FINAL

Uniform Federal Policy
Quality Assurance Project Plan (UFP-QAPP)

for

Per-and Polyfluoroalkyl Substances (PFAS)
Site Inspection Work Plan

at

National Aeronautics and Space Administration (NASA)
Jet Propulsion Laboratory (JPL)
Pasadena, California

Contract No. W912PL21D0021
Delivery Order No.: W912PL21F0046

US Army Corps of Engineers,
Los Angeles District
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Los Angeles, California 90017-3401

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October 2022

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ATTACHMENT A ..... Laboratory DoD ELAP Certification and Standard Operating Procedures
ATTACHMENT B ..... Project Standard Operating Procedures and Instrument Manuals
ATTACHMENT C ..... Field Data Collection Forms

ATTACHMENT D ..... Notification Levels

LIST OF ACRONYMS

°C	degrees Centigrade
APP	Accident Prevention Plan
bgs	below ground surface
CA	corrective action
CAS	Chemical Abstract Service
CCV	continuing calibration verification
CIT	Certified Instructional Trainer
COR	Contracting Officer Representative
CQC	Contractor Quality Control
CSM	conceptual site model
CSP	Certified Safety Professional
DL	detection limit (laboratory)
DoD	Department of Defense
DOECAP	United States Department of Energy Consolidated Audit Program
DQI	data quality indicator
ELAP	Environmental Laboratory Program
EPA	U.S. Environmental Protection Agency
ft	feet/foot
GW	groundwater
HAZWOPER	Hazardous Waste Operations and Emergency Response
ICAL	initial calibration
ICV	initial calibration verification
IDW	investigation derived Waste
LC/MS/MS	liquid chromatography - tandem mass spectrometry
LCS	laboratory control sample
LDC	Laboratory Data Consultants, Inc.
LOD	limit of detection
LOQ	level of quantitation
MB	method blank
MD	matrix duplicate
MPC	measurement performance criteria
MS	matrix spike/mass spectrometer
MSD	matrix spike duplicate
MW	Monitoring Well
NASA	National Aeronautics and Space Administration
ng/L	nanograms per liter
PE	Professional Engineer
PFBS	perfluorobutanesulfonic acid
PG	Professional Geologist
PM	Project Manager
PMP	Project Management Professional
PSL	Project Screening Level
QA	Quality Assurance
QC	Quality Control
QSM	Quality Systems Manual
RPD	relative percent difference
RSL	Regional Screening Level
RT	Retention time
SEDD	Staged Electronic Data Deliverable
SSHP	Site Safety and Health Plan
SOP	Standard Operating Procedure
SWRCB	State Water Resources Control Board
UFP-QAPP	Uniform Federal Policy-Quality Assurance Project Plan

This QAPP was developed in accordance with the Intergovernmental Data Quality Task Force, Uniform Federal Policy of Quality Assurance Project Plans, Optimized UFP-QAPP Worksheets, March 2012

QAPP Worksheet #1 & 2: Title and Approval Page
(Uniform Federal Policy-Quality Assurance Project Plan [UFP-QAPP] Manual Section 2.1)
(EPA 2106-G-05 Section 2.2.1)

1. Project Identifying Information

a. Site name/project name: Per- and Polyfluoroalkyl Substances (PFAS) Site Inspection (SI)
b. Site location/number: National Aeronautics and Space Administration's (NASA's) Jet Propulsion Laboratory (JPL) in Pasadena, CA
c. Contract Number: W912PL21D0021 Delivery Order W912PL21F0046

2. Federal Funding Agent: National Aeronautics and Space Administration **(**NASA)

Restoration Project Manager: _____
Signature

Mr. Steve Slaten; NASA, October 2022
Printed Name/Title/Date

3. Investigative Organization: G2S LLC

Investigative Organization's Project Manager: _____
Signature

Mr. Keith Fields, P.E., PMP; G2S LLC, October 2022
Printed Name/Organization/Date

4. Investigative Organization: G2S LLC

Investigative Organization's QA Officer: _____
Signature

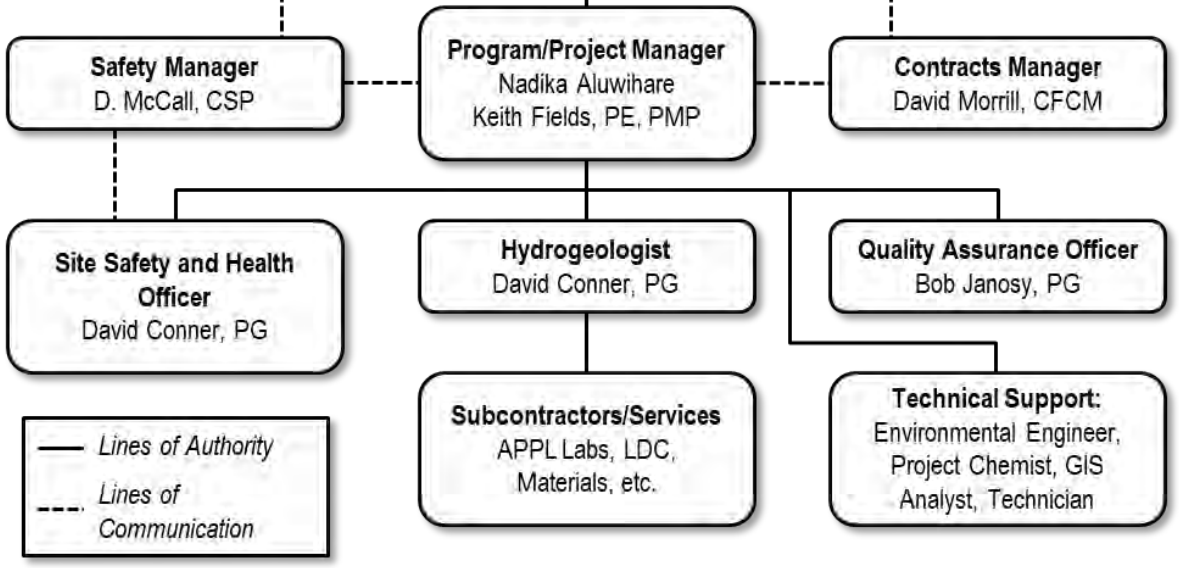
Mr. Bob Janosy, P.G.; G2S LLC, October 2022
Printed Name/Organization/Date

Plans and reports from previous investigations relevant to this project

Tetra Tech, 2021. *Per-and Polyfluoroalkyl Substances Preliminary Assessment Report for Jet Propulsion Laboratory*. February.

QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution
(UFP-QAPP Manual Section 2.3 and 2.4)
(EPA 2106-G-05 Section 2.2.3 and 2.2.4)

Name	Organization	Title
Mr. Steven Slaten	NASA	Remedial Project Manager (RPM)
Ms. Janice Opperman	USACE	Contracting Officer's Representative (COR)
Ms. Patricia Bonilla	USACE	Contracting Officer
Ms. Claudia Garcia	USACE	Program Manager
To Be Determined	USACE	Field Representative



QAPP Worksheet #4, 7, & 8: Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)
(EPA 2106-G-05 Section 2.2.1 and 2.2.7)

Project Personnel	Title	Education/Experience	Specialized Training/Certifications	Telephone Number	Signature/Date
Organization: NASA					
Steve Slaten	Remedial Project Manager			818-393-6683	
Organization: United States Army Corps of Engineers (USACE), Los Angeles District					
Claudia Garcia	Program Manager			602-230-6924	
Janice Opperman	Contracting Officer Representative (COR)			858-430-2810	
Organization: G2S LLC					
Keith Fields	Program Manager	B.S. Civil Engineering; 20 years of site characterization and remedial action selection, design, and implementation	Professional Engineer (PE), Project Management Professional (PMP)	614-792-2896	
Nadika Aluwihare	Project Manager	B.A. Business Administration and Accounting; 16 years of experience managing environmental investigation contracts	-	443-453-9521	
David McCall	Safety and Health Manager	B.S. Occupational Safety and Health Technology/Technician, MS Environmental Science; >25 years of experience developing, implementing, and monitoring H&S programs	Certified Safety Professional (CSP), Certified Instructional Trainer (CIT)	740-504-9714	
David Conner	SSHO/Site Superintendent/Project Hydrogeologist	B.S. Geology; 20 years of site characterization and remediation experience including Contractor Quality Control (CQC) Manager experience	Professional Geologist (PG), 40-Hour Hazardous Waste Operations (HAZWOPER)	626-298-5715	

Project Personnel	Title	Education/Experience	Specialized Training/Certifications	Telephone Number	Signature/Date
Ben Dettorre	Project Chemist	BS Chemistry, BAS Construction Management, 25 years of experience in the areas of characterization, remediation/decommissioning, and waste management; both environmental and facilities	DOE Consolidated Audit Program (DOECAP) Lead Auditor for analytical chemistry	865-266-0076	
Bob Janosy	Quality Assurance (QA) Officer	B.S. and M.S. Geological Sciences; 20 years of site characterization and remediation experience	PG; HAZWOPER 40-hour OSHA; USACE CQM	614-593-5541	
Organization: Agriculture & Priority Pollutants Laboratories (APPL), Inc., Clovis, CA					
Gregory Salata	Laboratory Project Manager	B.A. Chemistry, UCSD, Ph.D. Oceanography, TAMU, 34 years experience in laboratory and client services		559-862-2133	
Paula McCartney	Laboratory Quality Assurance Manager	B.A. Chemistry, WVU, over 30 years laboratory experience		559-862-2118	
Organization: Laboratory Data Consultants (LDC)					
Pei Geng	Project Manager	M.S. Organic Chemistry B.S. Environmental Chemistry		760-827-1100 extension 141	

*Signatures indicate personnel have read and agree to implement the QAPP as written

QAPP Worksheet #6: Communication Pathways
(UFP-QAPP Manual Section 2.4.2)
(EPA 2106-G-05 Section 2.2.4)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Point of Contact with NASA Project Manager (PM)	NASA Project Manager	Steve Slaten	818-393-6683	All materials and information about the project will be forwarded via e-mail to the NASA PM by G2S LLC's PM immediately upon completion/receipt.
Technical Issues with USACE	USACE	Janice Opperman	830-221-8025	Technical issues that arise will be discussed via phone with NASA and USACE.
Manage all Project Phases	G2S LLC Project Manager	Keith Fields	614-792-2896	Overall management of the project. Maintain lines of communication between USACE, NASA, regulators, and subcontractors via phone and e-mail. Single point of contact for USACE PM.
QAPP Changes in the Field	Site Supervisor	David Conner	626-298-5715	Notify G2S LLC PM by phone and email changes to QAPP made in the field and the reasons immediately. The G2S LLC PM will notify NASA and USACE via e-mail immediately upon receipt. NASA will notify regulators via e-mail within 1 week of being notified.
Daily Field Progress Reports	Site Supervisor	David Conner	626-298-5715	Site Supervisor will email or fax daily field progress reports to the G2S LLC PM, who will review and distribute to NASA and USACE via e-mail.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Reporting Laboratory Data Quality Issues	Project Chemist	Ben Dettorre	865-266-0076	All QA/Quality Control (QC) issues with project field samples will be reported to G2S LLC PM immediately. The issue and possible solutions will be identified and the G2S LLC PM will notify NASA and USACE via phone and e-mail immediately of issues and proposed solutions.
Field Corrective Actions	Project Hydrogeologist	David Conner	626-298-5715	The need for corrective actions for field issues will be determined by the Project Geologist. Any corrective actions will be immediately communicated to the USACE and NASA via phone and e-mail. NASA will notify regulators within 1 week via e-mail of being notified of field corrective actions that impact the technical approach.
Release of Analytical Data	Project Chemist	Ben Dettorre	865-266-0076	No analytical data can be released until validation is completed and the Project Chemist has approved the release. Once the validation is complete and data approved, G2S LLC will utilize the data within the appropriate reports.
QAPP Amendments	NASA Project Manager	Steve Slaten	818-393-6683	Any major changes to the UFP-QAPP must be reviewed and approved by the NASA and USACE. QAPP Amendments will be submitted to the regulators via e-mail for review and approval.

QAPP Worksheet #9: Project Planning Session Summary and Participants Sheet (UFP-QAPP Manual Section 2.5.1) (EPA 2106-G-05 Section 2.2.5)

Complete this worksheet for each project planning session held. Identify project team members who are responsible for planning the project.

Project Name: PFAS SI
Site Name: NASA JPL
Site Location: Microsoft Teams

Kick-Off Meeting
Date: 8/27/21
Purpose of Session: Determine a path forward for the site.

