1. Disconnect and discard previously used tubing from the pump.
2. Completely disassemble the pump, being careful not to lose the check balls, O-rings, ferrules, or other small parts.
3. Remove and discard the pump bladder.

4. Clean all parts with tap water and detergent, using a brush if necessary to remove particulate matter and surface films.
5. Rinse thoroughly with tap water.
6. Rinse thoroughly with deionized water.
7. Install a new pump bladder.
8. Reassemble the pump and wrap it in aluminum foil or store it in a decontaminated pump storage tube.

6.2.4 Small Tools/Samplers

The following procedures are used to decontaminate small tools/samplers (e.g., stainless steel bowls, sample trowels, and hand augers).

1. Wash the tools/samplers with detergent and tap water, using a brush to remove particulate matter and surface film.
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with deionized water.
4. Wrap the tools/samplers in aluminum foil or place them in a clean plastic bag.

6.2.5 Drilling and Direct-Push Technology Sampling Equipment

These procedures are used for drilling and direct-push technology (DPT) sampling activities involving the construction of monitoring wells to be used for collecting groundwater samples or for collecting soil and groundwater samples.

6.2.5.1 Drill and DPT Rig

Any portion of the drill or DPT rig or backhoe over the borehole or sample location that has come into contact with soil or groundwater (mast, backhoe bucket, drilling platform, hoist, cathead) should be steam cleaned (detergent and high-pressure hot water) between boreholes or sample locations. A decontamination pad should be constructed as specified in the project-specific plans to contain soil and decontamination fluids.

6.2.5.2 Downhole Drilling and DPT Equipment

The following is the standard procedure for field cleaning augers, drill stems, rods, tools, and associated equipment.

1. Wash the equipment with tap water and detergent, using a brush if necessary to remove particulate matter and surface film. Steam cleaning may be necessary to remove matter that

is difficult to remove with the brush. Drilling equipment that is steam cleaned should be place on racks above the floor of the decontamination pad. Hollow-stem augers, drill rods, drive casing, and other equipment that is hollow or has holes that transmit water or drilling fluids should be cleaned on the inside with vigorous brushing or steam cleaning.

2. Rinse the equipment with tap water.
3. Remove the equipment from the decontamination pad and cover it with clean plastic or reinstall the equipment on the drill rig.

6.3 QUALITY CONTROL

The effectiveness of the decontamination procedures is monitored by submitting samples of rinse water to the laboratory for low-level analyses of the parameters of interest, also referred to as equipment blanks. An attempt should be made to select different sampling devices each time devices are decontaminated to ensure that a representative sampling of all devices is obtained over the length of the project. Equipment blanks should be collected as specified in the project-specific planning documents.

7.0 RECORDS

Documentation generated as a result of this procedure is collected and recorded in a field logbook in accordance with procedures listed in SOP 300.04: *Field Logbook Use and Maintenance*.

8.0 REVISION HISTORY

Revision 0		Initial Release
Revision 1	December 2010	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2		Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	July 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 4	February 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 5	June 18, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 2.01 to 411.02.

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Procedure # SOP-ENV-01	Title: DEER TISSUE SAMPLING	Revision # 00
Effective Date: 06/01/2022	Approved By: Todd Belanger	Last Revised: n/a

1. PURPOSE

The purpose of this SOP is to describe the general methods to be employed when collecting deer tissue samples for analysis during environmental investigations where PFAS compounds are part of the subject of investigation. The **SOP PFAS ENV-01 PFAS Sampling Guidance** provides an in-depth discussion of prohibited and approved materials and should be used in conjunction with this SOP.

2. RESPONSIBILITIES

Role	SOP-specific Responsibilities
Project Biologist	Specifies the types and quantities of 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/QAPP. 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.

3. RELEVANT DEFINITIONS

Term	Definition
PFAS	Per- and poly-fluoroalkyl substances

4. REQUIRED EQUIPMENT

Equipment	Brief Description of Function and Purpose
Sampling tools	Disposable stainless-steel scalpels, stainless steel bowls, PFAS-free gloves, PFAS-free water.
Sample containers	Sample bags (verified PFAS-free) provided by the analytical laboratory. Coolers for sample shipment.
Logbook	Paper or electronic field forms for documenting field activities. No weatherproof field books.
Chain-of-custody (CoC) forms	For tracking sample details and chain-of-custody, and for providing instruction on sample analysis to analytical laboratory.

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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 designated safety representative will review the relevant site-specific activity hazard analyses (AHAs) prior to implementing this SOP. Any health and safety products will follow the guidance provided in **SOP No. 411.01 PFAS Sampling**. Additional PPE may be required for sampling personnel such as waders and personal floatation devices. Ensure that these materials that will come in contact with the sampling media do not consist of water-resistant coatings or other PFAS containing materials or substances.

5.2. General Requirements for all Sample Methods

5.2.1. Documentation

5.2.1.1. The Sampling Team Leader or designee shall record the description of sample locations, sample type, and any other relevant or notable details on the Field Sampling forms and/or on project-specific sampling forms. Whenever possible, the Sampling Team Leader or designee shall also record the sample locations based on the deer harvest areas provided (**Exhibit 1**) on the Field Sampling form (**Exhibit 2**). The Sampling Team Leader or designee shall record other information as specified in the approved work plan, including completion of daily field notes.

5.2.2. Sample 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, QAPP and **SOP No. 411.01 PFAS Sampling**. 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 samples shall be analyzed in the field and/or at the analytical laboratory as described in the approved work plan/QAPP. The Sampling Team Leader or designee shall collect the quantities and types of Quality Assurance (QA)/QC samples specified in the approved work plan/QAPP to ensure proper QC review of each sampling event.

5.3. Sampling Methods for Deer Tissue Sampling

Deer tissue sampling includes all types of deer tissue used in analytical biological sampling including, but not limited to, muscle and liver tissue.

Deer tissue samples may be collected using several methods depending on the timing and type of harvest. Direct coordination with state agents or local hunters may be utilized to harvest deer so the level of cooperation may vary sample to sample. This SOP will outline methodology for obtaining sufficient deer tissue without accidental PFAS contamination after the deer has been harvested.

5.3.1. Preparation for Deer Tissue Sampling

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

1. The Sampling Team Leader shall provide notifications to local hunters through coordination with State and local agencies and the hunt manager. This notification will request cooperation with

sampling efforts. Instruction will be provided to hunters on where to bring harvested deer so that samples can be collected. Hunters will specifically be instructed to retain the liver for sampling, rather than discarding it when field dressing deer.

2. The Sampling Team Leader shall review the applicable section(s) of the work plan/QAPP to confirm the sample location, quantities, required sample containers, and other relevant information.
3. Once notified of a harvest to be sampled the Sampling Team Leader shall determine the optimal sampling procedure and equipment required to collect the sample, unless already specified in the work plan.
4. The Sampling Team will navigate to the sample location, make initial observations, and complete the required documentation (see Section 5.2.1).
5. The Sampling Team shall review **SOP No. 411.01 PFAS Sampling** and document any deviations from the SOP and their solutions.
6. The Sampling Team shall don clean, powder free nitrile gloves before each sampling event.
7. 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 sample location. All equipment utilized shall be decontaminated prior to use.

5.3.2. Collection of Deer Tissue Samples

5.3.2.1. Local hunters will be notified in advance of the survey and participating hunters will be instructed to contact the Sample Team Leader. Level of participation may vary case by case and the Sample Team Leader will need to coordinate with the hunters to sample harvested deer in a safe, secure location, either where the deer was harvested or a secondary location.

5.3.2.2. Following the preparatory actions (Section 5.3.1), the Sampling Team shall complete the following steps to collect samples from deer harvested:

1. Record all applicable observations about the harvest including: time, date, location, position of projectile entry hole, sex, approximate age, height and weight, or anything else specified by the Work Plan.
2. Using a new disposable scalpel for each animal, expose muscle tissue in rump area to be sampled. Using scalpel, remove 100-200 grams (approximately fist-sized) of tissue to sample.
    a. Use stainless steel knives only, preferably disposable scalpels, and PFAS-free gloves
    b. Do not include skin in tissue sample. Try to make sure that hair/feathers are not included with the tissue sample.
    c. Do not collect sample from area that may have been contaminated with lead from the bullet used to kill the animal. Make sure that the sample area is away from the path of the bullet.
3. Rinse sample with PFAS-free water prior to packaging to remove any excess hair, blood, dirt, etc.
4. Repeat steps 1 and 2 removing 100-200g of liver tissue.
5. Samples will be transferred directly into laboratory provided containers once the tissue is removed and washed.
6. When sample containers are filled, secure the containers and place on ice as soon as possible. Samples must be protected by from light and stored at a temperature between 0 – 6 °C (32 – 43 °F) before and during transport.
7. Perform post-sampling activities (Section 5.3.3)

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5.3.3. Post Sampling Activities for Tissue Sampling

The following steps shall be completed once tissue 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 were collected, including necessary QC samples as specified in the approved work plan/QAPP.
3. The Sampling Team Leader or designee shall ensure the samples are properly stored until they can be shipped for analysis.
4. The Sampling Team will decontaminate reusable sampling equipment as described in Section 5.4.2 or as specified in the approved work plan/QAPP.
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 Equipment Decontamination

Depending on the equipment used there may be no need for Decontamination. If the Sample Team chooses to use disposable stainless-steel scalpels and disposable PFAS-free gloves then equipment is single use and must be properly disposed of after each sample is received. If reusable sample equipment is used the decontamination process outlined in Section 5.4.2. must be followed after each sample is collected.

5.4.1. PFAS-free Water

5.4.1.1. The term PFAS-free water is defined here as water that does not contain significant concentrations of any compound in a specific PFAS analyte list that is being analyzed at a project-defined level. The significant concentrations depend on project data quality objectives and could, for instance, be less than the laboratory reporting limit, <1/2 the limit of quantitation, or other defined criteria for the specific PFAS compound of interest (ITRC, 2022). Note: The confirmation of PFAS-free water should always be performed prior to the commencement of work. Site or public water supplies have been identified in many instances to contain detectable levels of PFAS.

5.4.1.2. One important consideration for each project site is to identify a PFAS-free water source to use for decontamination of sampling equipment when applicable. The decontamination of sampling tools or small equipment parts can be performed using laboratory-supplied verified PFAS-free water. Other water can only be used for decontamination purposes if it has been analyzed and shown to be PFAS-free as defined for the project.

5.4.2. Decontamination Procedures

5.4.2.1. Sampling equipment should be thoroughly decontaminated before mobilization to each investigation area and between sample locations at each investigation area or as required in the site-specific QAPP and **SOP NO. 411.02 Sampling Equipment Cleaning and Decontamination**. Field sampling equipment, including knives, bowls, and other nondedicated equipment used at each sample location, requires cleaning between uses.

5.4.2.2. Decontamination of reusable sampling equipment:

1. Upon donning a new pair of nitrile gloves, equipment will be:
2. Rinsed and scrubbed in a bucket with a mix of Alconox® (or similar) cleaning solution and potable water;
3. Rinsed in a bucket of clean potable PFAS-free water;
4. Second rinse using reagent-grade methanol;
5. Rinse using deionized water;
6. Final rinse with laboratory-provided, "PFAS-free" water, as appropriate;

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Effective Date: 06/01/2022	Approved By: Todd Belanger	Last Revised: n/a

7. All rinsate should be collected in a sealed pail for disposal.

5.4.2.3. 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
ITRC PFAS Fact Sheets, Interstate Technology Regulatory Council.	PFAS guidance on sampling and avoiding cross contamination.
New York State Department of Environmental Conservation (NYSDEC), 2021. Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs. June 2021.	Project state PFAS guidance.

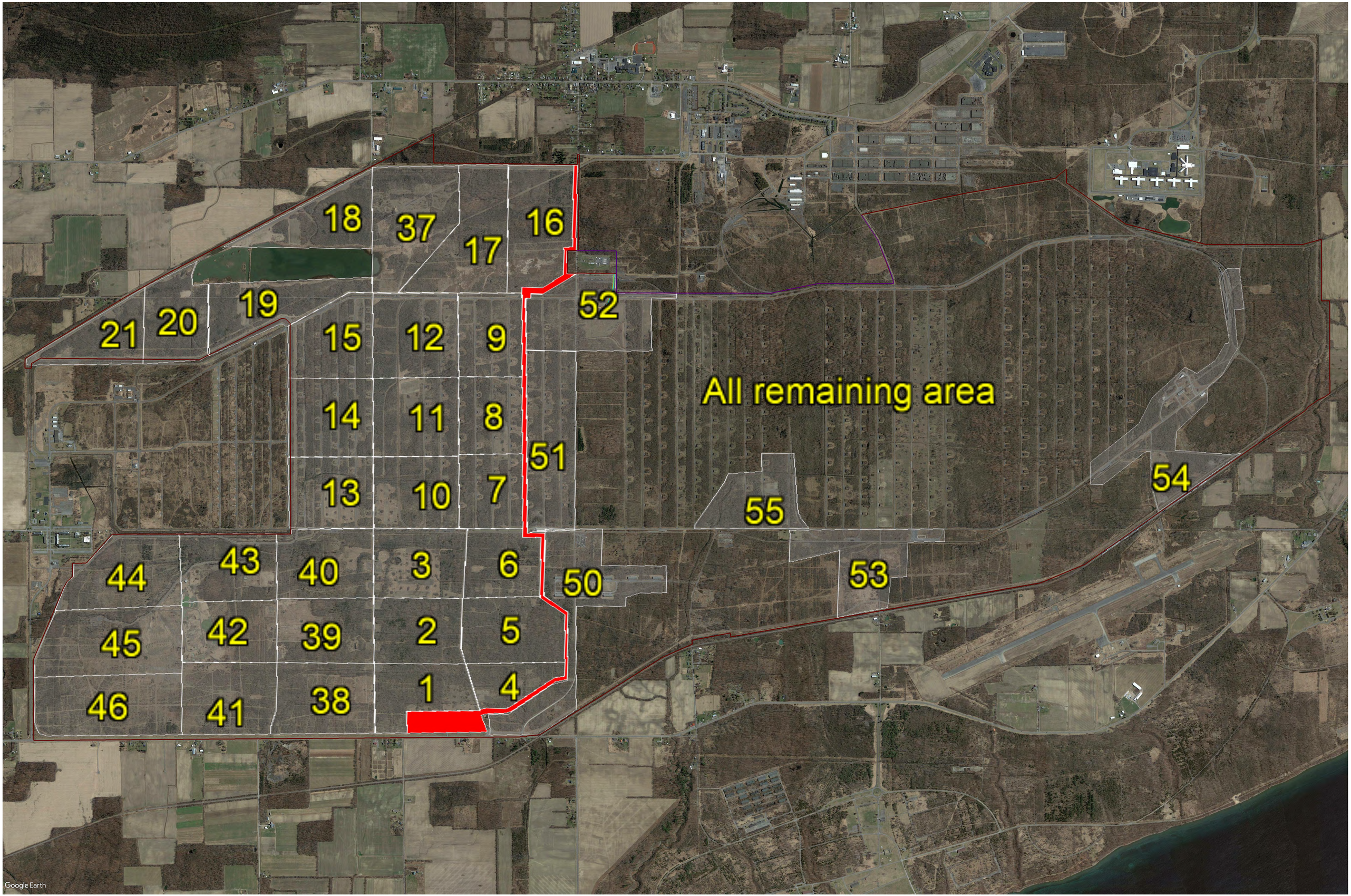
7. EXHIBITS

Exhibit 1: Deer Harvest Areas Map

Exhibit 2: Deer Tissue Sampling Form

8. REVISION HISTORY

Rev.	Date	Summary of Changes	Reason for Revision
00	06/10/2022	Initial Release	n/a


18
37
17
16
19
21
20
15
12
9
52
All remaining area
14
11
8
51
13
10
7
54
55
44
43
40
3
6
50
53
45
42
39
2
5
46
41
38
1
4
Google Earth

Biota Sampling Form
Parsons

SITE NAME: Seneca Army Depot, Romulus, NY
PROJECT NUMBER:
SAMPLE DATE
SAMPLER(S):

DESCRIPTION OF ANIMAL SAMPLED

SPECIES:
SEX:
DATE & TIME HARVESTED:
APPROXIMATE WEIGHT:
APPROXIMATE LOCATION HARVESTED:
PROJECTILE TYPE USED IN HARVEST:
ENTRY/EXIT WOUND LOCATION:

SAMPLE 1

SAMPLE ID: **SAMPLE TIME:**

MEDIA SAMPLED: MUSCLE LIVER OTHER:

QA/QC SAMPLES

DUPLICATE SAMPLE COLLECTED: YES NO

DUPLICATE SAMPLE ID: **SAMPLE TIME:**

MS/MSD SAMPLE COLLECTED YES NO

MS SAMPLE ID: **SAMPLE TIME:**

MSD SAMPLE ID: **SAMPLE TIME:**

COMMENTS:

SAMPLE 2

SAMPLE ID: **SAMPLE TIME:**

MEDIA SAMPLED: MUSCLE LIVER OTHER:

QA/QC SAMPLES

DUPLICATE SAMPLE COLLECTED: YES NO

DUPLICATE SAMPLE ID: **SAMPLE TIME:**

MS/MSD SAMPLE COLLECTED YES NO

MS SAMPLE ID: **SAMPLE TIME:**

MSD SAMPLE ID: **SAMPLE TIME:**

COMMENTS:

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ATTACHMENT 3

LABORATORY CERTIFICATION

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SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

EUROFINS LANCASTER LABORATORIES ENVIRONMENT TESTING LLC
2425 New Holland Pike
Lancaster, PA 17601
Kenneth Boley Phone: 717-556-9413

ENVIRONMENTAL

Valid To: November 30, 2024 Certificate Number: 0001.01

In recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.4 of the DoD/DOE Quality Systems Manual for Environmental Laboratories, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F^-, O_2), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Demands				
COD	----------	EPA 410.4	EPA 410.4	----------
Total Organic Carbon	----------	EPA 9060A SM 5310C-2014	EPA 9060A SM 5310C-2014	EPA 9060A SM 5310C-2014 Lloyd Kahn
Anions				
Ammonia	----------	EPA 350.1	EPA 350.1	SM 4500-NH3 B/C-2011
Fluoride	----------	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A
Nitrate (as N)	----------	EPA 300.0 EPA 353.2 EPA 9056A	EPA 300.0 EPA 353.2 EPA 9056A	EPA 300.0 EPA 353.2 EPA 9056A
Nitrite (as N)	----------	EPA 300.0 EPA 353.2 EPA 9056A	EPA 300.0 EPA 353.2 EPA 9056A	EPA 300.0 EPA 353.2 EPA 9056A

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Nitrate Nitrite Total	----------	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A
Bromide	----------	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A	----------
Chloride	----------	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A
Sulfate	----------	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A	EPA 300.0 EPA 9056A
Wet Chemistry				
Alkalinity	----------	SM 2320B-2011	SM 2320B-2011	----------
Corrosivity	----------	----------	SW-846 Chapter 7	SW-846 Chapter 7
Conductivity		SM 2510B-2011	SM 2510B-2011	----------
Cyanide	----------	EPA 9012B	EPA 9012B	EPA 9012B
Filterable Residue (TDS)	----------	SM 2540C-2015	SM 2540C-2015	----------
Flashpoint	----------	EPA 1010A/B	EPA 1010A/B	EPA 1010A/B
Grain Size	----------	----------	----------	ASTM D422 MOD
Hardness	----------	EPA 130.2 SM 2340B-2011 SM 2340C-2011	EPA 130.2 SM 2340B-2011 SM 2340C-2011	----------
Hexavalent Chromium Digestion	----------	----------	----------	EPA 3060A
Hexavalent Chromium	----------	EPA 218.6 EPA 7196A EPA 7199	EPA 7196A EPA 7199	EPA 7196A EPA 7199
Ignitability	----------	----------	40 CFR 261.21	40 CFR 261.21
Non-filterable Residue (TSS)	----------	SM 2540D-2015	SM 2540D-2015	----------
Paint Filter	----------	----------	----------	EPA 9095B
pH	----------	SM 4500 H+B-2011 EPA 9040B/C	EPA 9040B/C	EPA 9045C/D
Phenol	----------	EPA 9066	EPA 9066	----------
Reactivity Prep	----------	----------	SW-846 Chapter 7.3	SW-846 Chapter 7.3
Reactive Cyanide	----------	----------	EPA 9012B	EPA 9012B
Reactive Sulfide	----------	----------	EPA 9034	EPA 9034
Sulfide	----------	EPA 376.1 EPA 376.2 SM 4500 S2D-2011 SM 4500 S2F-2011	EPA 376.1 EPA 376.2 SM 4500 S2D-2011 SM 4500 S2F-2011	----------
Total Kjeldahl Nitrogen (TKN)	----------	EPA 351.2	EPA 351.2	EPA 351.2
Total Residue	----------	SM 2540B-2015	SM 2540B-2015	SM 2540G-2015
Metals				
Metals Digestion	----------	EPA 3005A	EPA 3005A	EPA 3050B
Aluminum	EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Antimony	EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Arsenic	EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Barium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Beryllium	EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Boron	----------	EPA 200.7 EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Cadmium	EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Calcium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Chromium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Cobalt	EPA 200.7	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Copper	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Iron	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Lead	EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Lithium	EPA 200.7	EPA 200.7 EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Molybdenum	----------	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Magnesium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Manganese	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Mercury	EPA 245.1	EPA 245.1 EPA 7470A	EPA 245.1 EPA 7470A	EPA 7471A EPA 7471B
Nickel	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Potassium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Selenium	EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Silicon	----------	EPA 200.7 EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Silver	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Sodium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Strontium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Sulfur	EPA 200.7	EPA 200.7 EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Thallium	EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Thorium	----------	EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Tin	EPA 200.7	EPA 200.7 EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Titanium	----------	EPA 200.7 EPA 200.8 EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Tungsten	----------	EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Uranium	----------	EPA 200.8 EPA 6020B	EPA 6020B	EPA 6020B
Vanadium	EPA 200.7	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Zinc	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B	EPA 6010C/D EPA 6020B
Zirconium	----------	EPA 6010C/D	EPA 6010C/D	EPA 6010C/D
Purgeable Organics (Volatiles)				
Volatile Preparation	----------	EPA 5030C	EPA 5030C	EPA 5035A
Acetone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acetonitrile	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acrolein	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acrylonitrile	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Allyl chloride	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Amyl Alcohol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Amyl Methyl Ether	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butyl Alcohol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butyl Formate	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Benzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromochloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromodichloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromoform	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromomethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Butanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
n-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
sec-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Carbon disulfide	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Carbon tetrachloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chloro-1,3-butadiene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Chloroacetonitrile	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1-Chlorobutane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chlorodifluoromethane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chloroethyl Vinyl Ether	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloroform	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1-Chlorohexane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chlorotoluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
4-Chlorotoluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Cyclohexane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Cyclohexanone	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Di-Isopropyl ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dibromochloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dibromo-3-chloropropane	EPA 524.2	EPA 8260C/D EPA 8011	EPA 8260C/D EPA 8011	EPA 8260C/D
Dibromomethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dibromoethane (EDB)	----------	EPA 8260C/D EPA 8011	EPA 8260C/D EPA 8011	EPA 8260C/D
1,2-Dichlorobenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3-Dichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Dichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,4-dichloro-2-butene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dichlorodi-fluoromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
cis-1,2-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,2-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dichlorofluoromethane	EPA 524.2	----------	----------	----------
1,2-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2,2-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloropropene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
cis-1,3-Dichloropropene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,3-Dichloropropene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Dioxane	----------	EPA 8260C/D EPA 8260C/D SIM	EPA 8260C/D EPA 8260C/D SIM	EPA 8260C/D EPA 8260C/D SIM
Ethanol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethyl ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Ethyl Methacrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethyl Tert-Butyl Ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Freon-113	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Gasoline Range Organics (GRO) [Volatile Petroleum Hydrocarbons (VPH)]	----------	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101
Heptane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Hexane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Hexanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Hexachlorobutadiene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Hexachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Isopropyl Alcohol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Isopropylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Isopropyltoluene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylacrylonitrile	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Acetate	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Acrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Iodide	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylene Chloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Methacrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Tert-Butyl Ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
4-Methyl-2-pentanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylcyclohexane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Nitropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Naphthalene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Pentachloroethane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Propionitrile	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
n-Propylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Styrene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tert-Amyl Ethyl Ether	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,1,2-Tetrachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,2,2-Tetrachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tetrachloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tetrahydrofuran	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Toluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,3-Trichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,4-Trichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,1-Trichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,2-Trichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Trichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Trichlorofluoromethane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,3-Trichloropropane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,4-Trimethylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3,5-Trimethylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
130BVinyl Acetate	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Vinyl Chloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Xylenes, Total	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Xylene (o-Xylene)	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3+1,4-Xylene (m+p Xylene)	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Extractable Organics (Semivolatiles)				
Acenaphthene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Acenaphthylene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Acetophenone	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Acetylaminofluorene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Alkylated PAHs	----------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
4-Aminobiphenyl	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Amino-4,6-dinitrotoluene	----------	EPA 8330B	EPA 8330B	----------
4-Amino-2,6-dinitrotoluene	----------	EPA 8330B	EPA 8330B	----------
Aniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Anthracene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Atrazine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzaldehyde	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzoic acid	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzo (a) anthracene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (b) fluoranthene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (k) fluoranthene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (ghi) perylene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (a) pyrene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (e) pyrene	----------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
Benzyl Alcohol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Biphenyl	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
bis (2-Chloroethoxy) Methane	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
bis (2-Chloroethyl) Ether	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
bis (2-Ethylhexyl) Phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
4-Bromophenylphenyl Ether	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Butyl benzyl Phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Caprolactam	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Carbazole	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Carbon Range Organics C8-C44 (including subsets of this range i.e. HRO, MRO, ORO, RRO)	----------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
4-Chloroaniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Chloro-3-methylphenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Chlorobenzilate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Chloronaphthalene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Chloronaphthalene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Chlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Chlorophenyl phenyl ether	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Chrysene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Cresols (Methyl phenols)	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
cis-/trans-Diallate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Diamino-6-nitrotoluene	----------	EPA 8330B	EPA 8330B	----------
2,6-Diamino-4-nitrotoluene	----------	EPA 8330B	EPA 8330B	----------
Dibenzo (a,h) acridine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Dibenzo (a,h) anthracene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Dibenzofuran	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
1,2-Dichlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,3-Dichlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,4-Dichlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3,3-Dichlorobenzidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diesel Range Organics (DRO) [Extractable Petroleum Hydrocarbons (EPH)]	----------	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005 AK102/103 AK102/103-SV	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005 AK102/103 AK102/103-SV	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005 AK102/103