Participants			
Name	**Organization**	**Title/Role**	**E-mail/Phone**
Steven Slaten	NASA	Cleanup Manager	sslaten@nasa.gov/818.393.6683
Janice Opperman	USACE Los Angeles District	COR	janice.s.opperman@usace.army.mil/ 858.430-2810
Claudia Garcia	USACE Los Angeles District	Project Manager	claudia.garcia@usace.army.mil/ 602.230.6924
Nadika Aluwihare	G2S LLC	Program Manager	nadika.aluwihare@tideh2o.net/ 210.240.9188
David Conner	G2S LLC	Hydrogeologist	david.conner@tideh2o.net/626.298.5715
Sara McGarity	G2S LLC	Environmental Engineer	sara.mcgarity@tideh2o.net/443.963.9793
Keith Fields	G2S LLC	Project Manager	keith.fields@tideh2o.net/614.778.2618

Notes/Comments/Action Items:

The action items from this meeting included: (1) to coordinate a monthly tag-up meeting every third Thursday at 0830 Pacific Time comprised of NASA, USACE, and G2S LLC Project Teams, and (2) to ascertain whether NASA requires additional headquarters-level reviews of work plans and reports.

QAPP Worksheet #10: Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)
(EPA 2106-G-05 Section 2.2.5)

The conceptual site model (CSM) is presented in the Work Plan Sections 2, 3 and 4.

CSM components can be found in the following sections of the work plan:

CSM Component	Work Plan Section
Background information	Section 2: Site Background
Sources of known or suspected hazardous waste	Section 2.2: Site History
Known or suspected contaminants or classes of contaminants	Section 4.1.1: Potential Contaminant Sources
Primary release mechanism	Section 2.2: Site History and Section 4.1.2 Affected Media
Secondary contaminant migration	Section 4.1.2 Affected Media
Fate and transport considerations	Sections 4.1.2 Affected Media, 4.1.3 Potential Human Receptors and Exposure Pathways, and 4.1.4 Potential Ecological Receptors and Exposure Pathways
Potential receptors and exposure pathways	Sections 4.1.3 Potential Human Receptors and Exposure Pathways and 4.1.4 Potential Ecological Receptors and Exposure Pathways
Land use considerations	Section 3.6: Land Use
Key physical aspects of the site (e.g., site geology, hydrology, topography, climate)	Section 3: Environmental Setting
Current interpretation of nature and extent of contamination to the extent that it will influence project-specific decision-making.	Sections 4.1.1 Potential Contaminant Sources, 4.1.2 Affected Media, and 4.1.5: Contaminants of Potential Concern

Data Gaps include:

Data Gaps	Work Plan Section / SI Activities
Investigate the presence or absence of PFAS at or associated with AOPC 1: Emergency Landing Facility	Sections 4.2 SI Objectives and 4.3 SI Approach / Soil and Groundwater Sampling at locations identified in the PAR for PFAS analysis
Investigate the presence or absence of PFAS at or associated with AOPC 2 and AOPC 3: Waste Disposal Areas	
Investigate the presence or absence of PFAS at or associated with AOPC 4: Building 170 Fabrication Shop	
Investigate the presence or absence of PFAS at or associated with AOPC 5: Former Photography Labs.	

QAPP Worksheet #11: Project/Data Quality Objectives
(UFP-QAPP Manual Section 2.6.1)
(EPA 2106-G-05 Section 2.2.6)

Step 1 - Problem Statements:

The objective of the PFAS SI at NASA JPL is to implement the environmental investigations specified in Appendix F of the PFAS Preliminary Assessment Report for JPL (Tetra Tech, 2021). The preliminary assessment conducted at JPL is one of 10 PFAS PA's conducted at NASA Centers/Facilities to identify whether past or present activities may have resulted in a release of PFAS into the environment and to qualitatively evaluate potential exposure to PFAS in environmental media at and within a 1-mile radius of the facility. The PAR for JPL identified and recommended five AOPCs for further assessment.

Step 2 - Study Goals:

The goal of the SI is to determine the presence or absence of PFAS at or associated with the five AOPCs identified in the PAR as follows:

- AOPC 1: Emergency Landing Facility
- AOPC 2 and 3: Waste Disposal Areas (Seepage Pits and Waste Pits)
- AOPC 4: Building 170 Fabrication Shop (former chrome plating activities)
- AOPC 5: Former Building 218 and Building 291 Photography Labs

The presence or absence of PFAS will be evaluated through the collection of shallow soil samples associated with AOPC 1 and the collection of groundwater samples from existing wells to evaluate AOPCs 1 through 5. The approach for specific sampling locations and their association with each of the AOPCs is described in Section 4.3 of the SI Work Plan.

Step 3 - Information Inputs:

The data required to fill the data gaps include groundwater and soil analytical results for PFAS analysis from the targeted sampling locations associated with each of the AOPCs as identified in the SI Work Plan. The analytical data will be screened against project screening levels (see Worksheet #15) to determine if potential releases associated with any of the five AOPCs have impacted environmental media identified in the CSM.

Step 4 - Boundaries of the Study:

The spatial boundary of the study includes AOPCs 1 through 5 and the area downgradient of JPL, as depicted in Figure 6 of the Work Plan, in the vicinity of City of Pasadena wells Arroyo Well and Well 52. The target analytes for this study are limited to the list of PFAS compounds identified in Section 4.1.5 of the SI Work Plan. Potentially affected media at the Site include surface soil and groundwater associated with AOPC 1 and groundwater associated with AOPCs 1 through 5 related to potential contaminant sources associated with the Emergency Landing Facility, Waste Disposal Area, and chrome plating at the Fabrication Shop. Sampling results are not expected to vary depending on weather conditions. Analytical results for respective sampling locations will be applied to the AOPCs with which those specific sampling locations are identified. Some sampling locations are associated with multiple AOPCs as identified in Table 2 of the Work Plan.

The temporal boundary of the study is the fall of 2022. Groundwater and soil samples will be

collected in a single field event separate from the quarterly groundwater sampling event.

Step 5 - Analytical Approach:

All soil and groundwater samples will be analyzed for the list of PFAS compounds identified in Section 4.1.5 of the SI Work Plan. Groundwater samples will be collected from existing wells. Soil samples will be collected at shallow depths of 0-0.5 feet (ft) below ground surface (bgs) and 0.5 to 2.0 ft bgs at six locations as described in Table 2 of the SI Work Plan. Samples to be collected for quality control purposes are described in Section 5.4 and Table 4 of the SI Work Plan.

Soil sampling results will be used to determine if there was PFAS impact associated with a potential AFFF release during a one-time fire training exercise at AOPC 1. Sampling includes the collection of six soil samples from the southern edge of the helipad at AOPC 1 and six samples from the depression below the helipad (contingent upon accessibility). The exact sample locations will be as close as possible to the proposed locations; however, final locations will be based on an on-site evaluation of the physical features and the ability to access the proposed locations.

- If a sample cannot be collected where planned, then the decision process for changing the location of a soil sample will involve the site supervisor coordinating with the project manager to ensure the alternative proposed location meets the sampling objectives of the initial location. The project manager will communicate any field changes consistent with WS#6.

Groundwater sampling results will be used to evaluate potential PFAS impacts associated with the above-mentioned incident at AOPC 1, seepage and waste pits associated with AOPCs 2 and 3, chrome plating operations at AOPC 4, and photography lab activities at AOPC 5. Sampling locations have been selected to determine downgradient impacts which could have potentially been associated with each of the AOPCs and includes several wells screened at multiple depths encompassing distinct layers of the aquifer.

Detections in either soil or groundwater would indicate a potential releasee associated with the AOPC with which those specific sampling locations correspond.

Comparison of detections with screening levels will guide determination of next steps.

- If no detections exceed the project screening levels (PSLs), then it will be concluded that no releases have occurred which present unacceptable impacts to potential receptors and no additional sampling will be recommended. The results of the investigation would be documented in a final SI Report.
- If detections exceed the PSLs, then recommendations will be made in the final SI Report for additional investigation as part of an expanded SI. It is noted that PFAS has a rapidly changing regulatory context and laboratory analytical methodologies. See worksheet #15 for an explanation of PSLs.

Step 6 - Performance or Acceptance Criteria:

The hypothesis testing for presence or absence of contamination exceeding the screening levels involves specifying probability limits for decision errors.

One type of decision error, referred to as a false negative error, may arise if sampling or analyses fail to detect contamination that is present at levels of concern. This type of error would result in incorrectly concluding that contamination is not present at levels exceeding the screening level. This type of error will be minimized by optimizing the sampling design such

that some samples are collected where contamination is most likely to exist (biased sampling). Also, analytical detection limits will be used that are below the PSLs specified in Worksheet #15.

Incorrectly concluding that contamination is present at levels above the project screening level PSLs, when in fact it is not, called false positive error is another type of potential decision error. This type of error may result if analytical results of groundwater samples overestimate actual contaminant concentrations, if samples are cross-contaminated, or if contaminants are misidentified. To minimize the potential for this type of error, appropriate sampling, and analytical methods (including thorough decontamination procedures, sampling less likely to be contaminated wells first, etc.) will be employed, and all laboratory data will undergo third-party validation to identify any problems that could lead to this type of decision error.

Step 7 – Plan for Obtaining Data

The sampling design is based on a bias sampling design. Soil and groundwater sampling locations and depth intervals have been selected such that they are most likely to detect a release to groundwater or soil. See Worksheet #17 for additional details.

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

See worksheet #28 for detailed description of laboratory measurement performance criteria (MPC).
MPC for groundwater sampling stabilization parameters are discussed in Work Plan Section 5.6

Measurement Performance Criteria Table for Groundwater

- PFAS – Groundwater (Liquid chromatography - tandem mass spectrometry (LC/MS/MS) compliant with Department of Defense (DoD) QSM 5.3 Table B-15)

Matrix: Aqueous
Analytical Group or Method: See above
Concentration Level: Low

Data Quality Indicator	**QC Sample or Measurement Performance Activity**	**Measurement Performance Criteria (MPC)**
Precision – Overall	Field duplicates	Relative Percent Difference (RPD) ≤ 30% for target compounds detected in parent sample and field duplicate ≥ Level of Quantitation (LOQ)
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 25%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	See Table C-44. Method PFAS by LC/MS/MS Compliant with QSM Table B-15 Aqueous Matrix
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	See Table C-44. Method PFAS by LC/MS/MS Compliant with QSM Table B-15 Aqueous Matrix
Accuracy/Bias - Contamination	Field Equipment Blanks	No target analyte concentrations ≥ LOQ.
Data Completeness	Number of valid samples. See also Worksheet #34	95%

Measurement Performance Criteria Table for Soil

- PFAS – Soil (LC/MS/MS compliant with DoD QSM 5.3 Table B-15)

Matrix: Soil
Analytical Group or Method: See above
Concentration Level: Low

Data Quality Indicator	**QC Sample or Measurement Performance Activity**	**Measurement Performance Criteria (MPC)**
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 25%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	See Table C-45. Method PFAS by LC/MS/MS Compliant with QSM Table B-15 Solid Matrix
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	See Table C-45. Method PFAS by LC/MS/MS Compliant with QSM Table B-15 Solid Matrix
Accuracy/Bias - Contamination	Field Equipment Blanks	No target analyte concentrations ≥ LOQ.
Data Completeness	Number of valid samples. See also Worksheet #34	95%

QAPP Worksheet #13: Secondary Data Criteria and Limitations
(UFP-QAPP Manual Section 2.7)
(EPA 2106-G-05 Chapter 3: QAPP Elements For Evaluating Existing Data)

Secondary Data	Data Source	Data uses relative to current project	Factors affecting the reliability of data and limitations on data use
Historical records and contaminant data	Per- and Polyfluoroalkyl Substances Preliminary Assessment Report for JPL (Tetra Tech, 2021).	Evaluation of historical data in the interpretation of results of the current investigation	Very recent report with comprehensive data.
Environmental setting of JPL site and surroundings	Per- and Polyfluoroalkyl Substances Preliminary Assessment Report for JPL (Tetra Tech, 2021).	Context for interpretation of current data and potential pathways and receptors	Very recent report with comprehensive data.
Habitat and ecological setting	Per- and Polyfluoroalkyl Substances Preliminary Assessment Report for JPL (Tetra Tech, 2021).	Context for interpretation of current data, potential pathways, and receptors	Very recent report with comprehensive data.
Meteorological	National Weather Service	Seasonal fluctuations in precipitation and other climatic conditions	Published data available for the past 15 years.