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
2,4-Dichlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,6-Dichlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diethyl Phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Dimethoate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
p-Dimethylaminoazobenze	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
7,12-Dimethylbenz (a) anthracene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dimethylphenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Dimethyl Phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
3,3'-Dimethylbenzidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Di-n-butyl Phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Di-n-octyl phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
3,5-Dinitroaniline	----------	EPA 8330B	EPA 8330B	----------
1,3-Dinitrobenzene	----------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E
1,4-Dinitrobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dinitrophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dinitrotoluene	----------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E
2,6-Dinitrotoluene	----------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E
1,4-Dioxane	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Diphenylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diphenyl ether	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2-Diphenylhydrazine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Ethyl Methanesulfonate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Fluoroanthene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Fluorene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Hexachlorobenzene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Hexachlorobutadiene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachlorocyclo-pentadiene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachloroethane	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachloropropene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	----------	EPA 8330B	EPA 8330B	----------

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Indeno (1,2,3-cd) Pyrene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Isodrin	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Isophorone	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Isosafrole	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3-Methycholanthrene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Methyl-4,6-dinitrophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Methyl methane sulfonate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Methylnaphthalene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
2-Methylnaphthalene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
2-Methylphenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Methylphenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Naphthalene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
1,4-Naphthoquinone	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Naphthylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Naphthylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitroquinoline-1-oxide	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Nitroaniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3-Nitroaniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitroaniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Nitrobenzene	----------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E
Nitroglycerin	----------	EPA 8330B	EPA 8330B	----------
2-Nitrophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitrophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Nitrotoluene	----------	EPA 8330B	EPA 8330B	----------
3-Nitrotoluene	----------	EPA 8330B	EPA 8330B	----------
4-Nitrotoluene	----------	EPA 8330B	EPA 8330B	----------
5-Nitro-o-toluidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitroso-di-n-butylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodiethylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodimethylamine	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
n-Nitrosomethylethylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosomorpholine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodi-n-propylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodiphenylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosopiperidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosopyrrolidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	----------	EPA 8330B	EPA 8330B	EPA 8330B MOD
2,2-Oxybis (1-chloropropane)	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachloronitrobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentaerythritol Tetranitrate (PETN)	----------	EPA 8330B	EPA 8330B	----------
Perylene	----------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
Phenacetin	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Phenanthrene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Phenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Picoline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pronamide	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pyrene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Pyridine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Safrole	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2,4,5- Tetrachlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,3,4,6-Tetrachlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetraethyl dithiopyrophosphate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetraethy lead	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetryl	----------	EPA 8330B	EPA 8330B	----------
Thionazin	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
o-Toluidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2,4-Trichlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,3,5-Trinitrobenzene	----------	EPA 8330B	EPA 8330B	----------
2,4,5-Trichlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4,6-Trichlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
O,O,O-Tri-ethylphosphorothioate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4,6-Trinitrotoluene	----------	EPA 8330B	EPA 8330B	----------
Organochlorine Pesticides				
Aldrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
alpha-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
beta-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
delta-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
gamma-BHC (Lindane)	----------	EPA 8081B	EPA 8081B	EPA 8081B
alpha-Chlordane	----------	EPA 8081B	EPA 8081B	EPA 8081B
Chlordane (Technical)	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDD	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDE	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDT	----------	EPA 8081B	EPA 8081B	EPA 8081B
4,4'-DDD	----------	EPA 8081B	EPA 8081B	EPA 8081B

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
4,4'-DDE	----------	EPA 8081B	EPA 8081B	EPA 8081B
4,4'-DDT	----------	EPA 8081B	EPA 8081B	EPA 8081B
Dieldrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
Dinoseb	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Endosulfan I (alpha)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endosulfan II (beta)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endosulfan Sulfate	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin Aldehyde	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin Ketone	----------	EPA 8081B	EPA 8081B	EPA 8081B
gamma-Chlordane	----------	EPA 8081B	EPA 8081B	EPA 8081B
Heptachlor	----------	EPA 8081B	EPA 8081B	EPA 8081B
Heptachlor Epoxide	----------	EPA 8081B	EPA 8081B	EPA 8081B
Hexachlorobenzene	----------	EPA 8081B	EPA 8081B	EPA 8081B
Hexachlorocyclopentadiene	----------	EPA 8081B	EPA 8081B	EPA 8081B
Methoxychlor	----------	EPA 8081B	EPA 8081B	EPA 8081B
Mirex	----------	EPA 8081B	EPA 8081B	EPA 8081B
Toxaphene	----------	EPA 8081B	EPA 8081B	EPA 8081B
PCBs (Aroclors)				
PCB-1016 (Arochlor)	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1221	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1232	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1242	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1248	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1254	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1260	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1262	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1268	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB congeners (209)	----------	EPA 1668A EPA 1668C	EPA 1668A EPA 1668C	EPA 1668A EPA 1668C
Herbicides				
2,4,5-T	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4,5-TP (Silvex)	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4-D	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4-DB	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dalapon	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dicamba	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dichlorprop	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dinoseb	----------	EPA 8151A	EPA 8151A	EPA 8151A
MCPA	----------	EPA 8151A	EPA 8151A	EPA 8151A
MCPP	----------	EPA 8151A	EPA 8151A	EPA 8151A
Pentachlorophenol	----------	EPA 8151A	EPA 8151A	EPA 8151A
PCB Homologues				
Monochlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Dichlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Trichlorobiphenyls	----------	EPA 680	EPA 680	EPA 680

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Tetrachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Pentachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Hexachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Heptachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Octachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Nonachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Decachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Dioxins/Furans				
2,3,7,8-TCDD	EPA 1613B	EPA 8290A	EPA 8290A	EPA 8290A
2,3,7,8-TCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8-PeCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
2,3,4,7,8-PeCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8-PeCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8-HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,6,7,8-HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
2,3,4,6,7,8-HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8,9-HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8,-HxCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,6,7,8-HxCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8,9-HxCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,6,7,8-HpCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8,9-HpCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,6,7,8-HpCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
OCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
OCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total HpCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total HpCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total HxCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total PeCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total PeCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total TCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total TCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
Misc. Headspace Analysis				
Carbon dioxide	----------	RSK-175	RSK-175	----------
Ethane	----------	RSK-175	RSK-175	----------
Ethene	----------	RSK-175	RSK-175	----------
Methane	----------	RSK-175	RSK-175	----------
Acetylene	----------	RSK-175	RSK-175	----------
Propane	----------	RSK-175	RSK-175	----------
Hazardous Waste Characteristics				
342BToxicity Characteristic Leaching Procedure	----------	----------	EPA 1311	EPA 1311
343BSynthetic Precipitation Leaching Procedure	----------	----------	EPA 1312	EPA 1312

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
344BASTM Leaching Procedure	----------	----------	ASTM D3987-85	ASTM D3987-85
Other				
Perchlorate	----------	EPA 6850	EPA 6850	EPA 6850
Hydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Formaldehyde	----------	----------	EPA 8315A	EPA 8315A
Methylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
1,1-Dimethylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Acetic Acid	----------	EPA 8015D	EPA 8015D	----------
Butryic acid	----------	EPA 8015D	EPA 8015D	----------
Lactic Acid	----------	EPA 8015D	EPA 8015D	----------
Propionic Acid	----------	EPA 8015D	EPA 8015D	----------
Pyruvic Acid	----------	EPA 8015D	EPA 8015D	----------
Citric Acid	----------	EPA 8015D	EPA 8015D	----------
Formic Acid	----------	EPA 8015D	EPA 8015D	----------
Oxalic Acid	----------	EPA 8015D	EPA 8015D	----------
Quinic Acid	----------	EPA 8015D	EPA 8015D	----------
Succinic Acid	----------	EPA 8015D	EPA 8015D	----------
Tartaric Acid	----------	EPA 8015D	EPA 8015D	----------
Volatile Preparation	----------	EPA 5030C	EPA 5030C	EPA 5035 EPA 5035A
352BOrganic Extraction/Cleanup	----------	EPA 3510C EPA 3511 EPA 3660B, 3620C, 3665A	EPA 3510C EPA 3511 EPA 3660B, 3620C, 3665A	EPA 3546 EPA 3550C EPA 3660B, 3620C, 3665A, 3640A

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
Per and Polyfluoroalkyl Substances (PFAS)			
N-ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
N-methyl perfluorooctanesulfonamidoacetic Acid (NMeFOSAA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorobutanesulfonic Acid (PFBS)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorodecanoic Acid (PFDA)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
		EPA Draft Method 1633	EPA Draft Method 1633
Perfluorododecanoic Acid (PFDoA)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluoroheptanoic Acid (PFHpA)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorohexanesulfonic Acid (PFHxS)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorohexanoic Acid (PFHxA)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorononanoic Acid (PFNA)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorooctanesulfonic Acid (PFOS)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorooctanoic Acid (PFOA)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorotetradecanoic Acid (PFTeDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorotridecanoic Acid (PFTrDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluoroundecanoic Acid (PFUnA)	EPA 537 EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
		EPA Draft Method 1633	EPA Draft Method 1633
Hexafluoropropylene oxide dimer acid (HF-PODA)	EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
4,8-Dioxa-3*H*-perfluorononanoic acid (ADONA)	EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	EPA 537.1 EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorobutanoic Acid (PFBA)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluoropentanoic Acid (PFPeA)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2-FTS)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluoropentanesulfonic Acid (PFPeS)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2-FTS)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluoroheptanesulfonic Acid (PFHpS)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorononanesulfonic Acid (PFNS)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
374BPerfluorodecanesulfonic Acid (PFDS)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
10:2 Fluorotelomersulfonic Acid (10:2-FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15
Perfluorododecanesulfonic Acid (PFDoS)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluorohexadecanoic Acid (PFHxDA)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15
Perfluorooctadecanoic Acid (PFODA)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15
Perfluorooctanesulfonamide (PFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
N-methyl perfluorooctanesulfonamidoethanol (NMeFOSE)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
N-methyl perfluorooctanesulfonamide (NMeFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
		EPA Draft Method 1633	EPA Draft Method 1633
N-ethyl perfluorooctanesulfonamidoethanol (NEtFOSE)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
N-ethylperfluorooctanesulfonamide (NEtFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluoro-3-methoxypropanoic acid (PFMPA)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluoro-4-methoxybutanoic acid (PFMBA)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	EPA 533	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
3-Perfluoropropylpropanoic acid (3:3 FTCA)	---	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
2*H*,2*H*,3*H*,3*H*-Perfluorooctanoic acid (5:3 FTCA)	---	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633
3-Perfluoroheptylpropanoic acid (7:3 FTCA)	---	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633	PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633

End of DoD ELAP section of scope

In addition, in recognition of the successful completion of the A2LA evaluation process (including an assessment of the

laboratory's compliance with ISO IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, and for the test methods applicable to Kentucky Statute KRS 224.60-130(2)(a), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F^-, O_2), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid
Other				
Perchlorate	Food & Food Products EPA 6850	EPA 6850	EPA 6850	EPA 6850
Hydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Methylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
1,1-Dimethylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Volatile Preparation	----------	EPA 5030A EPA 5030C	EPA 5030A EPA 5030C	EPA 5035 EPA 5035A
Organic Extraction/ Cleanup	EPA 3546 EPA 3550C EPA 3660B EPA 3620C EPA 3665A EPA 3640A	EPA 3510C EPA 3511 EPA 3660B EPA 3620C EPA 3665A	EPA 3510C EPA 3511 EPA 3660B EPA 3620C EPA 3665A	EPA 3546 EPA 3550C EPA 3660B EPA 3620C EPA 3665A EPA 3640A

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid
Kentucky UST Program				
Metals				
Arsenic	----------	----------	EPA 6010B	EPA 6010B
Barium	----------	----------	EPA 6010B	EPA 6010B
Cadmium	----------	----------	EPA 6010B	EPA 6010B
Chromium	----------	----------	EPA 6010B	EPA 6010B
Lead	----------	----------	EPA 6010B	EPA 6010B
Mercury	----------	----------	EPA 7470A	EPA 7471A
Selenium	----------	----------	EPA 6010B	EPA 6010B
Silver	----------	----------	EPA 6010B	EPA 6010B
Purgeable Organics (Volatiles)				

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid
Diesel Range Organics (DRO)	----------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
Gasoline Range Organics (GRO)	----------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
Wyoming Storage Tank Program				
Metals				
Cadmium	----------	----------	EPA 6010C	EPA 6010C
Chromium	----------	----------	EPA 6010C	EPA 6010C
Chromium (Total, hexavalent)	----------	----------	EPA 7196A	EPA 7196A
Lead	----------	----------	EPA 6010C	EPA 6010C
Purgeable Organics (Volatiles)				
Volatile Preparation	----------	----------	EPA 5030C EPA 5030C	EPA 5035 EPA 5035A
Benzene	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
1,2-Dichloroethane	----------	----------	EPA 8260D	EPA 8260D
1,2-Dibromoethane	----------	----------	EPA 8011	EPA 8011
Diisopropyl Ether	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Ethyl Benzene	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Ethyl tert-butyl Ether	----------	----------	EPA 8260D	EPA 8260D
Methyl tert-butyl Ether	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Naphthalene	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Toluene	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Tert-amyl Methyl Ether	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Tert-butyl Alcohol	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Xylenes, total	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Gasoline Range Organics (GRO C6-C10)	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Extractable Organics (Semivolatiles)				
Diesel Range Organics (DRO C10-C32)	----------	----------	EPA 8015C w/ EPA 3630 cleanup	EPA 8015C w/ EPA 3630 cleanup

End of KY, WY, and ISO 17025 section of scope

In recognition of the successful completion of the A2LA evaluation process, including an assessment of the laboratory's compliance with ISO/IEC 17025:2017 accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and, in the analyte, categories identified below:

Food and Feed (WHO 29)	**Food/Feed**
2,3,7,8-TCDD	EPA 1613B
2,3,7,8-TCDF	EPA 1613B
1,2,3,7,8-PeCDF	EPA 1613B
2,3,4,7,8-PeCDF	EPA 1613B
1,2,3,7,8-PeCDD	EPA 1613B
1,2,3,4,7,8-HxCDF	EPA 1613B
1,2,3,6,7,8-HxCDF	EPA 1613B
2,3,4,6,7,8-HxCDF	EPA 1613B
1,2,3,7,8,9-HxCDF	EPA 1613B
1,2,3,4,7,8-HxCDD	EPA 1613B
1,2,3,6,7,8-HxCDD	EPA 1613B
1,2,3,7,8,9-HxCDD	EPA 1613B
1,2,3,4,6,7,8-HpCDF	EPA 1613B
1,2,3,4,7,8,9-HpCDF	EPA 1613B
1,2,3,4,6,7,8-HpCDD	EPA 1613B
OCDF	EPA 1613B
OCDD	EPA 1613B
Food and Feed (WHO 29)	**Food/Feed**
6 marker PCBs (PCB28, PCB52, PCB101, PCB138, PCB153, and PCB180)	EPA 1668C
(PCB77, PCB81, PCB105, PCB114, PCB118, PCB123, PCB126, PCB156, PCB157, PCB167, PCB169, and PCB189)	EPA 1668C

Parameter/Analyte	**Tissue**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
12 Dioxin-like PCBs (dl-PCBs)/coplanar PCBs (PCB77, PCB81, PCB105, PCB114, PCB118, PCB123, PCB126, PCB156, PCB157, PCB167, PCB169, & PCB189)	EPA 1668C	----------	----------	----------

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
441B**Per and Polyfluoroalkyl Substances (PFAS)**			
442BN-ethyl perfluorooctane-sulfonamidoacetic acid (NetFOSAA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
443BN-methyl perfluorooctane-sulfonamidoacetic acid (NMeFOSAA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
444BPerfluorobutanesulfonic acid (PFBS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
445BPerfluorodecanoic acid (PFDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
446BPerfluorododecanoic acid (PFDoDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
447BPerfluoroheptanoic acid (PFHpA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
448BPerfluorohexanesulfonic acid (PFHxS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
449BPerfluorohexanoic acid (PFHxA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
450BPerfluorononanoic acid (PFNA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
451BPerfluorooctanesulfonic acid (PFOS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
452BPerfluorooctanoic acid (PFOA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
453BPerfluorotetradecanoic acid (PFTeDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
454BPerfluorotridecanoic acid (PFTrDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
455BPerfluoroundecanoic acid (PFUnDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid (HFPODA)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
456BPerfluoro-n-butanoic acid (PFBA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
457BPerfluoro-n-pentanoic acid (PFPeA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
458B8:2 Fluorotelomersulfonic acid (8:2FTS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
459B4:2 Fluorotelomersulfonic acid (4:2-FTS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
460BPerfluoropentanesulfonic acid (PFPeS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
461B6:2 Fluorotelomersulfonic acid (6:2-FTS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoroheptanesulfonic acid (PFHpS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
462BPerfluorononanesulfonic acid (PFNS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
463BPerfluorodecanesulfonic acid (PFDS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
464B10:2 Fluorotelomersulfonic acid (10:2-FTS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
465BPerfluorododecanesulfonic acid (PFDoDS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
466BPerfluorohexadecanoic acid (PFHxDA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
467BPerfluorooctadecanoic acid (PFODA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
468BPerfluorooctanesulfonamide (PFOSA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
469B2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMePFOSAE)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
470BN-methylperfluoro-1-octanesulfonamide (NMePFOSA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
471B2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtPFOSAE)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
472BN-ethylperfluoro-1-octanesulfonamide (NEtPFOSA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod

Accredited Laboratory

A2LA has accredited

EUROFINS LANCASTER LABORATORIES ENVIRONMENTAL, LLC

Lancaster, PA

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.4 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated *April 2017*).

Presented this 21st day of November 2022.

Mr. Trace McInturff, Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 1.01
Valid to November 30, 2024

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

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ATTACHMENT 4

DATA VALIDATION STANDARD OPERATING PROCEDURE

LDC: SOP 4.96 DOD.0 Data Qualification for Perfluoroalkyl and Polyfluoroalkyl Substances Using DoD QSM 5.3 Table B-15

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SOP #4.96DOD.0

DATA QUALIFICATION FOR PERFLUOROALKYL AND POLYFLUOROALKYL SUBSTANCES USING DOD QSM 5.3 TABLE B-15

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
Aqueous Sample Preparation	Each sample and associated batch QC samples.	Solid Phase Extraction (SPE) must be used unless samples are known to contain high PFAS concentrations (e.g., Aqueous Film Forming Foam (AFFF) formulations). Inline SPE is acceptable. Entire sample plus bottle rinsate must be extracted using SPE. Known high PFAS concentration samples require serial dilution be performed in duplicate. Documented project approval is needed for samples prepared by serial dilution as opposed to SPE.	NA.	NA.	Samples with > 1% solids may require centrifugation prior to SPE extraction. Pre-screening of separate aliquots of aqueous samples is recommended.	Qualify associated detect results as estimated (J) and non-detect results as X, exclusion of data recommended, if SPE was not performed or the entire sample plus bottle rinsate was not extracted. Using professional judgment, no data qualification is required when SPE is not performed, if project was approved for serial dilution as opposed to SPE. Qualify associated detect results as estimated (J) and non-detect results as X, exclusion of data recommended, if serial dilution is not performed in duplicate. Reviewer may need to verify compliance using the laboratory SOP.
Solid Sample Preparation	Each sample and associated batch QC samples.	Entire sample received by the laboratory must be homogenized prior to subsampling.	NA.	NA.	NA.	Qualify associated detect results as estimated (J) and non-detect results as X, exclusion of data recommended, if entire sample was

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
						not homogenized prior to subsampling. Reviewer may need to verify compliance using the laboratory SOP.
Biota Sample Preparation	Each sample and associated batch QC samples.	Sample prepared as defined by the project (e.g., whole fish versus filleted fish).	NA.	NA.	NA.	Qualify associated detect results as estimated (J) and non-detect results as X, exclusion of data recommended, if biota samples were prepared incorrectly. Reviewer may need to verify compliance using the laboratory SOP.
AFFF and AFFF Mixture Samples Preparation	Each sample and associated batch QC samples.	Each field sample must be prepared in duplicate (equivalent to matrix duplicate). Serial dilutions must be performed to achieve the lowest LOQ possible for each analyte.	NA.	NA.	Adsorption onto bottle is negligible compared to sample concentration so subsampling is allowed. Multiple dilutions will most likely have to be reported in order to achieve the lowest LOQ possible for each analyte.	Qualify associated detect results as estimated (J) and non-detect results as X, exclusion of data recommended, if samples were not prepared in duplicate or serial dilution was not performed. Reviewer may need to verify compliance using the laboratory SOP.
Sample Cleanup Procedure	Each sample and associated batch QC samples. Not applicable to AFFF and AFFF Mixture Samples.	ENVI-Carb™ or equivalent must be used on each sample and batch QC sample.	NA.	Flagging is not appropriate.	Cleanup should reduce bias from matrix interferences.	Qualify associated detect results as estimated (J), if clean-up was not performed.

DATA QUALIFICATION FOR PERFLUOROALKYL AND POLYFLUOROALKYL SUBSTANCES USING DOD QSM 5.3 TABLE B-15

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
Mass Calibration	Instrument must have a valid mass calibration prior to any sample analysis. Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL).	Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ±0.5 amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard.	If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance.	Flagging is not appropriate.	Problem must be corrected. No samples may be analyzed under a failing mass calibration. The mass calibration is updated on an as-needed basis (e.g., QC failures, ion masses fall outside of the ±0.5 amu of the true value, major instrument maintenance is performed, or the instrument is moved).	Qualify all associated results as X, exclusion of data recommended, if mass calibration was not performed at the required frequency or does not comply with acceptance criteria.
Mass Spectral Acquisition Rate	Each analyte, Extracted Internal Standard (EIS) Analyte.	A minimum of 10 spectra scans are acquired across each chromatographic peak.	NA.	Flagging is not appropriate.	NA.	Qualify all associated results as X, exclusion of data recommended, if less than 10 spectra scans are acquired.
Calibration, Calibration Verification, and Spiking Standards	All analytes.	Standards containing both branched and linear isomers must be used when commercially available. PFAS method analytes may consist of both branched and linear isomers, but quantitative standards that contain the linear and branched isomers do not exist for all method analytes. For PFAS that do not have a quantitative branched and	NA.	Flagging is not appropriate.	Standards containing both branched and linear isomers are to be used during method validation and when reestablishing retention times, to ensure the total response is quantitated for that analyte. Technical grade standards cannot be used for quantitative analysis.	Using professional judgment, for PFAS that may consist of both branched and linear isomers, qualify associated detect and non-detect results as estimated (J/UJ), if quantitative standards contained linear isomers only. For PFAS that may consist of both branched and linear

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
		linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative standard.				isomers, qualify associated detect and non-detect results as estimated (J/UJ), if the total response was quantitated using linear isomers only.
Sample PFAS Identification	All analytes detected in a sample.	The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFBA and PFPeA). Documentation of the primary and confirmation transitions and the ion ratio is required. In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50- 150%. Signal to Noise Ratio (S/N) must be ≥ 10 for all ions used for quantification and must be ≥ 3 for all ions used for confirmation.	NA.	PFAS identified with Ion ratios that fail acceptance criteria must be flagged. Any quantitation ion peak that does not meet the maximization criteria shall be included in the summed integration and the resulting data flagged as “estimated, biased high”.	For example: Ion Ratio = (quant ion abundance/ confirm ion abundance) Calculate the average ratio (A) and standard deviation (SD) using the ICAL standards. An acceptance range of ratio could be within A ±3SD for confirmation of detection.	Qualify all associated results as X, exclusion of data recommended, if documentation of the primary and confirmation transitions or ion ratio cannot be provided. Qualify all associated results as X, exclusion of data recommended, if ion ratio is <50% or >150%. S/N ratio is <10 for the quant ion, S/N ratio is <3 for the confirmation ion and the quant ion and confirmation ion are not present or do not maximize simultaneously.

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
		Quant ion and confirmation ion must be present and must maximize simultaneously (±2 seconds).				Use professional judgment to qualify associated detects if the ion ratio criteria were not met but the S/N ratio and the quantitation ion/confirmation ion maximized simultaneously.
Ion Transitions (Precursor-> Product)	Every field sample, standard, blank, and QC sample.	In order to avoid biasing results high due to known interferences for some transitions, the following transitions must be used for the quantification of the following analytes: PFOA: 413 → 369 PFOS: 499 → 80 PFHxS: 399 → 80 PFBS: 299 → 80 4:2 FTS: 327 → 307 6:2 FTS: 427 → 407 8:2 FTS: 527 → 507 NEtFOSAA: 584 → 419 NMeFOSAA: 570 → 419 If these transitions are not used, the reason must be technically justified and documented (e.g., alternate transition was used due to observed interferences).	NA.	Flagging is not appropriate	NA.	Use professional judgment to qualify associated detects when the listed transitions are not used. The laboratory may indicate that alternate transitions were used due to interferences with listed transitions.
Initial Calibration (ICAL)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	The isotopically labeled analog of an analyte (Extracted Internal Standard Analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation).	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ICAL has passed. External Calibration is not allowed for any analyte. Calibration can be linear (minimum of 5 standards)	Qualify all associated results as X, exclusion of data recommended, if an acceptable initial calibration was not performed prior to sample

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
		Commercial PFAS standards available as salts are acceptable providing the measured mass is corrected to the neutral acid concentration. Results shall be reported as the neutral acid with appropriate CAS number. If a labeled analog is not commercially available, the Extracted Internal Standard Analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (Internal Standard Quantitation) Analytes must be within 70-130% of their true value for each calibration standard. ICAL must meet one of the two options below: Option 1: The RSD of the RFs for all analytes must be ≤ 20%. Option 2: Linear or non-linear calibrations must have r2 ≥ 0.99 for each analyte.			or quadratic (minimum of 6 standards); weighting is allowed.	analysis. Qualify all associated results as X, exclusion of data recommended, if results were determined using external calibration. Qualify all associated results as X, exclusion of data recommended, if PFAS salt standards were used for calibration but measured mass was not corrected to the neutral acid concentration. Qualify associated detect and non-detect results as estimated (J/UJ), if ICAL values < 70% or > 130% of true value. Qualify associated detect and non-detect results as estimated (J/UJ), if ICAL RSD > 20% or r^2 < 0.99 for each analyte.