QAPP Worksheet #14/16: Project Tasks and Schedule
(UFP-QAPP Manual Section 2.8.1)
(EPA 2106-G-05 Section 2.2.4)

Activity	Responsible Party	Planned Start Date	Planned Completion Date	Deliverable(s)	Deliverable Due Date
Prepare and Submit Draft PMP/QASP	G2S LLC	July 23, 2021	August 23, 2021	Draft PMP/QASP	August 23, 2021
Prepare and Submit Final PMP/QASP	G2S LLC	September 7, 2021	September 20, 2021	Final PMP/QASP	September 20, 2021
Prepare and Submit Client Draft UFP-QAPP and SSHP/APP	G2S LLC	September 21, 2021	November 1, 2021	Client Draft UFP-QAPP and SSHP/APP	November 1, 2021
Prepare and Submit Draft UFP-QAPP and SSHP/APP	G2S LLC	November 23, 2021	May 6, 2022	Draft UFP-QAPP and SSHP/APP	May 6, 2022
Prepare and Submit Final UFP-QAPP and SSHP/APP	G2S LLC	May 6, 2022	August 31, 2022	Final UFP-QAPP and SSHP/APP	August 31, 2022
Prepare and Conduct Site Inspection Field Work	G2S LLC	September 1, 2022	October 31, 2022	PFAS Site Inspection Field Work	October 31, 2022
Conduct Soil Sampling	G2S LLC	October 21, 2022	October 21, 2022		
Conduct Groundwater Sampling	G2S LLC	September 26, 2022	September 30, 2022		
Prepare and Submit Draft Field Work Completion Report	G2S LLC	November 1, 2022	January 31, 2023	Draft Field Work Completion Report	January 31, 2023
Prepare and Submit Final Field Work Completion Report	G2S LLC	February 1, 2023	February 28, 2023	Final Field Work Completion Report	February 28, 2023
Prepare and Submit Client PFAS SI Report	G2S LLC	February 1, 2023	March 31, 2023	Client Draft PFAS SI Report	March 31, 2023
Prepare and Submit Draft PFAS SI Report	G2S LLC	April 1, 2023	April 30, 2023	Draft PFAS SI Report	April 30, 2022
Prepare and Submit Final PFAS SI Report	G2S LLC	May 1, 2023	May 31, 2023	Final PFAS SI Report	May 31, 2023

QAPP Worksheet #15: Reference Limits and Evaluation
(UFP-QAPP Manual Section 2.8.1)

The Project Screening Levels (PSLs) shown in the tables below are based on PFAS regulatory and precautionary health-based values for drinking water and soil as identified in the PFAS Preliminary Assessment Report (Tetra Tech, 2021) and relevant updates from the State Water Resources Control Board (SWRCB) (SWRCB, 2021) and EPA (USEPA, 2022).

The list of 32 PFAS compounds included in the suite of analytes presented below encompasses PFAS compounds from the California Water Boards Order for the Determination of the Presence of Per- and Polyfluoroalkyl Substances at Publicly Owned Treatment Works (SWRCB, 2020), the California Water Boards Order for the Determination of the Presence of Per- and Polyfluoroalkyl Substances at Chrome Plating Facilities (SWRCB, 2019b), and compounds listed in DoD QSM 5.3 Table B-15 (DoD, 2019). The Project Quantitation Limit Goals are based on the lab's LOQ. The Limit of Detection (LOD), laboratory Detection Limit (DL), and LOQ are provided by APPL laboratory.

Groundwater PSLs are based on the SWRCB Drinking Water Notification Levels for perfluorobutanesulfonic acid (PFBS) (SWRCB, 2021), PFOA, and PFOS (SWRCB, 2019a). The respective Notification Level Issuances are included in Attachment D. Notification levels are a nonregulatory, precautionary health-based measure for concentrations in drinking water that warrant notification and further monitoring and assessment (SWRCB, 2019a). These were selected as the most conservative PSLs based on presence of downgradient drinking water systems. EPA Regional Screening Levels (RSLs) for tap water are based on the Summary Table (TR=1E-06, HQ=0.1) updated May 2022 (EPA, 2022). The RSLs are risk-based screening levels, calculated using the latest toxicity values, default exposure assumptions and physical and chemical properties. PSLs have not been established for the remainder of the PFAS compounds; however, the regulatory status and analytical methods associated with PFAS compounds is evolving rapidly. Presence or absence of the remaining compounds will be determined by the current ability of analytical methods to detect such compounds.

Matrix: Groundwater
Analytical Group: PFAS (LC/MS/MS compliant with DoD QSM 5.3 Table B-15)
Concentration Level: Low

Analyte	CAS[1] Number	Project Screening Level (PSL) (ng/L)[2]	PSL Reference	EPA RSLs[8] (ng/L)	Project Quantification Limit Goal (ng/L)	Laboratory-specific		
						LOQ[5] (ng/L)	LOD[6] (ng/L)	DL[7] (ng/L)
FTS 4:2	757124-72-4	DNE	NA[4]	DNE	4.0	4.0	2.0	0.14
FTS 6:2	27619-97-2	DNE	NA	DNE	4.0	4.0	2.0	0.16
FTS 8:2	39108-34-4	DNE	NA	DNE	4.0	4.0	2.0	0.04
N-MEFOSA	31506-32-8	DNE	NA	DNE	4.0	4.0	2.0	0.22

Analyte	CAS[1] Number	Project Screening Level (PSL) (ng/L)[2]	PSL Reference	EPA RSLs[8] (ng/L)	Project Quantification Limit Goal (ng/L)	Laboratory-specific		
						LOQ[5] (ng/L)	LOD[6] (ng/L)	DL[7] (ng/L)
N-ETFOSAA	2991-50-6	DNE	NA	DNE	1.0	1.0	0.5	0.27
N-MEFOSAA	2355-31-9	DNE	NA	DNE	1.0	1.0	0.5	0.28
PFBA	375-22-4	DNE	NA	DNE	4.0	4.0	2.0	0.15
PFBS	375-73-5	500	SWRCB, 2021	600	1.0	1.0	0.5	0.07
PFDA	335-76-2	DNE	NA	DNE	1.0	1.0	0.5	0.22
PFDOA	307-55-1	DNE	NA	DNE	1.0	1.0	0.5	0.19
PFDS	335-77-3	DNE	NA	DNE	1.0	1.0	0.5	0.32
PFHPA	375-85-9	DNE	NA	DNE	1.0	1.0	0.5	0.08
PFHPS	375-92-8	DNE	NA	DNE	1.0	1.0	0.5	0.12
PFHXA	307-24-4	DNE	NA	DNE	1.0	1.0	0.5	0.13
PFHXS	355-46-4	39	Tap Water RSL - EPA, 2022	39	1.0	1.0	0.5	0.04
PFNA	375-95-1	5.9	Tap Water RSL - EPA, 2022	5.9	1.0	1.0	0.5	0.20
PFNS	98789-57-2	DNE	NA	DNE	1.0	1.0	0.5	0.26
PFOA	335-67-1	5.1	SWRCB, 2020b	6	1.0	1.0	0.5	0.33
PFOS	1763-23-1	6.5	SWRCB, 2020b	4	1.0	1.0	0.5	0.13
PFOSA	754-91-6	DNE	NA	DNE	4.0	4.0	2.0	0.22
PFPEA	2706-90-3	DNE	NA	DNE	2.0	2.0	1.0	0.10
PFPES	630402-22-1	DNE	NA	DNE	1.0	1.0	0.5	0.11
PFTEDA	376-06-7	DNE	NA	DNE	1.0	1.0	0.5	0.36
PFTRDA	72629-94-8	DNE	NA	DNE	1.0	1.0	0.5	0.43
PFUDA	2058-94-8	DNE	NA	DNE	1.0	1.0	0.5	0.37
HFPO-DA	13252-13-6	6	Tap Water RSL - EPA, 2022	6	2.0	2.0	1.0	0.18

Analyte	CAS[1] Number	Project Screening Level (PSL) (ng/L)[2]	PSL Reference	EPA RSLs[8] (ng/L)	Project Quantification Limit Goal (ng/L)	Laboratory-specific		
						LOQ[5] (ng/L)	LOD[6] (ng/L)	DL[7] (ng/L)
11-Cl-PF3OUdS	763051-92-9	DNE	NA	DNE	2.0	2.0	1.0	0.16
9-Cl-PF3ONS	756426-58-1	DNE	NA	DNE	2.0	2.0	1.0	0.08
ADONA	919005-14-4	DNE	NA	DNE	2.0	2.0	1.0	0.21
N-ETFOSA	4151-50-2	DNE	NA	DNE	8.0	8.0	6.0	3.0
N-ETFOSE	1691-99-2	DNE	NA	DNE	8.0	8.0	6.0	3.0
N-MEFOSA	31506-32-8	DNE	NA	DNE	8.0	8.0	6.0	3.0
N-MEFOSE	24448-09-7	DNE	NA	DNE	8.0	8.0	6.0	3.0

[1]CAS = Chemical Abstract Service [2]DNE = Does not exist [3]RSL = Regional Screening Level [4]NA =Not Applicable
[5]LOQ = Level of Quantitation [6]LOD = Limit of Detection [7]DL = Detection Limit [8]TR=1E-06, HQ=0.1, November, 2021.

The soil PSLs are based on the EPA Residential Soil Regional Screening Level (RSL) updated May 2022 (EPA, 2022), and the soil PSLs for PFOA. The summary table and results of the calculations are included in Attachment D. These were selected as the most conservative PSLs. PSLs have not been established for the remainder of the PFAS compounds; however, the regulatory status and analytical methods associated with PFAS compounds is evolving rapidly. Presence or absence of the remaining compounds will be determined by the current ability of analytical methods to detect such compounds.

Matrix: Soil
Analytical Group: PFAS (LC/MS/MS compliant with DoD QSM 5.3 Table B-15)
Concentration Level: Low

Analyte	CAS[1] Number	Project Screening Level (PSL) (ug/kg)[2]	PSL Reference[4]	Project Quantitation Limit Goal (ug/kg)	Laboratory-specific		
					LOQ[5] (ug/kg)	LOD[6] (ug/kg)	DL[7] (ug/kg)
FTS 4:2	757124-72-4	DNE	NA	1.0	1.0	0.40	0.20
FTS 6:2	27619-97-2	DNE	NA	1.0	1.0	0.40	0.20
FTS 8:2	39108-34-4	DNE	NA	1.0	1.0	0.40	0.15
N-MEFOSA	31506-32-8	DNE	NA	1.0	1.0	0.90	0.49
N-ETFOSAA	2991-50-6	DNE	NA	1.0	1.0	0.40	0.20
N-MEFOSAA	2355-31-9	DNE	NA	1.0	1.0	0.40	0.20
PFBA	375-22-4	DNE	NA	1.0	1.0	0.40	0.10
PFBS	375-73-5	1,900	Residential Soil RSL[3] EPA, 2022	1.0	1.0	0.40	0.10
PFDA	335-76-2	DNE	NA	1.0	1.0	0.40	0.15
PFDOA	307-55-1	DNE	NA	1.0	1.0	0.40	0.15
PFDS	335-77-3	DNE	NA	1.0	1.0	0.40	0.20
PFHPA	375-85-9	DNE	NA	1.0	1.0	0.40	0.10
PFHPS	375-92-8	DNE	NA	1.0	1.0	0.40	0.15
PFHXA	307-24-4	DNE	NA	1.0	1.0	0.40	0.10

Analyte	CAS[1] Number	Project Screening Level (PSL) (ug/kg)[2]	PSL Reference[4]	Project Quantitation Limit Goal (ug/kg)	Laboratory-specific		
					LOQ[5] (ug/kg)	LOD[6] (ug/kg)	DL[7] (ug/kg)
PFHXS	355-46-4	130	Residential Soil RSL[3] EPA, 2022	1.0	1.0	0.40	0.15
PFNA	375-95-1	19	Residential Soil RSL[3] EPA, 2022	1.0	1.0	0.40	0.10
PFNS	98789-57-2	DNE	NA	1.0	1.0	0.40	0.10
PFOA	335-67-1	19	Residential RSL[3] EPA, 2022b	1.0	1.0	0.40	0.15
PFOS	1763-23-1	13	Residential RSL[3] EPA, 2022b	1.0	1.0	0.40	0.10
PFOSA	754-91-6	DNE	NA	1.0	1.0	0.40	0.10
PFPEA	2706-90-3	DNE	NA	1.0	1.0	0.40	0.15
PFPES	630402-22-1	DNE	NA	1.0	1.0	0.40	0.10
PFTEDA	376-06-7	DNE	NA	1.0	1.0	0.40	0.20
PFTRDA	72629-94-8	DNE	NA	1.0	1.0	0.40	0.10
PFUDA	2058-94-8	DNE	NA	1.0	1.0	0.40	0.10
HFPO-DA	13252-13-6	23	Residential Soil RSL[3] EPA, 2022	1.0	1.0	0.40	0.20
11-Cl-PF3OUdS	763051-92-9	DNE	NA	1.0	1.0	0.40	0.20
9-Cl-PF3ONS	756426-58-1	DNE	NA	1.0	1.0	0.40	0.20
ADONA	919005-14-4	DNE	NA	1.0	1.0	0.40	0.20
N-ETFOSA	4151-50-2	DNE	NA	1.0	1.0	0.40	0.20
N-ETFOSE	1691-99-2	DNE	NA	1.0	1.0	0.40	0.20
N-MEFOSA	31506-32-8	DNE	NA	1.0	1.0	0.40	0.20
N-MEFOSE	24448-09-7	DNE	NA	1.0	1.0	0.40	0.20

[1]CAS = Chemical Abstract Service
[2]DNE = Does not exist
[3]RSL = Regional Screening Level
[4]NA = Not applicable
[5]LOQ = Level of Quantitation
[6]LOD = Limit of Detection
[7]DL = Detection Limit

QAPP Worksheet #17: Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.1)

This worksheet describes the sampling design and the basis for its selection. The sampling design type for this study is a judgmental design because the goal of this SI is to assess presence or absence of PFAS in soil and groundwater associated with potential releases in AOPC 1 through AOPC 5 to refine the CSM, but it is not intended to support statistical conclusions. Refer to Work Plan Section 4 for additional information. Details of sampling design and rationale are as follows:

1. The physical boundaries for the area under study (include maps or diagrams).	See Work Plan Section 4.3 and Figures 5 and 6.
2. The time period being represented by the collected data.	The time period being represented is current and future groundwater and soil conditions representative of potential historical PFAS releases at the site associated with AOPC1 through AOPC5. The exact date of the potential release at AOPC1 is not known. The waste and seepage pits associated with AOPC 2 and AOPC 3 were in use from approximately 1945 to 1960. Potential use of PFAS-containing compounds associated with chrome plating operations would have been most likely between 1988 and 1994. Potential use of PFAS-containing compounds associated with the photo labs would most likely have been between the late 1960s and early 2000s.
3. The descriptions and basis for dividing the site into sampling areas (e.g., decision units, exposure units, etc.) that support the decision statements documented on Worksheet #11.	The site has been divided into five AOPCs based on a PFAS Preliminary Assessment Report performed in 2021. See Work Plan Section 2 for a description of each area and Work Plan Figure 2 depicting structures associated with each of the AOPCs.
4. The basis for the number and placement of samples within sampling areas.	See Work Plan Section 4.3 and Table 2 for the basis for the number and placement of samples within each sampling area. Summarizing: • The approach to sampling groundwater in association with AOPC 1 will be to sample two wells (MW-4 and MW-16) at various depth intervals and analyze samples for PFAS. Locations coincide with the stormwater conveyance from the potential release (MW-4) and downgradient of the downstream stormwater outfall (MW-16). The number and placement of samples are sufficient to determine if PFAS has impacted groundwater at concentrations greater than the PSLs as a result of activities at AOPC 1. • The approach to soil sampling is to sample surface soils from six locations at two depths (0-0.5 and 0.5-2.0 ft bgs) adjacent to the helipad and, in the depression below the helipad in AOPC 1. The number and placement of samples is sufficient to detect

	if PFAS impacted soil is present due to AFFF storage in Building 242A and the one-time training exercise at the helipad. • The approach to groundwater sampling in association with AOPCs 2 and 3 is to sample six existing monitoring wells (MW-4, MW-16, MW-12, MW-15, MW-17, and MW-24) at various depth intervals and analyze samples for PFAS. Locations include areas downgradient of waste and seepage pits, downgradient of the Site, as well as a historical source area associated with the JPL Superfund Site. The number and placement of samples are sufficient to determine if PFAS has impacted groundwater at concentrations greater than the PSLs as a result of activities at AOPCs 2 and 3. • The approach to groundwater sampling associated with AOPC 4 will be to sample one well (MW-4) at various depth intervals and analyze samples for PFAS. The selected location is downgradient of the building associated with former chrome plating operations. The number and placement of samples are sufficient to determine if PFAS has impacted groundwater at concentrations greater than the PSLs as a result of activities at AOPC 4. • The approach to groundwater sampling associated with AOPC 5 will be to sample one well (MW-5) and analyze samples for PFAS. The selected location is downgradient of Former Building 218 and Building 291. The number and placement of samples are sufficient to determine if PFAS has impacted groundwater at concentrations greater than the PSLs as a result of activities at AOPC 5.
5. If sample locations are specified in the QAPP, descriptions of how actual sample positions will be located once in the field. (Include maps or diagrams).	Proposed soil sampling locations are shown in Figure 5. Exact locations will be as close as possible to the proposed locations; however, final locations will be based on an on-site evaluation of physical features and the ability to access proposed locations. All groundwater samples will be collected from existing wells as shown in Figure 6.
6. If a sample cannot be collected where planned, the decision process for changing the location.	The decision process for changing the location of a soil sample will involve the site supervisor coordinating with the project manager to ensure the alternative proposed location meets the sampling objectives of the initial location. If for some reason a sample cannot be collected from an existing monitoring well, the site supervisor will coordinate with the project manager to determine there is an alternate location that can meet the sampling objectives of the initial location or if the remainder of the proposed locations will satisfy sampling objectives. The project manager will communicate any field changes consistent with WS#6.
7. If sample locations will be determined in the field, the decision process for doing so.	General locations for soil sampling have been established, and the exact location will be selected based on professional judgement of the onsite supervisor. Groundwater samples will be collected from existing wells.

8. Contingencies in the event field conditions are different than expected and could have an effect on the sample design.	Field conditions are not expected to have a significant impact on sample design since groundwater samples will be collected from existing wells and soil sample collection locations are limited. In the event that field conditions require, professional judgement will be used to refine sample design to overcome field conditions such that presence or absence of soil and groundwater contamination at the Site can still be assessed.

QAPP Worksheet #18: Sampling Locations and Methods
(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.1 and 2.3.2)

The following table provides the list of individual samples to be collected, the planned analytical methods and sampling Standard Operating Procedures (SOPs).

See Work Plan Figures 5 and 6 for locations.

Sampling Location / ID Number	Matrix	Depth (ft bgs)	Type	Analyte/ Analytical Group	Sampling SOP Reference	Comments
MW–4 / MW-4-S1-MMDDYY	GW	147 – 157	Grab	PFAS	SOP No. 2 – Groundwater Sampling (PFAS Specific)	AOPC 1 – 4: Downgradient of multiple AOPC
MW-4 / MW-4-S2-MMDDYY	GW	237 – 247	Grab			
MW-4 / MW-4-S4-MMDDYY	GW	389 – 399	Grab			
MW-4 / MW-4-S5-MMDDYY	GW	509 – 519	Grab			
MW-5 / MW-5-MMDDYY	GW	85 – 135	Grab			AOPC 5: Downgradient
MW–12 / MW-12-S1-MMDDYY	GW	135 – 145	Grab			AOPC 2 – 3: Downgradient of waste pits and seepage pits at JPL
MW–12 / MW-12-S2-MMDDYY	GW	240 – 250	Grab			
MW–12 / MW-12-S3-MMDDYY	GW	315 – 325	Grab			
MW–12 / MW-12-S4-MMDDYY	GW	430 – 440	Grab			
MW–12 / MW-12-S5-MMDDYY	GW	546 – 556	Grab			
MW–15 / MW-15-MMDDYY	GW	19 – 69	Grab			
MW–16 / MW-16-MMDDYY	GW	230 – 280	Grab			
MW-17 / MW-17-S1-MMDDYY	GW	246 – 256	Grab			
MW-17 / MW-17-S2-MMDDYY	GW	336 – 376	Grab			
MW-17 / MW-17-S3-MMDDYY	GW	466 – 476	Grab			
MW-17 / MW-17-S4-MMDDYY	GW	578 – 588	Grab			
MW-17 / MW-17-S5-MMDDYY	GW	723 – 733	Grab			
MW-24 / MW-24-S1-MMDDYY	GW	275 – 285	Grab			
MW-24 / MW-24-S2-MMDDYY	GW	370 –380	Grab			
MW-24 / MW-24-S3-MMDDYY	GW	430 – 440	Grab			
MW-24 / MW-24-S4-MMDDYY	GW	550 – 560	Grab			
MW-24 / MW-24-S5-MMDDYY	GW	675 – 685	Grab			

Sampling Location / ID Number	Matrix	Depth (ft bgs)	Type	Analyte/ Analytical Group	Sampling SOP Reference	Comments
Field Duplicate 1 (DUP-1-MMDDYY)	GW	Same as primary sample	Grab			QA/QC
Field Duplicate 2 (DUP-2-MMDDYY)	GW	Same as primary sample	Grab			QA/QC
Lab MS/MSD	GW	Same as primary sample	Grab			QA/QC
SB-1 / SB-1-0.5-MMDDYY	Soil	0 – 0.5	Grab		SOP No. 5 – Soil Sampling for Chemical Analysis (PFAS Specific)	AOPC 1 – Locations at former AFFF storage site and training exercise
SB-1 / SB-1-1.5-MMDDYY	Soil	0.5 – 2.0	Grab			
SB-2 / SB-2-0.5-MMDDYY	Soil	0 – 0.5	Grab			
SB-2 / SB-2-1.5-MMDDYY	Soil	0.5 – 2.0	Grab			
SB-3 / SB-3-0.5-MMDDYY	Soil	0 – 0.5	Grab			
SB-3 / SB-3-1.5-MMDDYY	Soil	0.5 – 2.0	Grab			
SB-4 / SB-4-0.5-MMDDYY	Soil	0 – 0.5	Grab			AOPC 1 – Locations downgradient of former AFFF storage, representing transport from site during rain event
SB-4 / SB-4-1.5-MMDDYY	Soil	0.5 – 2.0	Grab			
SB-5 / SB-5-0.5-MMDDYY	Soil	0 – 0.5	Grab			
SB-5 / SB-5-1.5-MMDDYY	Soil	0.5 – 2.0	Grab			
SB-6 / SB-6-0.5-MMDDYY	Soil	0 – 0.5	Grab			
SB-6 / SB-6-1.5-MMDDYY	Soil	0.5 – 2.0	Grab			
Equipment Blank – GW Sampling (EQP-1)	W	NA	Grab		SOP No. 3 - Sample Handling and Management (PFAS Specific)	QA/QC
Equipment Blank – GW Sampling (EQP-2)	W	NA	Grab			QA/QC
Field Blank-1 (1 per day of sampling)	W	NA	Grab			QA/QC

GW = Groundwater
NA = Not applicable
MS/MSD = Matrix Spike/Matrix Spike Duplicate
W = Water

QAPP Worksheet #19 & 30: Sample Containers, Preservation, & Hold Times
(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.2)

Matrix	**Analytical Group**	**Analytical and Preparation Method / SOP Reference**	**Containers** (number, size, and type)	**Sample volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time** (preparation / analysis)
Soil	PFAS	Preparation Method/SOP: EPA 537-mod / DoD QSM 5.3 Table B-15 / PRE537 Analysis Method/SOP: EPA 537-mod, DoD QSM 5.3 Table B-15 / HPL537 APPL's DoD/DoE Certificate, expiration date 06/30/2023	1 x 50 mL HDPE (HDPE Bottle and Cap; will not contain Teflon or other potential contaminating chemicals)	1g	Cool to < 6°C	Extract within 28 days of collection/ Analyze within 28 days of extraction
Water	PFAS	Preparation Method/SOP: EPA 537-mod / DoD QSM 5.3 Table B-15 / PRE537 Analysis Method/SOP: EPA 537-mod, DoD QSM 5.3 Table B-15 / HPL537 APPL's DoD/DoE Certificate, expiration date 06/30/2023	2 x 250mL HDPE (Non-potable Water) (HDPE Bottle and Cap; will not contain Teflon or other potential contaminating chemicals)	250mL	Cool to < 6°C	Extract within 28 days of collection/ Analyze within 28 days of extraction (Non-potable Water)

QAPP Worksheet #20: Field Quality Control Sample Summary – Site Characterization
(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.5)

The following table provides a summary of the types of samples to be collected and analyzed for the project.