DATA QUALIFICATION FOR PERFLUOROALKYL AND POLYFLUOROALKYL SUBSTANCES USING DOD QSM 5.3 TABLE B-15

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
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.	Calculated for each analyte and EIS.	None. See below.
Retention Time (RT) window width	Every field sample, standard, blank, and QC sample.	RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the daily calibration verification or, on days when ICAL is performed, from the midpoint standard of the ICAL. Analytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to analyte and labeled analog pairs.	Correct problem and reanalyze samples.	NA.	Calculated for each analyte and EIS.	Qualify associated detect results as presumptive and estimated (NJ), if the retention time criteria were not met.
Instrument Sensitivity Check (ISC)	Prior to analysis and at least once every 12 hours.	Analyte concentrations must be at LOQ; concentrations must be within ±30% of their true values.	Correct problem, rerun ISC. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ISC has met acceptance criteria. ISC can serve as the initial daily CCV.	Qualify associated detect and non-detect results as estimated (J/UJ), if ISC concentrations are not within ±30% of their true values or the ISC was not performed at the required frequency.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Analyte concentrations must be within ±30% of their true value.	Correct problem, rerun ICV. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified.	Qualify associated detect and non-detect results as estimated (J/UJ), if ICV concentrations are not within ±30% of their true values or if the ICV

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
						was not performed at the required frequency.
Continuing Calibration Verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence.	Concentration of analytes must range from the LOQ to the mid-level calibration concentration. Analyte concentrations must be within ±30% of their 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(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Instrument Sensitivity Check (ISC) can serve as a bracketing CCV.	Qualify associated detect and non-detect results as estimated (J/UJ), if CCV concentrations are not within ±30% of their true values or if the CCV was not performed at the required frequency.
Instrument Blanks	Immediately following the highest standard analyzed and daily prior to sample analysis.	Concentration of each analyte must be ≤ ½ the LOQ. Instrument Blank must contain EIS to enable quantitation of contamination.	If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met. If sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria (>1/2 LOQ), they must be reanalyzed.	Flagging is only appropriate in cases when the sample cannot be reanalyzed and when there is no more sample left.	No samples shall be analyzed until instrument blank has met acceptance criteria. Note: Successful analysis following the highest standard analyzed determines the highest concentration that carryover does not occur. When the highest standard analyzed is not part of the calibration curve, it cannot be used to extend out the calibration range, it is used only to document a higher concentration at which carryover still does not occur.	Qualify associated detect results as B, if the sample concentration is < 10X the instrument blank concentration.

DATA QUALIFICATION FOR PERFLUOROALKYL AND POLYFLUOROALKYL SUBSTANCES USING DOD QSM 5.3 TABLE B-15

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
Extracted Internal Standard (EIS) Analytes	Every field sample, standard, blank, and QC sample.	Added to solid sample prior to extraction. Added to aqueous samples, into the original container, prior to extraction. For aqueous samples prepared by serial dilution instead of SPE, added to final dilution of samples prior to analysis. Extracted Internal Standard Analyte recoveries must be within 50% to 150% of ICAL midpoint standard area or area measured in the initial CCV on days when an ICAL is not performed.	Correct problem. If required, re-extract and reanalyze associated field and QC samples. If recoveries are acceptable for QC samples, but not field samples, the field samples must be re-extracted and analyzed (greater dilution may be needed). Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.	Apply Q-flag and discuss in the Case Narrative only if reanalysis confirms failures in exactly the same manner.	Failing analytes shall be thoroughly documented in the Case Narrative. EIS should be 96% (or greater) purity. When the impurity consists of the unlabeled analyte, the EIS can result in a background artifact in every sample, standard and blank, if the EIS is fortified at excessive concentrations.	Qualify associated detect and non-detect results as estimated (J/UJ), if the EIS %R is either < 50% or > 150% of the ICAL mid-point standard area or the area in the initial CCV on days when ICAL not performed. Qualify associated detect results as estimated (J) and non-detect results as X, exclusion of data recommended, if EIS %R < 10%.
Method Blank (MB)	One per preparatory batch.	No analytes detected >½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, re-extract and reanalyze MB and all QC samples and field samples processed with the contaminated blank. Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	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 MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Qualify associated detect results as B, if the sample concentration is < 10X the method blank concentration.

DATA QUALIFICATION FOR PERFLUOROALKYL AND POLYFLUOROALKYL SUBSTANCES USING DOD QSM 5.3 TABLE B-15

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
Laboratory Control Sample (LCS)	One per preparatory batch.	Blank spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-extract and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	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.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Qualify associated detect and non-detect results as estimated (J/UJ), if LCS %R < DoD/DOE QSM Appendix C limits or project limits if specified (or laboratory limits if not specified). Qualify associated detect results as estimated (J), if LCS %R > DoD/DOE QSM Appendix C limits or project limits if specified (or laboratory limits if not specified). Non-detects will not be qualified. Qualify associated detect results as estimated (J) and non-detect results as X, exclusion of data recommended, if LCS %R < 10% or LCS was not analyzed.
Matrix Spike (MS)	One per preparatory batch. Not required for aqueous samples prepared by serial dilution instead of SPE.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C	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.	For matrix evaluation only. 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).	Qualify associated detect and non-detect results as estimated (J/UJ), if MS/MSD %R < DoD/DOE QSM Appendix C limits or project limits if

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
		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.				specified (or laboratory limits if not specified). Qualify associated detect results as estimated (J), if MS/MSD %R > DoD/DOE QSM Appendix C limits. Non-detect results will not be qualified. Qualify associated detect results as estimated (J) and non-detect results as X, exclusion of data recommended, if MS/MSD %R < 10%.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch. For MD: Each aqueous sample prepared by serial dilution instead of SPE.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is ≥ LOQ. The MD is a second aliquot of the field sample that has been prepared by serial dilution.	MSD recoveries should be evaluated using the same criteria as MS recoveries and associated results should be qualified using the procedures as for MS recoveries. Qualify associated detect results as estimated (J), if MS/MSD or MD RPD > 30. Non-detect results will not be qualified.

DATA QUALIFICATION FOR PERFLUOROALKYL AND POLYFLUOROALKYL SUBSTANCES USING DOD QSM 5.3 TABLE B-15

DoD QSM 5.3 Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water						
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**	**LDC Validation Action**
Post Spike Sample	Only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of < LOQ for analyte(s).	Spike all analytes reported as < LOQ into the dilution that the result for that analyte is reported from. The spike must be at the LOQ concentration to be reported for this sample as < LOQ. When analyte concentrations are calculated as < LOQ, the post spike for that analyte must recover within 70-130% of its true value.	When analyte concentrations are calculated as < LOQ, and the spike recovery does not meet the acceptance criteria, the sample, sample duplicate, and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.	Flagging is not appropriate.	When analyte concentrations are calculated as < LOQ, results may not be reported without acceptable post spike recoveries.	Qualify all associated results as estimated (J) and non-detect results as X, exclusion of data recommended, if an aqueous sample was prepared by serial dilution (vs SPE) and results are <LOQ but a post spike sample evaluation was not performed. Qualify associated detect and non-detect results as estimated (J/UJ), if Post Spike %R < 70%. Qualify associated detect results as estimated (J), if Post Spike %R > 130%. Non-detects will not be qualified.

LDC #:_____ **VALIDATION FINDINGS CHECKLIST** Page:___of___
Reviewer:_____
2nd Reviewer:_____

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?				
Was cooler temperature criteria met?				
II. LC/MS Instrument performance check				
Were the instrument performance reviewed and found to be within the validation criteria?				
III. Initial calibration and Initial Calibration Verification				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Were all percent relative standard deviations (%RSD) ≤ 20%?				
Was a curve fit used for evaluation? If yes, did the initial calibration meet the coefficient of determination (r^2) criteria of > 0.990?				
Were all analytes within 70-130% or percent differences (%D) ≤30% of their true value for each calibration standard?				
Was the signal to noise (S/N) ratio for all compounds within the validation criteria?				
Were the retention time windows properly established?				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?				
Were all percent differences (%D) of the initial calibration verification ≤ 30%?				
IV. Continuing calibration and Instrument Sensitivity Check				
Was a continuing calibration analyzed prior to sample analysis, after every 10 samples and at the end of the analytical sequence?				
Were all percent differences (%D) of the continuing calibration ≤ 30%?				
Were all the retention times within the acceptance windows?				
Was the signal to noise (S/N) ratio for all compounds within the validation criteria?				
Were all percent differences (%D) of the Instrument Sensitivity Check ≤ 30%?				
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?				
Was a laboratory blank analyzed for each matrix and concentration?				
Was there contamination in the laboratory blanks?				
VI. Field blanks				
Were field blanks identified in this SDG?				
Were target compounds detected in the field blanks?				
VII. Matrix spike/Matrix spike duplicates				
Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				

LDC #:_____ **VALIDATION FINDINGS CHECKLIST** Page:___of___

Reviewer:_____

2nd Reviewer:_____

Validation Area	Yes	No	NA	Findings/Comments
VIII. Laboratory control samples				
Was an LCS analyzed per extraction batch for this SDG?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Field duplicates				
Were field duplicate pairs identified in this SDG?				
Were target compounds detected in the field duplicates?				
X. Labeled compounds				
Were labeled compound percent recoveries (%R) within the QC limits?				
Were retention times within 0.4 minutes of the associated calibration standard?				
XI. Compound quantitation				
Did the laboratory reporting limits (i.e. DL, LOD, LOQ) meet the QAPP?				
Did reported results include both branched and linear isomers?				
Were the correct ion transition, labeled compound and relative response factor (RRF) used to quantitate the compound?				
Were compound retention times within 0.1 minutes of the associated labeled compound for compounds with a labeled analog?				
Were compound quantitation and reporting limits adjusted to reflect all sample dilutions and dry weight factors applicable to Stage 4 validation?				
XII. Target compound identification				
Was the signal to noise (S/N) ratio for all compounds within the validation criteria?				
Were two transitions and the ion transition ratio per analyte monitored and documented with the exception of PFBA and PFPeA?				
Were ion ratios between 50-150%?				
XIII. System performance				
System performance was found to be acceptable.				
XIV. Overall assessment of Data				
Overall assessment of data was found to be acceptable.				

ATTACHMENT 5

RI AND SI PROPOSED SAMPLE LOCATION FIGURES

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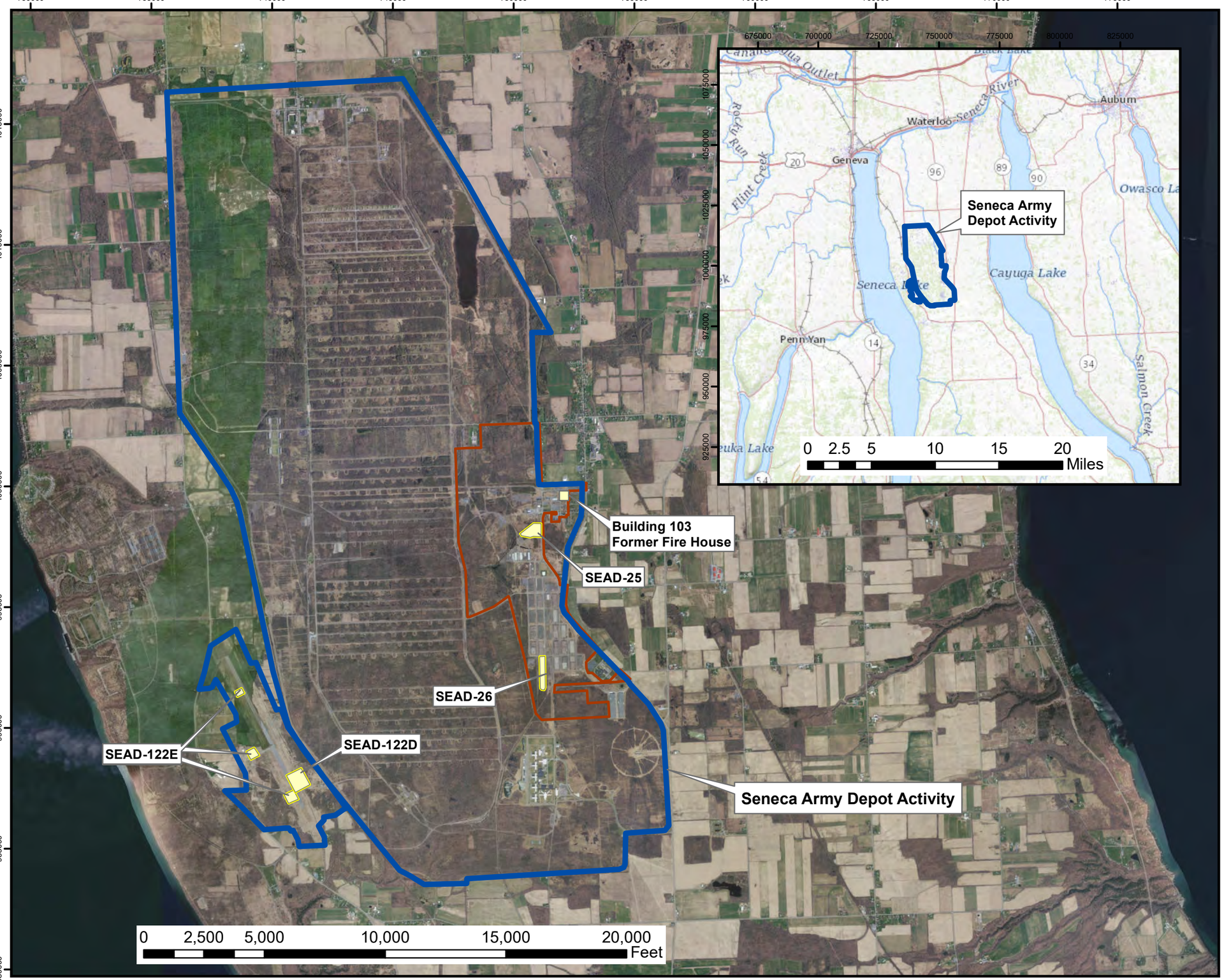


Legend

- Depot Boundary
- RI Areas of Concern
- Approximate PID Area Boundary where GW use is prohibited

Note:
Land use controls in place prohibit the use of groundwater within the Planned Industrial / Office Development (PID) and Warehousing Area and within the Airfield parcel which envelopes SEAD-122D and SEAD-122E.

DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 1:
RI SITE LOCATIONS

Scale As Shown	May 2022

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: O:\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_Site_Location.mxd

Legend

ESI Samples (Red symbol if exceedance)

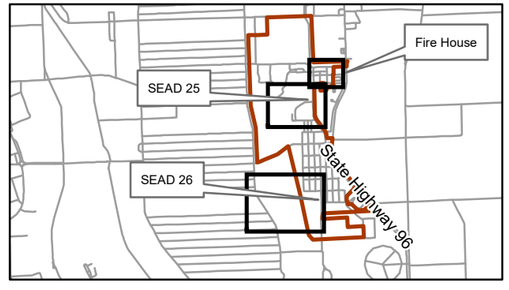
- Existing Till / Weathered Bedrock MW
- Existing Shallow Bedrock MW

Proposed RI Locations

- Proposed Till / Weathered Bedrock Well
- Proposed Shallow Bedrock Well
- ESI Stormwater Sample Location
- SEAD Boundary
- Groundwater Flow Direction
- Stormwater Drainage
- Surface Water Flow Direction
- Road
- Approximate PID Area Boundary where GW use is prohibited
- Former Fire House

Notes:

1) Symbol red if PFOA or PFOS >= 10 ng/L during ESI.
2) All well locations tentative based on accessibility and utility locations. Final determination to be made in the field.
3) Drainage pathway locations approximate. Storm drainage flow direction is to the south and southwest.
4)Existing wells with labels will be sampled once during the RI. Newly installed wells will be sampled twice.
ng/L = nanograms per liter



DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 2: FIREHOUSE – PROPOSED GROUNDWATER SAMPLING LOCATIONS

1 inch = 150 feet	January 2023

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_FH_GWSW_v2a.mxd

751400 751600 751800 752000 752200 752400

999800 999600 999400 999200 999000

East Patrol Road
East Patrol Road
East Patrol Road
Warehouse Street
Center Street
Quarters Drive
1st Avenue
2nd Avenue
3rd Avenue
State Highway 96

SBFH-01 SBFH-02 SBFH-03 SBFH-04 SBFH-05 SBFH-06 SBFH-07 SBFH-08 SBFH-09 SBFH-10 SBFH-11 SBFH-12 SBFH-13 SBFH-14 SBFH-15 SBFH-16 SBFH-17 SBFH-18 SBFH-19 SBFH-20 SBFH-21 SBFH-22 SBFH-23 SBFH-24 SBFH-25

MWFH-01 MWFH-02 MWFH-03 MWFH-04 MWFH-05 MWFH-09D MWFH-10D MWFH-12 MWFH-13 MWFH-14 MWFH-15 MWFH-15D MWFH-16 MWFH-16D

SEAD-42 SEAD-39 SEAD-33 SEAD-30

Former Fire House

0 65 130 260 390 520 Feet

Legend

ESI Samples (Red symbol if exceedance)

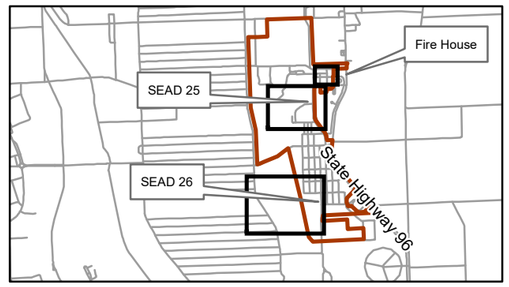
- Existing Till / Weathered Bedrock MW
- Existing Shallow Bedrock MW
- Proposed Surface Soil Sample
- Proposed Soil Samples Collected during Well Installation

Proposed RI Locations

- Proposed Till / Weathered Bedrock Well
- Proposed Shallow Bedrock Well
- ESI Soil Sample
- SEAD Boundary
- Former Fire House
- Stormwater Drainage
- Groundwater Flow Direction
- Road
- Surface Water Flow Direction
- Approximate PID Area Boundary where GW use is prohibited

Notes:

1) Twenty surface soil samples will be collected from 0 to 0.5 ft bgs. Locations tentative based on field conditions.
2) Eight subsurface (1.5 to 2 ft bgs) locations will be selected based on surface soil sampling results. Subsurface locations will target elevated surface soil concentrations.
3) Surface and subsurface soil samples will be collected during well installation at the identified locations.
4) Symbol red if PFOA or PFOS >= 10 ng/L during ESI.



DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

N

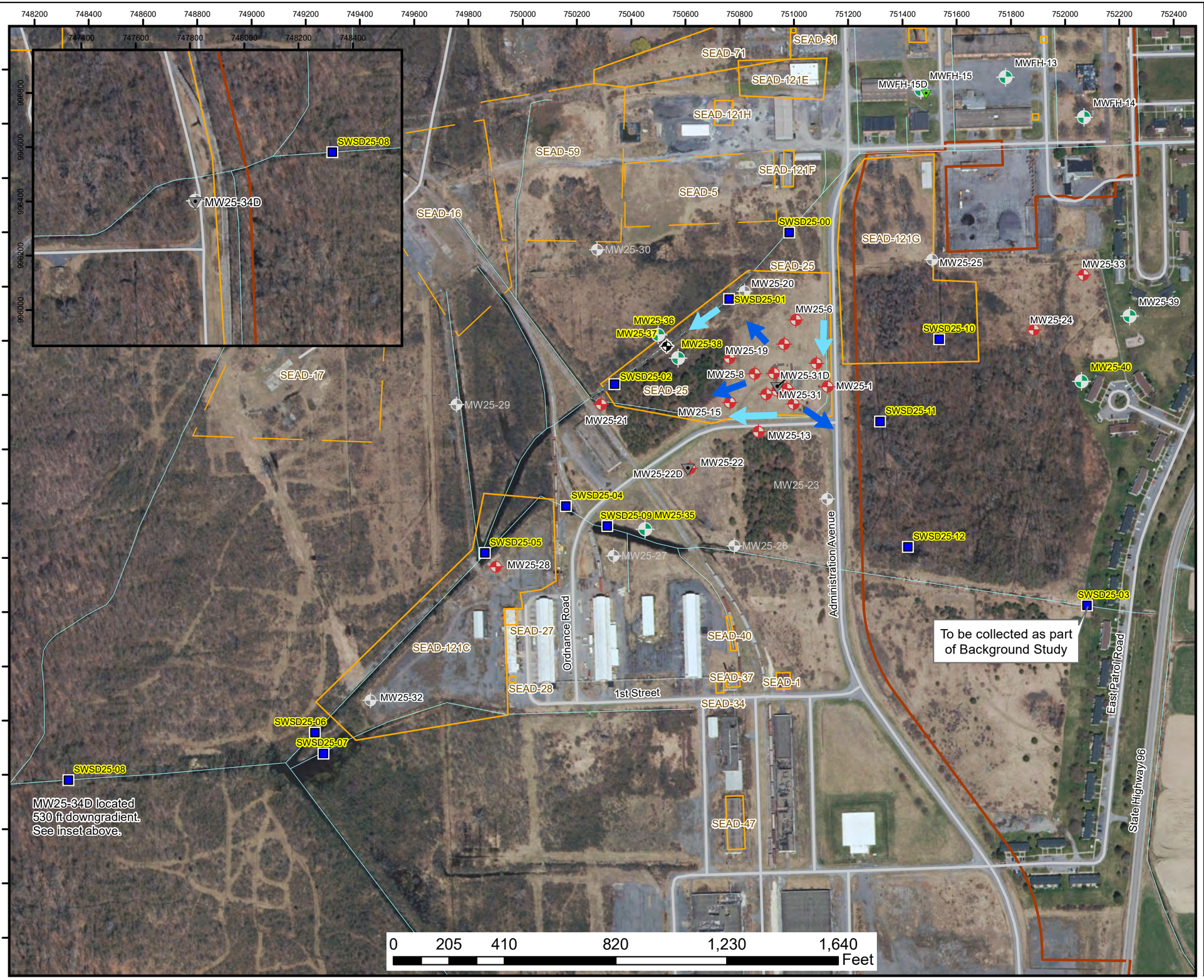
SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 3: FIREHOUSE – PROPOSED SOIL SAMPLING LOCATIONS

1 inch = 100 feet	January 2023

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_FH_SO_v2.mxd



Legend

ESI Sample (Red symbol if exceedance)

- Existing Till / Weathered Bedrock MW
- Existing Shallow Bedrock MW
- Proposed Sediment Sample
- Proposed Paired Surface Water and Sediment Sample

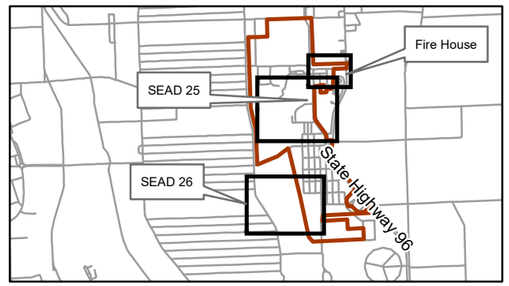
Proposed RI Locations

- Proposed Till / Weathered Bedrock Well
- Proposed Shallow Bedrock Well
- Proposed Piezometer

- RA Excavation Extents
- SEAD (dashed PFAS SI applies)
- Stormwater Drainage
- Approximate PID Area Boundary where GW use is prohibited
- Road
- Groundwater Flow Direction
- Surface Water Flow Direction

Notes:

1) Symbol red if PFOA or PFOS >= 10 ng/L during ESI.
2) All well locations tentative based on accessibility and utility locations. Final determination to be made in the field.
3) Drainage pathway locations approximate. Storm drainage flow direction is to the south and southwest.
4) Predominant groundwater flow direction is radial near the RA excavation area (former SEAD-25 pad) transitioning towards the southwest outside the SEAD-25 AOC.
5) Eighteen existing wells wells with white labels will be sampled once during the RI. Newly installed wells (yellow labels) will be sampled twice.



DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

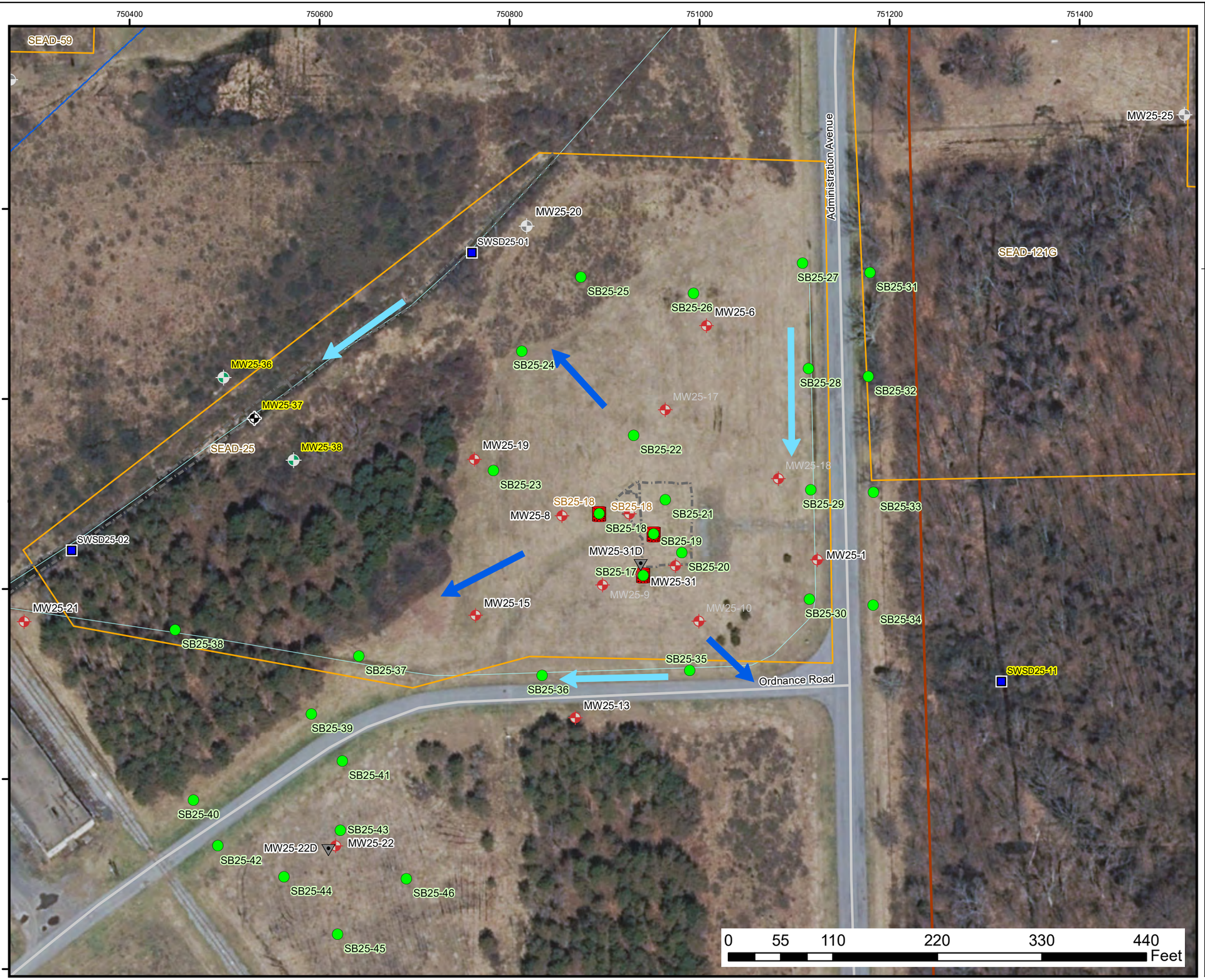
SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 4: SEAD-25 – PROPOSED GROUNDWATER, SURFACE WATER AND SEDIMENT LOCATIONS

1 inch = 350 feet	January 2023

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_S25_PadArea_GWSW_v2.mxd



Legend

ESI Samples (Red symbol if

- Existing Till / Weathered Bedrock MW
- Existing Shallow Bedrock MW
- Proposed Paired Surface Water and Sediment
- ESI Soil Sample

Proposed RI Locations

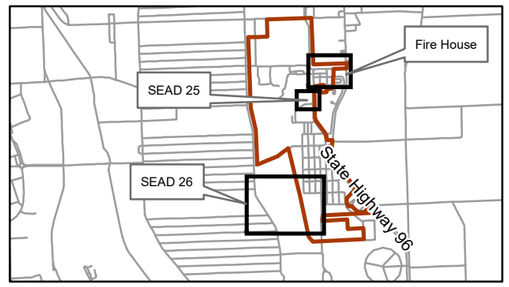
- Proposed Till / Weathered Bedrock Well
- Proposed Shallow Bedrock Well
- Proposed Piezometer
- Proposed Surface Soil Sample

- RA Excavation Extents
- SEAD Boundary
- Stormwater Drainage
- Road
- Approximate PID Area Boundary where GW use is prohibited
- Groundwater Flow Direction
- Surface Water Flow Direction

Notes:

1) Thirty surface soil samples will be collected from 0 to 0.5 ft bgs. Locations tentative based on field conditions.