Matrix	Analytical Group	Field Samples	Field Duplicates	Matrix Spike/ Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trip Blanks	Other	Total # of Analyses
GW	PFAS	22	3	1	1 per day	1	0	SB[1] TBD	28 (+SB)
Soil	PFAS	12	2	1	0	1	0	0	16

SB = Source Blank

[1] If water for equipment blanks, field blanks, and sampling equipment is provided by an on-site source rather than the laboratory, the source will be sampled for PFAS by LC/MS/MS compliant with QSM Table B-15 prior to use to confirm lack of detectable PFAS compounds.

QAPP Worksheet #21: Field SOPs
(UFP-QAPP Manual Section 3.1.2)
(EPA 2106-G-05 Section 2.3.2)

The following table documents the specific field procedures being implemented.

Field SOPs and field instrument manuals are provided in Attachment B.

Reference Number	**Title, Revision Date and / or Number**	**Originating Organization**	**Equipment Type (if SOP provides multiple options)**	**Modified for Project Work?** (Y/N)	**Comments**
SOP No. 1	Water Level Measurement (PFAS Specific), Revised 11/01/21, Rev. 0.0	G2S	N/A	Y	Modified for PFAS sampling requirements
SOP No. 2	Groundwater Sampling (PFAS Specific), Revised 11/03/21, Rev. 0.0	G2S	N/A	Y	Modified for PFAS sampling requirements
SOP No. 3	Sample Handling and Management (PFAS Specific), Revised 11/01/21, Rev. 0.0	G2S	N/A	Y	Modified for PFAS sampling requirements
SOP No. 4	Sampling Equipment Decontamination (PFAS Specific), Revised 11/01/21, Rev. 0.0	G2S	N/A	Y	Modified for PFAS sampling requirements
SOP No. 5	Soil Sampling for Chemical Analysis (PFAS Specific), Revised 11/01/21, Rev. 0.0	G2S	N/A	Y	Modified for PFAS sampling requirements
SOP No. 6	Investigation Derived Waste (IDW) Management (PFAS Specific), Revised 10/12/22, Rev. 1.0	G2S	N/A	Y	Analytical suite for IDW modified for local disposal characterization requirements.

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.1.2.4)
(EPA 2106-G-05 Section 2.3.6)

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
Horiba U-50 Multi-Parameter Water Quality Checker	Calibration	Manufacturer's instructions	Site Supervisor	Daily before use	Autocalibration with confirmation using transparent and black cup	Re-calibrate or clean probes
	Maintenance			As needed	NA	NA
Eurotech TN-100/T-100 Portable Turbidity Meter	Calibration	Manufacturer's instructions	Site Supervisor	Weekly	Calibration confirmation with four-point turbidity standards	Re-calibrate if as necessary
	Maintenance			As needed	NA	NA
Solinst Water Level Meter	Maintenance	Manufacturer's instructions	Site Supervisor	As needed	NA	NA
Honeywell PID	Calibration	Manufacturer's instructions	Site Supervisor	As indicated by instrument display	Two of three-point calibration	Re-calibrate or clean probes
	Maintenance			As needed	NA	NA
Redi-Flo 2 Submersible Pump with GeotechVFD	Maintenance	Manufacturer's instructions	Site Supervisor	As needed	NA	NA
Trimble GeoXH 6000 handheld unit (or equivalent)	Maintenance (accuracy < 1 meter, real time)	Manufacturer's instructions	Site Supervisor	As needed	NA	NA
Extech 407750 Digital Sound Level Meter	Calibration	Manufacturer's instructions	Site Supervisor	As indicated by instrument display	External calibrator 1kHz sine at 94.0dB, reading 94.0dB	
	Maintenance			As needed	NA	NA

QAPP Worksheet #23: Analytical SOP References Table
(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
Preparatory Methods						
PRE537	SPE for Aqueous and Solid PFAS Samples EPA 537.1 and 537M DoD QSM Table B-15, Rev. 8, 08/24/22	Definitive	Water: PFAS	NA	APPL Inc., Clovis, CA	N
Analytical Methods						
HPL537	Instrumental analysis of PFAS by Sciex TQ5500 LC-MS/MS (EPA 537.1 and 537M DoD QSM Table B-15), Rev 9, 08/31/22	Definitive	Soil and Water: PFAS	LC-MS-MS	APPL Inc., Clovis, CA	N

QAPP Worksheet #24: Analytical Instrument Calibration
(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS-MS	Mass Calibration	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. Unit resolution is demonstrated when the value of the peak width at half-height is within 0.5 ± 0.1 amu.	If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance.	Analyst or certified instrument technician	HPL537
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 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 the retention times. Quantitate samples by integrating the total response accounting for all peaks using the ICAL linear isomer for quantitation.	Quantitate samples by integrating the total response accounting for all peaks using the ICAL linear isomer for quantitation.	Analyst or certified instrument technician	HPL537

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS-MS	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). 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 each analyte must be < 20%. Option 2: Linear or non-linear calibrations must have r^2>0.99 for each analyte	Correct problem, then repeat ICAL. 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) or quadratic (minimum of 6 standards); weighting is allowed.	Analyst or certified instrument technician	HPL537
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 CCV is used.	NA	Analyst or certified instrument technician	HPL537

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS-MS	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.	Analyst or certified instrument technician	HPL537
LC-MS-MS	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.	Analyst or certified instrument technician	HPL537
LC-MS-MS	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 that fails, repeat ICAL.	Analyst or certified instrument technician	HPL537

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS-MS	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.	Analyst or certified instrument technician	HPL537
LC-MS-MS	Mass Spectral Acquisition Rate	Each analyte, Extracted Internal Standard (EIS) Analyte	A minimum of 10 spectra scans are acquired across each chromatographic peak.	NA	Analyst or certified instrument technician	HPL537

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS-MS	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. Instrument Sensitivity Check (ISC) can serve as a bracketing CCV.	Analyst or certified instrument technician	HPL537

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, testing and Inspection
(UFP-QAPP Manual Section 3.2.3)
(EPA 2106-G-05 Section 2.3.6)

Instrument Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference
LC-MS-MS	Change guard cartridge	N/A	Review pressure profile	As needed, based on pressure profile and chromatography	CCV	Replace with new guard cartridge	Analyst or certified instrument technician	HPL537
LC-MS-MS	Change analytical column	N/A	Check peak tailing, decreased sensitivity, retention time changes, etc.	When chromatography indicates	Passing ICAL	Replace with another analytical column	Analyst or certified instrument technician	HPL537
LC-MS-MS	Check and replace mobile phase as needed	N/A	N/A	As needed	N/A	Prepare fresh mobile phase solution	Analyst or certified instrument technician	HPL537
LC-MS-MS	Tune MS	N/A	Monitor detector response. Warning flags indicating a decrease in data quality include: a decreased detector response, elevated baseline or calibration inconsistencies	Yearly	Peak widths within 0.5 +/- 0.1Da	A service call should be placed with the manufacturer	Analyst or certified instrument technician	HPL537
LC-MS-MS	Clean Source	Inspect source	Inspect curtain plate. If significant residue is present proceed with cleaning procedures.	Daily	N/A	N/A	Analyst or certified instrument technician	HPL537

QAPP Worksheet #26 & #27: Sample Handling, Custody, and Disposal
(UFP-QAPP Manual Section 3.3)
(EPA 2106-G-05 Section 2.3.3)

Responsibility for maintaining custody of samples from sample collection through disposal is as follows:

Sampling Organization: G2S LLC
Laboratory: APPL Laboratory
Method of sample delivery (shipper/carrier): Courier
Number of days from reporting until sample disposal: 30 days

Activity	Organization and title or position of person responsible for the activity	SOP reference
Sample labeling and chain of custody form completion	G2S LLC – David Conner, Site Superintendent	SOP No. 3 Sample Handling and Management (PFAS Specific)
Packaging and courier coordination	G2S LLC – David Conner, Site Superintendent	
Sample receipt, inspection, & log-in	Megan Salata, APPL, Inc.	APPL SHR001
Sample custody and storage	Megan Salata, APPL, Inc.	APPL SHR001
Sample Disposal	Megan Salata, APPL, Inc.	APPL SHR012

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix	Water and Soil
Analytical Group	PFAS
Analytical Method / SOP Reference	EPA Method EPA 537M/QSM 5.3 Table B-15

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
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). Known high PFAS concentration samples will be performed by serial dilution instead of SPE.	NA.	Analyst Lab QA Officer Project Chemist	NA.	NA.
Solid Sample Preparation	Each sample and associated batch QC samples.	Entire sample received by the laboratory must be homogenized prior to subsampling.	NA.	Analyst Lab QA Officer Project Chemist	NA.	NA.
Instrument Blanks	Immediately following the highest standard analyzed and daily prior to sample analysis.	Concentration of each analyte must be ≤ ½ the LOQ.	Re-analyze samples if concentrations are >1/2 LOQ.	Analyst Lab QA Officer Project Chemist	Accuracy / Sensitivity	QC acceptance criteria specified by DoD QSM v5.3

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
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.	Analyst Lab QA Officer Project Chemist	Accuracy/ Sensitivity	QC acceptance criteria specified by DoD QSM v5.3 Apply B-flag to all results for specific analytes in all samples in the associated preparatory batch.
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.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision - Analytical	QC acceptance criteria specified by DoD QSM v5.3

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
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 Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst Lab QA Officer Project Chemist	Precision – Analytical	For matrix evaluation, use QC acceptance criteria specified by DoD QSM v5.3
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.	Analyst Lab QA Officer Project Chemist	Precision – Analytical	For matrix evaluation, use QC acceptance criteria specified by DoD QSM v5.3

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Extracted Internal Standard Analytes (EIS)	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.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria specified by DoD QSM v5.3
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.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria specified by DoD QSM v5.3

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Sample PFAS Identification	Identify all positive sample detections per method.	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. Quant ion and confirmation ion must be present and must maximize simultaneously (±2 seconds).	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".	Analyst Lab QA Officer Project Chemist	Precision	Acceptance criterion are set to 50-150 and are compared to both the response of the midpoint of the calibration mid point, and the opening CCV.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
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	Analyst Lab QA Officer Project Chemist	Accuracy	NA

QAPP Worksheet #29: Project Documents and Records
(UFP-QAPP Manual Section 3.5.1)
(EPA 2106-G-05 Section 2.2.8)

Information for all documents and records that will be generated for the project are as follows:

Sample Collection and Field Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Field logbook	Site Supervisor – David Conner	Project Engineer	Project file
Chain-of-Custody Records	Site Supervisor – David Conner	Project Engineer	Project file
Courier Records	Site Supervisor – David Conner	Project Engineer	Project file
Contractor Production Reports	Site Supervisor – David Conner	Project Engineer	Project file
Photographs	Site Supervisor – David Conner	Project Engineer	Project file
Deviations	Site Supervisor – David Conner	Project Manager – Keith Fields	Project file
Correspondence	Site Supervisor – David Conner	Project Manager – Keith Fields	Project file
Corrective Action Reports	Site Supervisor – David Conner	Project Manager – Keith Fields	Project file
Sampling instrument calibration logs	Site Supervisor – David Conner	Project Engineer	Project file
Project Assessments			
Record	**Generation**	**Verification**	**Storage location/archival**
Field audit checklists	Site Supervisor – David Conner	Project Manager – Keith Fields	Project file
Data Verification Checklists	LDC, Inc.	Project Chemist	Project file
Data Validation Report	LDC, Inc.	Project Chemist	Project file
Data Usability Report	Project Chemist	Project Manager – Keith Fields	
Laboratory Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Analytical Report Package	APPL, Inc.	Project Chemist	Project file

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action
(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)
(EPA 2106-G-05 Section 2.4 and 2.5.5)

Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Field Sampling Technical Systems Audit	Site Supervisor G2S LLC	At start of sampling As required after	TBD	Written Field Audit as needed	10 days after receiving notification
DoD Environmental Laboratory Accreditation Program	Laboratory Validation Coordinator	Every 2 years	June 2023	NA	NA
Chemistry Data Audit	Project Geologist G2S LLC	Periodically	5 days after audit	Written Audit as needed	14 days after receiving notification
Data Validation System Audit	Project Manager G2S LLC	Periodically	5 days after audit	Written Audit as needed	14 days after receiving notification
Verbal Status Reports	Site Supervisor G2S LLC	Daily/Weekly	Daily/weekly conference calls	NA	NA
Technical Meetings or Teleconferences	Project Manager G2S LLC	Monthly during Field Work, or as needed	Monthly during Field Work, or as needed	Meeting minutes	10 days after meeting
Email status Reports	Project Manager G2S LLC	Monthly	Monthly	Email status report	Monthly

Assessment Response and Corrective Action:

Assessment Type	Responsibility for responding to assessment findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation
Chemistry Field Sampling Technical Systems Audit	Site Superintendent G2S LLC	Corrective Action Plan	10 days after receiving notification	Site Superintendent G2S LLC	Project Chemist G2S LLC
DoD Environmental Laboratory Accreditation Program	QA Coordinator or Technical Operations Manager Contract Laboratory	Corrective Action Plan	14 days after receiving notification	QA Coordinator or Technical Operations Manager Contract Laboratory	DoD ELAP Coordinator
Chemistry Data Audit	QA Coordinator or Technical Operations Manager Contract Laboratory	Corrective Action Plan, Re-submission of data	14 days after receiving notification	QA Coordinator or Technical Operations Manager Contract Laboratory	Project Chemist G2S LLC
Data Validation System Audit	QA Coordinator or Technical Operations Manager Contract Laboratory	Corrective Action Plan, Re-submission of data	14 days after receiving notification	QA Coordinator or Technical Operations Manager Contract Laboratory	Project Manager G2S LLC
DoD Environmental Laboratory Accreditation Program	APPL, Inc.	As per Assessment	As per Assessment	APPL, Inc.	APPL Inc. / DOD 3rd Part Auditor

QAPP Worksheet #34: Data Verification and Validation Inputs
(UFP-QAPP Manual Section 5.2.1 and Table 9)
(EPA 2106-G-05 Section 2.5.1)

Item	Description	Verification (completeness)	Validation (conformance to specifications)
	Planning Documents/Records		
1	Approved QAPP	X	
2	Approved Accident Prevention Plan/ Site Safety and Health Plan (APP / SSHP)	X	
3	Field SOPs	X	
4	Laboratory SOPs	X	
	Field Records		
5	Field Logbooks	X	
6	Equipment Calibration Logs	X	
7	Chain-of-Custody records	X	
8	Sampling diagrams/notes	X	
9	Drilling logs	X	
10	Monitoring well construction forms	X	
11	Field Data Collection forms	X	
12	DQCRs	X	
	Analytical Data Package		
13	Cover Sheet	X	X
14	Case narrative	X	X
15	Internal laboratory Chain-of-custody	X	X
16	Sample Receipt records	X	X
17	Sample chronology	X	X
18	Sample results	X	X
19	LOD / LOQ Establishment and verification	X	X
20	Standards traceability	X	X
21	Instrument calibration records	X	X
22	Definition of laboratory qualifiers	X	X
23	QC sample results	X	X
24	Corrective Action reports	X	X
25	Raw data	X	X
26	Electronic data deliverable	X	X
27	Data Validation Report	X	X

QAPP Worksheet #35: Data Verification Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Records Reviewed	Required Documents	Process Description	Responsible Person, Organization
Field Logbook, Boring Logs, and Well Construction Forms, Field Data Collection Forms	QAPP, Field SOPs	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed, and results are documented.	Daily – David Conner At conclusion of field activities – Keith Fields
Chain-of-custody forms	QAPP, Field SOPs	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Daily – David Conner At conclusion of field activities - Project Chemist
Laboratory Deliverable	QAPP, Lab SOPs	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the chain-of-custody forms to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Before release - Laboratory Project Manager Upon receipt - Project Chemist

Records Reviewed	Required Documents	Process Description	Responsible Person, Organization
Audit Reports, Corrective Action Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan. Establish that all QAPP required QC samples were run and met the required limits for precision and accuracy. Verify that sample results met the project quantitation limits specified in the QAPP. Conduct data Level 2b validation on 90% of samples and Level 4 validation (i.e., review of raw data to confirm laboratory calculations) on 10% of samples.	LDC – Project Chemist

QAPP Worksheet #36: Data Validation Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Analytical Group	Data Deliverable requirements	% of data packages to be validated	% of raw data received	% of results to be calculated	Validation Procedure	Validation Code
PFAS (LC/MS/MS compliant with DoD QSM 5.3 Table B-15)	SEDD Stage 2b PDF Level 4 Laboratory Report	100%	10%	10%	90% Level 2b 10% Level 4	S2bVE S4VE

See Worksheets #23 and #28 for analytical specifications and Worksheet #12 for measurement performance criteria.

QAPP Worksheet #37: Data Usability Assessment
(UFP-QAPP Manual Section 5.2.3 including Table 12)
(EPA 2106-G-05 Section 2.5.2, 2.5.3, and 2.5.4)

This worksheet documents the procedures that will be used to perform the data usability assessment.

Personnel responsible for participating in the data usability assessment:
Project Manager
Project Chemist
Project Geologist
Project Engineer

The data usability assessment will be documented in the PFAS SI Report.

The Data Usability Assessment will be performed by G2S LLC using validated data. After the Data Usability Assessment has been performed, data deemed appropriate for use will then be used for its various purposes including to evaluate presence/absence of PFAS at the Site. The following items will be assessed, and conclusions drawn based on their results:

Precision – Precision is measured through analysis of field and laboratory QC samples, namely field duplicates and MSDs. Field sampling precision is evaluated by calculating the relative percent difference (RPD) for target compounds detected in the parent sample and it's duplicate. Target compounds detected in both the parent sample and its field duplicate equal to or above the quantitation limit will be presented in tabular format and the RPD calculated. The RPDs will be checked against the measurement performance criteria presented on Worksheet #12, and RPDs exceeding criteria will be identified on the tables. RPD is calculated by dividing the difference between the two sample results (e.g., parent sample and duplicate) by the average of the two sample results and multiplying by 100.

Laboratory precision is evaluated through calculation of RPDs for all target compounds spiked into a field-designated MS and MSD sample. Any outliers identified (i.e., those compounds with RPDs outside control limits) will be reviewed to determine the source of the error. Sources of error include matrix interference, instrument error, and/or sampling error. Any conclusions about the precision of the analyses will be drawn, and any limitations on the use of the data will be described.

Accuracy/Bias Contamination – Results for all laboratory method blanks and equipment blanks will be evaluated. The results for of any target compound detected will be checked against the measurement performance criteria presented on Worksheet #12. Results for target compounds that exceed criteria will be tabulated. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Accuracy is reported as percent error and evaluates how close a measurement is to the actual value. Percent error is calculated by dividing the difference between the measured value and the known/control value by the known/control value and multiplying by 100.

Overall Accuracy/Bias – Overall accuracy and bias will be evaluated through matrix spikes, surrogate recoveries, laboratory control samples (LCSs), and second source calibration verification. Outliers will be evaluated and tabulated, using acceptance criteria presented on Worksheet #12. A discussion will follow summarizing overall accuracy/bias and whether the source of the error is instrument, matrix, or sampling related. Conclusions about the overall accuracy/bias of the analyses will be drawn, and any limitations on the use of the data will be described.

Sensitivity – The results for target compounds will be checked against the measurement performance criteria presented on Worksheet #12 and cross-checked against the quantitation limits presented on Worksheet #15. Results for target compounds with elevated quantitation limits will be identified and noted. A discussion will follow summarizing the results of the laboratory sensitivity. Any conclusions about the sensitivity of the analyses will be drawn and any limitations on the use of the data will be described.

Representativeness – The groundwater samples required for this project will be collected using standardized procedures modified for PFAS specific sampling, and at selected locations designed to provide a true representation of groundwater conditions associated with the AOPCs. Standardized, accepted analytical methods will be used to ensure that accurate, reproducible data are generated. To verify sample representativeness, field sample collection procedures, sample containers, and holding times will be reviewed for SOP conformance. Non-representative samples will be identified, narrated, and the impact on data quality objectives discussed.

Completeness – A completeness check will be done on the total number of planned results. Completeness criteria are presented on Worksheet #12. Completeness will be calculated for each target compound as the number of data points collected for each compound, divided by the total number of data points planned for each compound. A discussion will follow summarizing the calculation of data completeness. Any conclusions about the completeness of the data for each analyte will be drawn and any limitations on the use of the data will be described.

Reconciliation – Each of the MPCs presented on Worksheet #12 will be examined to determine if the objective was met. This examination will include a combined overall assessment of the results of each analysis pertinent to an objective. Each analysis will first be evaluated separately in terms of the major impacts observed from the Data Validation, Data Quality Indicators, and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Based on the quality determined, the usability of the data for each analysis will be determined. Based on the combined usability of the data from all analyses for an objective, it will be determined if the MPC was met and whether project screening levels were exceeded. The final report will include a summary of all the points that went into the reconciliation of each objective. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described.

REFERENCES

Department of Defense (DoD), 2019. *DoD Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3.* May.

Intergovernmental Data Quality Task Force, 2012. *Uniform Federal Policy of Quality Assurance Project Plans, Optimized UFP-QAPP Worksheets*. March.

State Water Resources Control Board (SWRCB), 2019a. *Media Release: State Water Board Updates Guidelines for Testing and Reporting PFOA and PFOS As It Assesses Scope of Problem.* August 23.

SWRCB, 2019b. *Order WQ 2019-0045-DWQ - Determination of the Presence of Per- and Polyfluoroalkyl Substances at Chrome Plating Facilities.* October 11.

SWRCB, 2020. *Order WQ 2020-0015-DWQ - Determination of the Presence of Per- and Polyfluoroalkyl Substances at Publicly Owned Treatment Works.* July 9.

SWRCB, 2021. *Notification Level Issuance, perfluorobutane sulfonic acid (PFBS).* March 5.

SWRCB, 2020a. *Notification Level Issuance, perfluorooctanesulfonic acid (PFOS).* February 14.

SWRCB, 2020b. *Notification Level Issuance, perfluorooctanoic acid (PFOA).* February 12.

Tetra Tech, 2021. *Per-and Polyfluoroalkyl Substances Preliminary Assessment Report for Jet Propulsion Laboratory prepared by Tetra Tech for NASA.* February.

United States Environmental Protection Agency (EPA), 2019. *Interim Recommendations to Address Groundwater Contaminated with Perfluorooctanoic Acid and Perfluorooctane Sulfonate.* https://www.epa.gov/pfas/draftinterimrecommendations-addressing-groundwater-contaminated-pfoa-and-pfos. 19 December.

EPA, 2022. *Regional Screening Level (RSL) Summary Table (TR=1E-06 HQ=0.1).* May.

ATTACHMENT A

LABORATORY DoD ELAP CERTIFICATION
AND
STANDARD OPERATING PROCEDURES

Accredited Laboratory

A2LA has accredited

AGRICULTURE & PRIORITY POLLUTANTS LABORATORIES, INC. (APPL, INC.)