2) Ten subsurface (1.5 to 2 ft bgs and above water table) locations will be selected based on surface soil sampling results. Subsurface locations will target elevated surface soil concentrations.



DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 5: SEAD-25 – PROPOSED SOIL SAMPLING LOCATIONS

1 inch = 100 feet	January 2023

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_S25_PadArea_SO_v2.mxd

748000 748500 749000 749500 750000 750500 751000 751500

993500 993000 992500 992000 991500 991000

SWSD26-06
SWSD26-08
SWSD26-05
SWSD26-07
SWSD26-04
SWSD26-09
SWSD26-10
PW26-01
PW26-02
MW26-25
MW26-24
MW26-23D
MW26-23
MW26-20
MW26-15
MW26-18
MW26-16
MW26-32D
MW26-13
MW26-28D
MW26-28
MW26-27
MW26-12
MW26-26
MW26-29
MW26-33
MW26-34
MW26-30
MW26-31
MW26-19
SEAD-121I
SEAD-26
SEAD-64A
SEAD-53
Dumpster
Fire Tower
Trailer
Container
Burned Automobiles
Former Bentonite Lined Pit
Autos
Storage Area
Helicopter Fuselage
Former Drum Storage Area

0 187.5 375 750 1,125 1,500 Feet

DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

Legend

ESI Samples (Red symbol if exceedance)

- Existing Till / Weathered Bedrock MW
- Existing Shallow Bedrock MW
- Surface Water Sample (Red symbol if exceedance)

Temporary Monitoring Wells (Abandoned) (Red symbol if exceedance during SI)

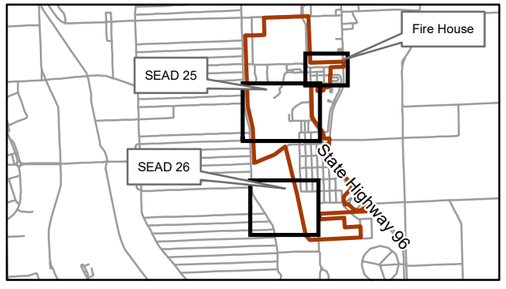
- Existing Till / Weathered Bedrock MW

Proposed RI Locations

- Proposed Till / Weathered Bedrock Well
- Proposed Paired Surface Water and Sediment Sample
- Porewater Sample

- Federal Wetlands
- RA Excavation Extents
- Former SEAD 26 Features
- SEAD Boundary
- Approximate PID Area Boundary where GW use is prohibited
- Drainage
- Road
- Groundwater Flow Direction
- Surface Water Flow Direction

Notes:
1) Symbol red if PFOA or PFOS >= 10 ng/L during ESI.
2) All well locations tentative based on accessibility and utility locations. Final determination to be made in the field.
3) Drainage pathway locations approximate. Storm drainage flow direction is to the west.
4) Nineteen existing wells with labels will be sampled once during the RI. Newly installed wells will be sampled twice .
5) Sampling locations SWSD26-11, -12 and -13 will be located in the wetland west of the pond and will be placed based on field conditions.
ng/L = nanograms per liter



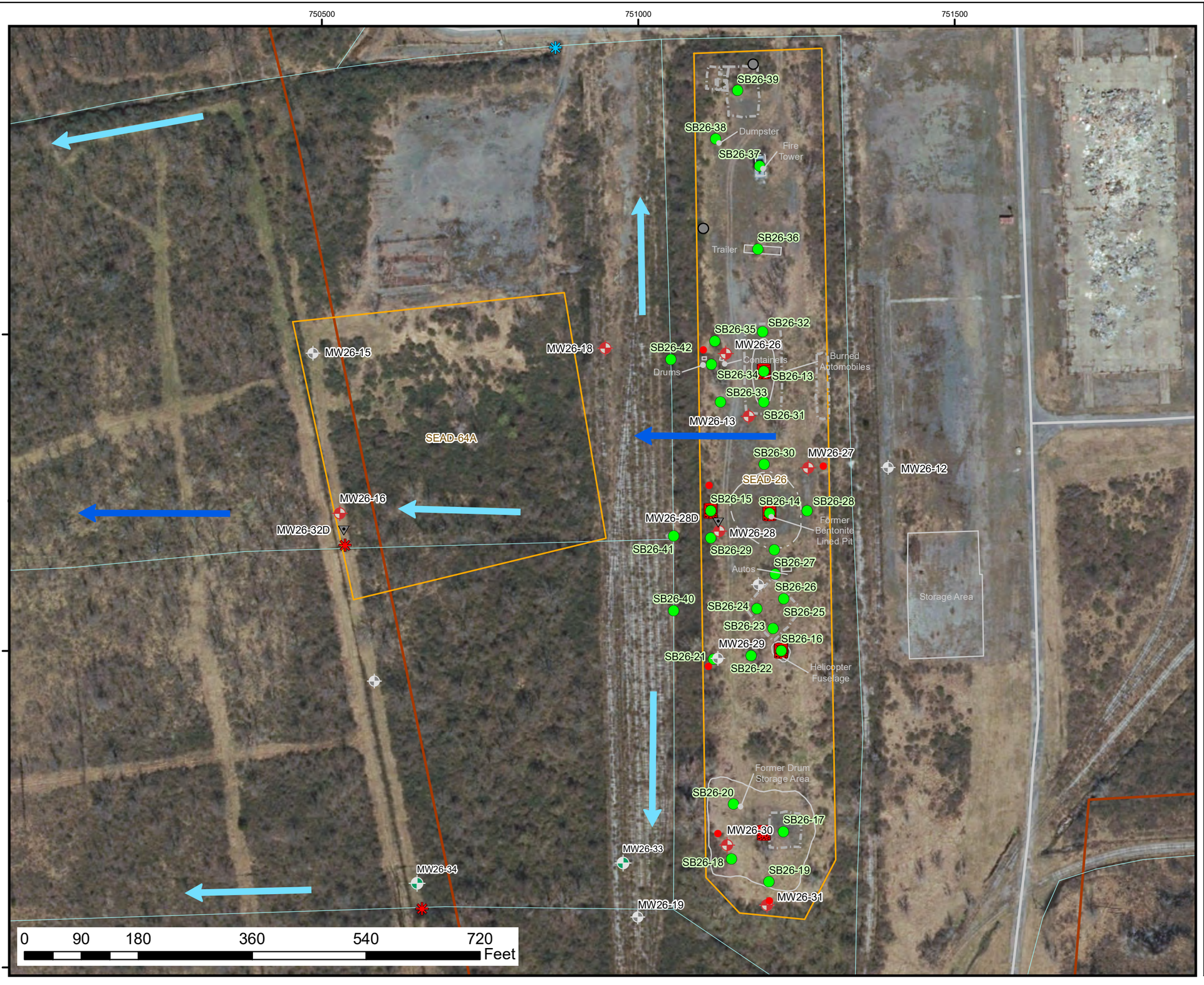
SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 6: SEAD-26 – PROPOSED GROUNDWATER, SURFACE WATER AND SEDIMENT SAMPLING LOCATIONS

1 inch = 300 feet	January 2023

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_S26_PadArea_GWSW_v2.mxd



Legend

ESI Samples (Red symbol if exceedance)

- Existing Till / Weathered Bedrock MW
- Existing Shallow Bedrock MW
- ESI Soil Sample
- Surface Water Sample (Red symbol if exceedance)

Temporary Monitoring Wells (Abandoned) (Red symbol if exceedance during SI)

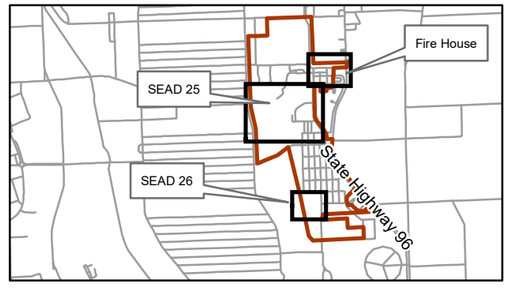
- Existing Till / Weathered Bedrock MW

Proposed RI Locations

- Proposed Till / Weathered Bedrock Well
- Proposed Soil Sample Locations
- RA Excavation Extents
- Former SEAD 26 Features
- SEAD Boundary
- Drainage Feature
- Approximate PID Area Boundary where GW use is prohibited
- Road
- Groundwater Flow Direction
- Surface Water Flow Direction

Notes:

1) Thirty surface soil samples will be collected from 0 to 0.5 ft bgs. Locations tentative based on field conditions.
2) Twelve subsurface (1.5 to 2 ft bgs) locations will be selected based on surface soil sampling results. Subsurface locations will target elevated surface soil concentrations.



DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 7: SEAD-26 – PROPOSED SOIL SAMPLING LOCATIONS

1 inch = 150 feet	May 2022

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_S26_PadArea_SO.mxd

736000 736500 737000 737500 738000 738500 739000 739500 740000 740500 741000 741500 742000 742500 743000

992000 991500 991000 990500 990000 989500 989000 988500 988000 987500 987000 986500

SWSD122E-01
MW122E-04
MW122E-05
SEAD-64D
Regional Groundwater Flow Direction
MW122E-06
SWSD122E-02
TMW-122E-24
SW122E-03
MW122E-07
MW122E-08
SW122E-04
MW122E-11
MW122E-09
Former Fire House
MW122E-10
County Fire Training Area
SWSD122D-01
MW122D-01
MW122D-02
MW122D-03
SEAD-122A
TMW-122E-14
MW122D-04
SWSD122D-02
MW122E-12
SWSD122E-05
SEAD-122C
MW122E-13
SWSD122E-06

0 375 750 1,500 2,250 3,000 Feet

Legend

Proposed RI Locations

- Proposed Till / Weathered Bedrock Well
- Proposed Paired Surface Water and Sediment Sample
- Proposed Surface Water Sample

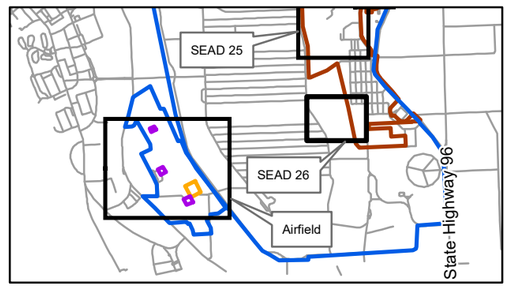
2018 SI Temporary Wells

- Groundwater Sample (PFOA or PFOS >= 10 ng/L)
- PFAS Sample Below State MCL

- SEAD-122D
- SEAD-122E
- SEAD (dashed PFAS SI)
- Drainage Feature
- Road
- Groundwater Flow Direction
- Surface Water Flow Direction

Notes:

1) Four bedrock wells will be installed at locations with maximum PFAS concentrations in overburden groundwater.
2) Surface water (SW) and sediment (SD) samples to be collected in drainage ditches and drainage outfalls.
3) If permanent water is not present in stormwater conveyances, stormwater samples will be collected after a precipitation event and sediment will be considered surface soil as these features do not support ecological receptors.
4) Regional groundwater flow is interpreted to be towards the southwest.
5) Stormwater flow will tend to flow away from airport infrastructure. SEAD 122D will drain to the NW and SE. The southern SEAD 122E AOC will drain to the SE. The central and northern SEAD 122E AOCs will drain west and have outfalls that discharge stormwater offsite.
6) The groundwater at all proposed well locations will be sampled twice.
7) All well locations tentative based on accessibility and utility locations. Final determination to be made in the field.



DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

N

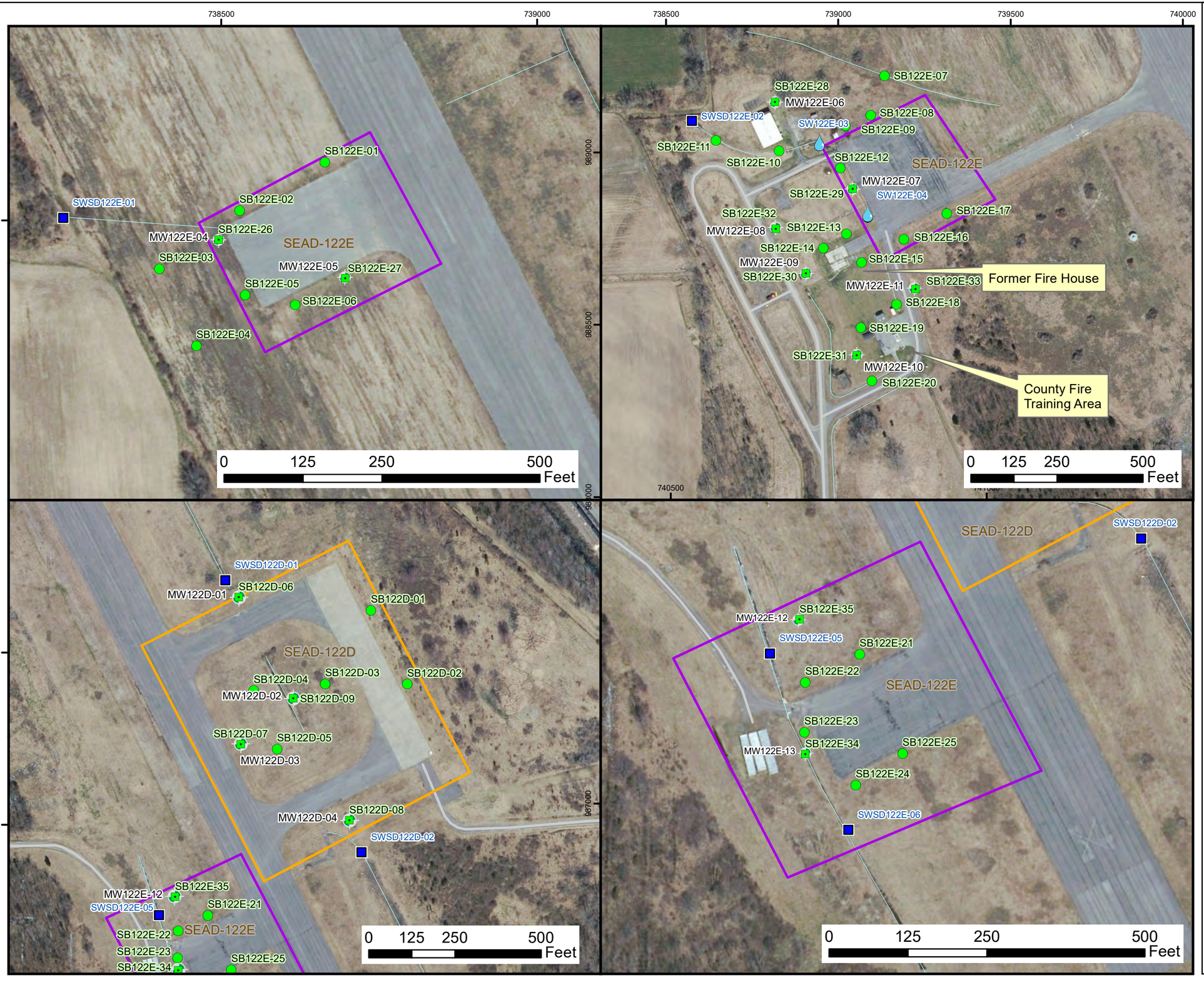
SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 8: AIRFIELD – PROPOSED GROUNDWATER, SURFACE WATER AND SEDIMENT LOCATIONS

1 inch = 600 feet	January 2023

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_Airfield_GWSW_v3.mxd



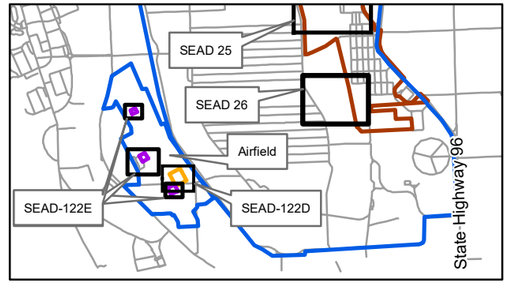
Legend

Proposed RI Locations

- Proposed Till / Weathered Bedrock Well
- Proposed Paired Surface Water and Sediment Sample
- Proposed Surface Water Sample
- Proposed Surface Soil Sample
- Proposed Soil Samples Collected during Well Installation
- SEAD-122D
- SEAD-122E
- Drainage Feature
- Road

Notes:

1) Thirty surface soil samples will be collected from 0 to 0.5 ft bgs. Locations tentative based on field conditions.
2) Ten subsurface (1.5 to 2 ft bgs) locations will be selected based on surface soil sampling results. Subsurface locations will target elevated surface soil concentrations.
3) Surface and subsurface soil samples will be collected during well installation at the identified locations.



DATA SOURCES
-Image: ESRI, 2016
-Parsons: Sample Locations, Boundaries

SENECA ARMY DEPOT ACTIVITY
PFAS EXPANDED SITE INVESTIGATION

FIGURE 9: AIRFIELD – PROPOSED SOIL SAMPLING LOCATIONS

Scale As Shown	February 2023

Coordinate System: NAD 1983 StatePlane New York Central FIPS 3102 Feet

PATH: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\RI\Maps\RI_WP\PFAS_RIWP_Airfield_SO_v2a.mxd

Figure 1A
Proposed SI Sample Locations

735000
740000
1015000
1010000
1005000

N
W
E
S

SEAD-21
SEAD-7
Fire House
Building 722
SEAD 006-R-01
SEAD-23
SEAD 003-R-01
SEAD 007-R-01
SEAD-70

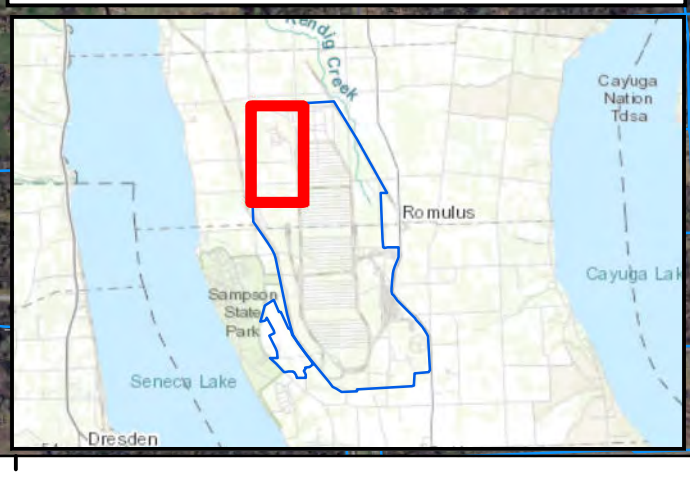
Legend

Proposed SI Locations
- Existing Well to Sample
- New Shallow Well
- Surface Water/Sediment
- Existing Wells
- General GW Flow Direction

PFAS AOCs
- PA/SI
- RI

Water Bodies
- Stream
- Drainage
- Former SEDA Boundary

0 600 1,200 2,400 Feet



Remedial Investigation at Four Known PFAS Sites and
Preliminary Assessment/Site Inspection at Suspected PFAS Sites
HydroGeoLogic, Inc., December 2022

Path: \\MABOS07FS01\Projects\PIT\Projects\Huntsville - MEGA\Seneca_HGL_PFAS\Deliverables\05 GIS\QAPP\Maps\SenecaPASSI_Index_DD_v5.mxd

Figure 1B
Proposed SI Sample Locations

750000

1005000

N
W
E
S

SEAD 002-R-01

SEAD 002-R-01

SEAD-46

SEAD-20

SEAD-67

Legend

Proposed SI Locations
- Existing Well to Sample
- New Shallow Well
- Surface Water/Sediment
- Existing Wells
- General GW Flow Direction

PFAS AOCs
- PA/SI
- RI

Water Bodies
- Stream
- Drainage
- Former SEDA Boundary

0 445 890 1,780
Feet

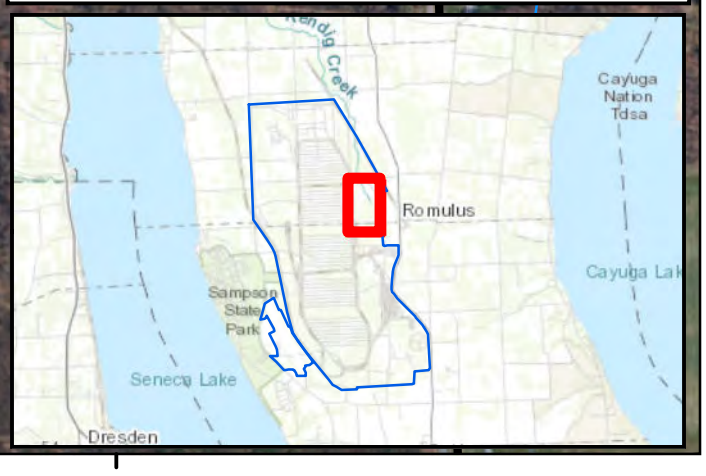


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Figure 1C
Proposed SI Sample Locations

750000

1000000

995000

N
W
E
S

SEAD-9
SEAD-10
SEAD-16
SEAD-59
SEAD-5
SEAD-17
SEAD-25
SEAD-22
SEAD-68

Fire House
Building 103

RI details shown in
RI Work Plan

0 350 700 1,400
Feet

Legend

Proposed SI Locations
- Existing Well to Sample
- New Shallow Well
- Surface Water/Sediment
- Existing Wells
- General GW Flow Direction

PFAS AOCs
- PA/SI
- RI

Water Bodies
- Stream
- Drainage
- Former SEDA Boundary

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Figure 1D
Proposed SI Sample Locations

750000

990000

985000

SEAD-64A

SEAD-26

RI details shown in RI Work Plan

SEAD-69

SEAD-64B

SEAD-64C

N

W

E

S

Legend

Proposed SI Locations

- Existing Well to Sample
- New Shallow Well
- Surface Water/Sediment
- Existing Wells
- General GW Flow Direction

PFAS AOCs

- PA/SI
- RI

Water Bodies

- Stream
- Drainage
- Former SEDA Boundary

0 500 1,000 2,000 Feet

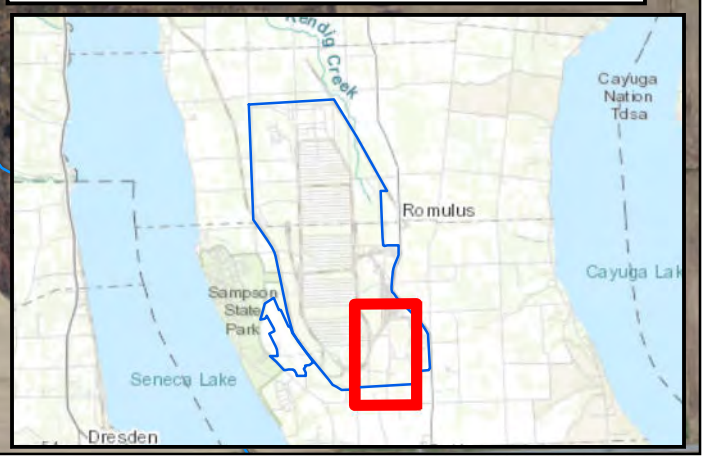

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Figure 1E
Proposed SI Sample Locations

740000

990000

985000

N
W
E
S

SEAD-64D

RI details shown in RI Work Plan

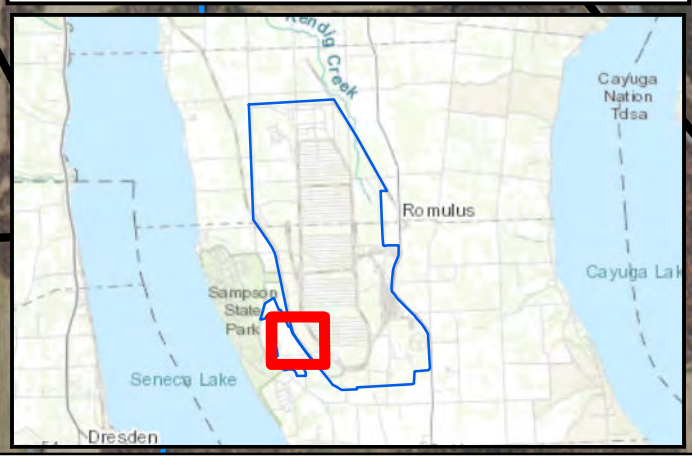
SEAD-122E

SEAD-122D

SEAD-11

SEAD-122E

0 445 890 1,780
Feet



Legend

Proposed SI Locations
- Existing Well to Sample
- New Shallow Well
- Surface Water/Sediment
- Existing Wells
- General GW Flow Direction

PFAS AOCs
- PA/SI
- RI

Water Bodies
- Stream
- Drainage
- Former SEDA Boundary

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Figure 1F
Proposed SI Sample Locations

740000

1000000

995000

N
W
E
S

SEAD-58

SEAD-24

SEAD-14

SEAD-6

SEAD-3

SEAD-15

SEAD-8

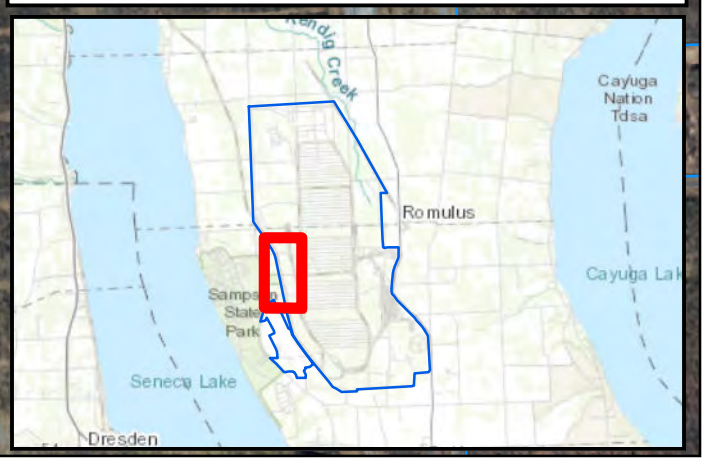
Legend

Proposed SI Locations
- Existing Well to Sample
- New Shallow Well
- Surface Water/Sediment
- Existing Wells
- General GW Flow Direction

PFAS AOCs
- PA/SI
- RI

Water Bodies
- Stream
- Drainage
- Former SEDA Boundary

0 475 950 1,900
Feet



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