Clovis, CA

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 5.3 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 28th day of July 2021.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 4064.01
Valid to June 30, 2023

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

AGRICULTURE & PRIORITY POLLUTANTS LABORATORIES, INC. (APPL, INC.)
908 N. Temperance Ave.
Clovis, CA 93611
Diane Anderson Phone: 559-275-2175

ENVIRONMENTAL

Valid To: June 30, 2023 Certificate Number: 4064.01

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 andthe requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.3 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

High Resolution Gas Chromatography/Mass Spectrometry, ICP-OES, ICP-Mass Spectrometry, Atomic Absorption Spectrometry, Gas Chromatography/ECD/FID, Liquid Chromatography- Mass Spectrometry, High Performance Liquid Chromatography, Ion Chromatography, Titrimetry

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
Metals			
Aluminum	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Antimony	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Arsenic	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Barium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Beryllium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Boron	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Cadmium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Calcium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Chromium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B

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Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
Cobalt	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Copper	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Iron	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Lead	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Magnesium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Manganese	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Mercury	EPA 245.1 EPA 7470A	EPA 7470A	EPA 7471A/7471B
Molybdenum	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nickel	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Potassium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Selenium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Silver	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Sodium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Strontium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Thallium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Tin	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Titanium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Total Phosphorus	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Vanadium	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Zinc	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Pesticides/Herbicides/PCBs/TPH/Fumigants			
1,2,3-Trichloropropane	EPA 8011	----------	----------
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011	----------	----------
1,2-Dibromomethane (EDB, Ethylene dibromide)	EPA 8011	----------	----------
DRO (Diesel Range Organics), C10-C28	EPA 8015B/8015C/8015D	EPA 8015B/8015C/8015D	EPA 8015B/8015C/8015D

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
RRO (Residual Range Organics), C25-C36	EPA 8015B/8015C/8015D	EPA 8015B/8015C/8015D	EPA 8015B/8015C/8015D
DRO (Diesel Range Organics), C10-C25	AK102	AK102	AK102
RRO (Residual Range Organics), C25-C36	AK103	AK103	AK103
MRH (Mid-range Hydrocarbons), C9-C18	KS-MRH/HRH	KS-MRH/HRH	KS-MRH/HRH
HRH (High-range Hydrocarbons), C19-C35	KS-MRH/HRH	KS-MRH/HRH	KS-MRH/HRH
DRO (Diesel Range Organics)	WA-NWTPH-Dx	WA-NWTPH-Dx	WA-NWTPH-Dx
RRO (Residual Range Organics)	WA-NWTPH-Dx	WA-NWTPH-Dx	WA-NWTPH-Dx
GRO (Gasoline Range Organics), C6-C10	EPA 8015B/8015C/ 8015D	EPA 8015B/8015C/8015D	EPA 8015B/8015C/8015D
GRO (Gasoline Range Organics), C6-C10	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
GRO (Gasoline Range Organics), C6-C10	AK101-MS	AK101-MS	AK101-MS
LRH (Low-Range Hydrocarbons), C5-C8	KS-LRH	KS-LRH	KS-LRH
GRO (Gasoline Range Organics)	WA-NWTPH-Gx	WA-NWTPH-Gx	WA-NWTPH-Gx
Methane	RSK-175	RSK-175	----------
Ethane	RSK-175	RSK-175	----------
Ethene	RSK-175	RSK-175	----------
4,4'-DDD	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDT	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
a-BHC	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
a-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Aldrin	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
b-BHC	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
d-BHC	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Dieldrin	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan I	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan II	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan sulfate	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Endrin	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
g-BHC (Lindane)	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
g-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Methoxychlor	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Toxaphene	EPA 8081A/8081B	EPA 8081A/8081B	EPA 8081A/8081B
Aroclor-1016/1242	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
Aroclor-1016 (PCB-1016)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Aroclor-1221 (PCB-1221)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Aroclor-1232 (PCB-1232)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Aroclor-1242 (PCB-1242)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Aroclor-1248 (PCB-1248)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Aroclor-1254 (PCB-1254)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Aroclor-1260 (PCB-1260)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Aroclor-1262 (PCB-1262)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Aroclor-1268 (PCB-1268)	EPA 8082/8082A	EPA 8082/8082A	EPA 8082/8082A
Dalapon	----------	EPA 8151/8151A	----------
Dicamba	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
Dichlorprop (2,4-DP)	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
Dinoseb	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
2,4-D	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
2,4-DB	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
MCPA	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
MCPP	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
Pentachlorophenol	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
Silvex (2,4,5-TP)	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
2,4,5-T	EPA 8151/8151A	EPA 8151/8151A	EPA 8151/8151A
Purgeable Organics (Volatiles-VOC)			
1,1,1,2-Tetrachloroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,1-Trichloroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2,2-Tetrachloroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2-Trichloroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2-Trichlorotrifluoroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloroethene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloropropene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,3-Trichlorobenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,3-Trichloropropane	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM
1,2,4-Trichlorobenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,4-Trimethylbenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dibromo-3-chloropropane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dibromoethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichlorobenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichloroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichloropropane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3,5-Trimethylbenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3-Dichlorobenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3-Dichloropropane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,4-Dichlorobenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,4-Dioxane	EPA 8260B/8260C/8260D/ EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D/ EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D/ EPA 8260B/8260C/8260D SIM

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
2,2-Dichloropropane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Butanone (Methyl ethyl ketone)	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Chloroethyl vinyl ether	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Chlorotoluene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Hexanone	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
4-Chlorotoluene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
4-methyl-2-pentanone	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acetone	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acetonitrile	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acrolein	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acrylonitrile	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Benzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromobenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromochloromethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromodichloromethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromoform	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromomethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Carbon disulfide	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Carbon tetrachloride	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chlorobenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloroform	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloromethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
cis-1,2-Dichloroethene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
cis-1,3-Dichloropropene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Cyclohexane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dibromochloromethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dibromomethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dichlorodifluoromethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethyl benzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethyl tert-butyl ether (ETBE)	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Hexachlorobutadiene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Hexachloroethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Iodomethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Isopropyl ether (DIPE)	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Isopropylbenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
m+p-Xylene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methylacetate	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methylcyclohexane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methylene chloride (Dichloromethane)	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Naphthalene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
n-Butyl benzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
n-Propylbenzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
o-Xylene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
p-isopropyl toluene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
sec-Butyl benzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
Styrene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
tert-Amyl methyl ether (TAME)	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
tert-Butyl Alcohol (t-Butanol)	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
tert-Butyl Benzene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
tert-Butyl Ethyl Ether (ETBE)	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Tetrachloroethene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Toluene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Total Xylenes	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
trans-1,2-Dichloroethene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
trans-1,3-Dichloropropene	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Trichloroethene	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM
Trichlorofluoromethane	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Vinyl Acetate	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Vinyl Chloride	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM
Extractable Organics (Semivolatiles-SVOC)			
1,1'-Biphenyl	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
1-Methylnaphthalene	EPA 8270D/8270E SIM	EPA 8270D/8270E SIM	EPA 8270D/8270E SIM
1,2,4,5-Tetrachlorobenzene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
1,2,4-Trichlorbenzene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
1,2-Dichlorobenzene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
1,3-Dichlorobenzene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
1,4-Dichlorobenzene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
1,4-Dioxane	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM /8270E	EPA 8270D/8270E EPA 8270D/8270E SIM
2-Methylnaphthalene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
2-(2-Methoxyethoxy)-ethanol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,3,4,6-Tetrachlorophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,4,5-Trichlorophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,4,6-Trichlorophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,4-Dichlorophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,4-Dimethylphenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,4-Dinitrophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,6-Dichlorophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2-Chloronaphthalene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2-Chlorophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2-Methyl-4,6-Dinitrophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2-Methylnaphthalene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2-Methylphenol (o-Cresol)	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
2-Nitroaniline	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
2-Nitrophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
3, 4-Methylphenol (m+p-Cresol)	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
3,3'-Dichlorobenzidine	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
3-Nitroaniline	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
4-Bromophenyl phenyl ether	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
4-Chloro-3-methylphenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
4-Chloroaniline	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
4-Chlorophenyl phenylether	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
4-Methylphenol (p-Cresol)	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
4-Nitroaniline	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
4-Nitrophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Acenaphthene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Acenaphthylene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Acetophenone	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Aniline	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Anthracene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Atrazine	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Benzaldehyde	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Benzidine	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Benzo(a)anthracene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Benzo(a)pyrene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Benzo(b)fluoranthene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Benzo(b+k)fluoranthene	EPA 8270D/8270E SIM	EPA 8270D/8270E SIM	EPA 8270D/8270E SIM
Benzo(g,h,i)perylene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Benzo(k)fluoranthene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Benzoic acid	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Benzyl alcohol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Benzyl butyl phthalate	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Biphenyl	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
bis(2-Chloroethoxy) methane	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
bis(2-Chloroethyl) ether	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
bis(2-Chloroisopropyl) ether	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Butyl benzyl phthalate	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Caprolactam	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Carbazole	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Chrysene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Dibenz(a,h)anthracene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
Dibenzofuran	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Diethyl phthalate	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Dimethyl phthalate	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Di-n-butyl phthalate	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Di-n-octyl phthalate	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Fluoranthene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Fluorene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Hexachlorobenzene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Hexachlorobutadiene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Hexachlorocyclopentadiene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Hexachloroehane	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Indeno(1,2,3-cd) pyrene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Isophorone	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Naphthalene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Nitrobenzene	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
N-nitrosodimethylamine	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
N-nitrosodi-n-propylamine	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
N-nitrosodiphenylamine	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Pentachlorophenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Phenanthrene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Phenol	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Pyrene	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM	EPA 8270D/8270E EPA 8270D/8270E SIM
Pyridine	EPA 8270D/8270E	EPA 8270D/8270E	EPA 8270D/8270E
Dioxins/Furans			
1,2,3,4,6,7,8,9-OCDD	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,4,6,7,8,9-OCDF	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,4,6,7,8-Hpcdd	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,4,6,7,8-Hpcdf	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,4,7,8,9-Hpcdf	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,4,7,8-Hxcdd	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,4,7,8-Hxcdf	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,6,7,8-Hxcdd	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,6,7,8-Hxcdf	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,7,8,9-Hxcdd	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,7,8,9-Hxcdf	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,7,8-Pecdd	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
1,2,3,7,8-Pecdf	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
2,3,4,6,7,8-Hxcdf	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
2,3,4,7,8-Pecdf	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
2,3,7,8-TCDD	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
2,3,7,8-TCDF	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
Hpcdd, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
Hpcdf, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
Hxcdd, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
Hxcdf, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
PCDD + PCDF, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
PCDD, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
PCDF, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
Pecdd, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
Pecdf, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
TCDD, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A
TCDF, total	EPA 8290/8290A	EPA 8290/8290A	EPA 8290/8290A

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste		AFFF
		Aqueous	Solid/Chemical	
Per- & Polyfluoroalkyl Substances (PFAS)				
FTS 4:2	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
FTS 6:2	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
FTS 8:2	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
N-EtFOSAA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
N-MeFOSAA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFBA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFBS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFDA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFDoA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFDS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFHpA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFHpS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste		AFFF
		Aqueous	Solid/Chemical	
PFHxA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFHxS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFNA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFNS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFOA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFOS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFOSA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFPeA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFPeS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFTeDA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFTrDA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFUdA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
HFPO-DA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
9-Cl-PF30NS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
11-Cl-PF30UdS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
ADONA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
N-EtFOSE	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
N-EtFOSA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste		AFFF
		Aqueous	Solid/Chemical	
N-MeFOSE	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
N-MeFOSA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFDoS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
3;3 FTA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
5:3 FTA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
7:3 FTA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
10:2 FTS	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFMPA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFMBA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
NFDHA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15
PFEESA	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with DoD QSM 5.3 Table B-15

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
Energetics			
Nitroguanidine	EPA 8321A/8321B Mod.	EPA 8321A/8321B Mod.	EPA 8321A/8321B Mod.
Guanidine Nitrate	EPA 8321A/8321B Mod.	EPA 8321A/8321B Mod.	EPA 8321A/8321B Mod.
Perchlorate	EPA 6850	EPA 6850	EPA 6850
1,3,5-Trinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
1,3-Dinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
2,4,6-Trinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
2,4-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
2,6-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
2-Amino-4,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
2-Nitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
2-Amino-4,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
2-Nitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
3-Nitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
3,5 Dinitroaniline	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
4-Amino-2,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
4-Nitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
Nitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
Nitroglycerin	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
Pentaerythritoltetranitrate (PETN)	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
Tetryl (Methyl-2,4,6-trinitrophenylnitramine)	EPA 8330A/8330B	EPA 8330A/8330B	EPA 8330A/8330B
Picric Acid	EPA 8321A/8321B	EPA 8321A/8321B	EPA 8321A/8321B
Inorganics			
Ignitability	----------	----------	EPA 1030
pH / Corrosivity	EPA 9040C	EPA 9040C	EPA 9045C/D
Chromium VI	EPA 218.6/218.7	----------	----------
Chromium VI	EPA 7199	EPA 7199	EPA 7199
Chromium VI	----------	----------	EPA 7196A
Bromide	EPA 300.0	EPA 300.0	EPA 300.0
Chloride	EPA 300.0	EPA 300.0	EPA 300.0
Fluoride	EPA 300.0	EPA 300.0	EPA 300.0
Nitrate as N	EPA 300.0	EPA 300.0	EPA 300.0
Nitrite + Nitrate as N	EPA 300.0	EPA 300.0	EPA 300.0
Nitrite as N	EPA 300.0	EPA 300.0	EPA 300.0
Orthophosphate as P	EPA 300.0	EPA 300.0	EPA 300.0
Phosphate and Orthophosphate - as P	EPA 300.0	EPA 300.0	EPA 300.0
Sulfate	EPA 300.0	EPA 300.0	EPA 300.0
Bromide	EPA 9056/9056A	EPA 9056/9056A	EPA 9056/9056A
Chloride	EPA 9056/9056A	EPA 9056/9056A	EPA 9056/9056A
Fluoride	EPA 9056/9056A	EPA 9056/9056A	EPA 9056/9056A
Nitrate as N (NO_3^- as N)	EPA 9056/9056A	EPA 9056/9056A	EPA 9056/9056A
Nitrite + Nitrate as N	EPA 9056/9056A	EPA 9056/9056A	EPA 9056/9056A
Nitrite as N	EPA 9056/9056A	EPA 9056/9056A	EPA 9056/9056A
Sulfate	EPA 9056/9056A	EPA 9056/9056A	EPA 9056/9056A
Phosphate and Orthophosphate - as P	EPA 9056/9056A	EPA 9056/9056A	EPA 9056/9056A
Phosphate/Orthophosphate/Total Phosphorus	----------	SM 4500 PE	SM 4500 PE
Cyanide, Total and Amenable	EPA 9014 SM 4500CN B,C, G	EPA 9010C EPA 9014 SM 4500CN B,C,E,G	EPA 9010C EPA 9014 SM 4500CN B,C,E,G
Ammonia as N	EPA 350.1	EPA 350.1	EPA 350.1
Total Kheldahl Nitrogen	EPA 351.2	EPA 351.2	EPA 351.2

Parameter/Analyte	Non-potable Water	Solid Hazardous Waste	
		Aqueous	Solid/Chemical
Nitrate as N	EPA 353.2	EPA 353.2	EPA 353.2
Nitriate + Nitrate as N	EPA 353.2	EPA 353.2	EPA 353.2
Nitrite as N	EPA 353.2	EPA 353.2	EPA 353.2
Nitrocellulose	EPA 353.2 Mod	EPA 353.2 Mod	EPA 353.2 Mod
Bicarbonate	SM 2320B	SM 2320B	SM 2320B
Carbonate	SM 2320B	SM 2320B	SM 2320B
Hydroxide	SM 2320B	SM 2320B	SM 2320B
Total Alkalinity (CaCO3)	SM 2320B	SM 2320B	SM 2320B
Specific Conductance, Conductivity (25°C)	SM 2510B	SM 2510B	SM 2510B
Total Dissolved Solids (TDS)	EPA 160.1 SM 2540C	EPA 160.1 SM 2540C	EPA 160.1 SM 2540C
Non-Filterable Residue (TSS)	SM 2540D	SM 2540D	SM 2540D
Ferrous Iron	SM 3500-Fe Bc	SM 3500-Fe Bc	----------
Sulfide	SM 4500-S_2F	SM 4500-S_2F	SM 4500-S_2F
Total Organic Carbon (TOC)	EPA 9060A SM 5310C	----------	WALKLEY-BLACK
Redox Potential (ORP)	SM 2580B	SM 2580B	----------
Sample Preparation			
TCLP Extraction	----------	EPA 1311	EPA 1311
SPLP Extraction	----------	EPA 1312	EPA 1312
Acid Digestion for Metals Analysis	EPA 3010A	EPA 3010A	----------
Acid Digestion for Metals Analysis	----------	EPA 3050B	EPA 3050B
Alkaline Digestion for Hexavalent Chromium	----------	EPA 3060A	EPA 3060A
Separatory Funnel Extraction	EPA 3510C	EPA 3510C	----------
Liquid-Liquid Extraction	EPA 3520C	EPA 3520C	----------
Soxhlet Extraction	----------	EPA 3540C	EPA 3540C
SPE Extraction for Explosives	EPA 3535A	EPA 3535A	----------
Ultrasonic Extraction	----------	EPA 3550B Mod.	EPA 3550B Mod.
Silica Gel Cleanup	EPA 3630C	EPA 3630C	EPA 3630C
Sulfur Cleanup	EPA 3660B	EPA 3660B	EPA 3660B
Sulfuric Acid - Permanganate Cleanup	EPA 3665A Mod.	EPA 3665A Mod.	EPA 3665A Mod.
Purge and Trap	EPA 5030B/5030C	EPA 5030B/5030C	EPA 5030B/5030C
Closed-System Purge and Trap Extraction	EPA 5035/5035A	EPA 5035/5035A	EPA 5035/5035A
Mercury Digestion	EPA 7470A	EPA 7470A	EPA 7471A/7471B
Incremental Sampling	----------	EPA 8330B, Appendix A	EPA 8330B, Appendix A
Waste Extraction Test (WET) (STLC)	----------	CCR Chapter 11, Article 5, Appendix II	CCR Chapter 11, Article 5, Appendix II
Organic Microwave Extractor	EPA 3546	EPA 3546	EPA 3546

Parameter/Analyte	Potable Water
Metals	
Mercury	EPA 245.1
Inorganics	
Chromium VI	EPA 218.6/218.7
Bromide	EPA 300.0
Chloride	EPA 300.0
Fluoride	EPA 300.0
Nitrate as N	EPA 300.0
Nitrite + Nitrate as N	EPA 300.0
Nitrite as N	EPA 300.0
Orthophosphate as P	EPA 300.0
Phosphate	EPA 300.0
Sulfate	EPA 300.0
Phosphate/Orthophosphate/Total Phosphorus	SM 4500 PE
Cyanide, total and Amenable	SM 4500CN B, C, E, G
Ammonia as N	EPA 350.1
Total Kheldahl Nitrogen	EPA 351.2
Nitrate as N	EPA 353.2
Nitriate + Nitrate as N	EPA 353.2
Nitrite as N	EPA 353.2
Nitrocellulose	EPA 353.2 Mod.
Bicarbonate	SM 2320B
Carbonate	SM 2320B
Hydroxide	SM 2320B
Total Alkalinity (CaCO3)	SM 2320B
Specific Conductance, Conductivity (25C)	SM 2510B
Total Dissolved Solids (TDS)	EPA 160.1
Ferrous Iron	SM 3500-Fe Bc
Sulfide	SM 4500-S_2F
Total Organic Carbon (TOC)	SM 5310C
Dissolved Organic Carbon (DOC)	SM 5310C
Per- & Polyfluoroalkyl Substances (PFAS)	
N-EtFOSAA	EPA 537.1
N-MeFOSAA	EPA 537.1
PFBS	EPA 537.1
PFDA	EPA 537.1
PFDoA	EPA 537.1
PFHpA	EPA 537.1
PFHxA	EPA 537.1
PFHxS	EPA 537.1
PFNA	EPA 537.1
PFOA	EPA 537.1
PFOS	EPA 537.1
PFTeDA	EPA 537.1
PFTrDA	EPA 537.1
PFUdA	EPA 537.1
HFPO-DA	EPA 537.1
9-Cl-PF30NS	EPA 537.1

Parameter/Analyte	Potable Water
11-Cl-PF30UdS	EPA 537.1
ADONA	EPA 537.1
PFBA	EPA 533
PFPeA	EPA 533
PFHxA	EPA 533
PFHpA	EPA 533
PFOA	EPA 533
PFNA	EPA 533
PFDA	EPA 533
PFUnA	EPA 533
PFDoA	EPA 533
PFBS	EPA 533
PFPeS	EPA 533
PFHxS	EPA 533
PFHpS	EPA 533
PFOS	EPA 533
4:2FTS	EPA 533
6:2FTS	EPA 533
8:2FTS	EPA 533
PFMPA	EPA 533
PFMBA	EPA 533
NFDHA	EPA 533
HFPO-DA	EPA 533
ADONA	EPA 533
PFEESA	EPA 533
9Cl-PF3ONS	EPA 533
11 Cl-PF3OUdS	EPA 533
Sample Preparation	
Mercury digestion	EPA 245.1
PFAS SPE	EPA 537.1

Standard Operating Procedure

Instrumental Analysis of Per- and Polyfluoroalkyl Substances (PFAS)
BY LC-MS/MS (EPA Method 537.1, 533 and DoD QSM Table B-15)

1 STATEMENT OF PURPOSE

1.A This procedure describes the proper way analyze extracts from solid and liquid matrices for PFAS using liquid chromatography tandem mass spectrometry by EPA Method 537.1, 533, and in house DoD QSM complaint methods.

2 INSTRUCTIONS

3 Scope and Application

3.A This method describes instrumental analysis of PFAS in extracts from solid and aqueous matrices using liquid chromatography tandem mass spectrometry according to EPA 537.1 , EPA 533, and in compliance with DoD QSM Table B-15.

3.B This method is restricted to use by or under the supervision of personnel who have been trained according to this SOP as PFAS instrument analysts for the purpose of analyzing extracts from water and soil samples using LCMS-MS and interpreting and quantitating sample data using isotope dilution. Each analyst must demonstrate the ability to generate acceptable results with this method. If an individual project has its own QAPP with client specific requirements that are different than the SOP, the QAPP overrides the SOP. This information will be specified in the comment section of the ARF.

4 Method Summary

4.A Samples are spiked with isotopologues then extracted using SPE or SLE.

4.B The analytes are separated by liquid chromatography using a Gemini® 3 µm C18 110 Å column (or equivalent) and detected by MS/MS.

4.C An analyte is identified in a sample by comparing the RT to the standard retention time and the relative retention time to isotopologues. When appropriate, the analyte can be confirmed by comparing the secondary transition associated to that compound and compared to the standards' ion ratios.

4.D Quantitation is achieved based on a minimum five-point calibration curve using the appropriate quantification method for the requested method. EPA 537.1 required the internal standard method with extracted surrogate, EPA 533 required isotope dilution with all commercially available isotopologues and isotope performance standards. The QSM table B-15 requires internal standard or isotope dilution. Isotope dilution will be performed when isotopologues are commercially available.

5 Detection Limits

5.A See section 23 Tables.

6 Definitions

See SOP# DOC019-Definitions

7 Interferences and Potential Problems

7.A All glassware must be meticulously cleaned. Wash glassware with detergent and tap water, rinse with tap water, followed by a reagent water rinse. If the glassware is to be used for methanol, a final rinse with HPLC grade methanol is recommended. Nonvolumetric glassware can be heated in a muffle furnace at 400 °C for 2 h or solvent rinsed. Volumetric glassware should be solvent rinsed and not be heated in an oven above 120 °C. Store clean glassware inverted or capped. Do not cover with aluminum foil because PFAAs can be potentially transferred from the aluminum foil to the glassware.

7.B 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 target 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, SPE sample transfer lines, etc. All items such as these must be routinely demonstrated to be free from interferences (less than 1/3 the MRL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent blanks.

7.B.1 Potential lab contaminant peaks associated with glassware, instrument tubing, or reagents may be effectively separated from target analyte peaks by using the Luna® 5 µm C18(2) 100 Å column; or equivalent.

7.C Matrix interference may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from source to source, depending upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent.4-5 Total organic carbon (TOC) is a good indicator of humic content of the sample. Under the LC conditions used during method development, matrix effects due to total organic carbon (TOC) were not observed.

7.D Relatively large quantities of the preservative are added to sample bottles for EPA 537.1. The potential exists for trace-level organic contaminants in these reagents. Interference from these sources should be monitored by analysis of laboratory reagent blanks with each preparatory batch, particularly when new lots of reagents are acquired.

7.E SPE cartridges can be a source of interference. The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

7.F NOTE: PFAA standards, extracts and samples should not come in contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAA analyte, IS (Internal Standards) and SUR (Surrogate) standards commercially purchased in glass ampules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers.

8 Health and Safety

8.A Lab coats, gloves, and safety glasses are to be used at all times.

9 Sample Preservation, Containers, Handling and Storage – See SOP PRE537

10 Quality Control

10.A Initial Demonstration of Capability (DOC):

10.B A DOC is completed according to SOP # QC006 when a new employee starts work and annually thereafter, using four replicate spikes at 1-4 times the LOQ.

10.C The acceptance criteria for all target analytes in the IDOC is 70-130% for accuracy and < 20%RPD for precision.

10.D For analytes that fail to meet acceptance criteria, repeat the IDOC before any sample analysis takes place.

10.E Method Detection Limits (MDL): Establish the limit of detection (LOD), limit of quantitation0 (LOQ), detection limit (DL), and method detection limit (MDL) according to SOP QC018.

10.F An MDL study must be analyzed at initial method setup and whenever there are major changes to the instrument.

10.F.1 The LODs and LOQs are confirmed quarterly. See QC018 Establishing MDL, LOD, LOQ for more information.

10.F.2 A retention time window study is performed at method set up and after every major instrument change such as a new column. Three injections of the standard or standards containing all analytes of interest including surrogates are made over the course of a 72-hour period. The retention time for each compound is recorded to three decimal places (e.g., 0.007). The width of the retention time window is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. The retention time window position is set using the mid point of the ICAL standard. On days when an ICAL is not performed, the initial CCV is used to set the window position. It is acceptable to set the retention time window wider than the study indicates to ensure positive identification of all compounds. It is not permissible to set the window width less than the retention time window study.

10.G On-going Quality Control for sample extraction -

10.G.1 Sample quality control for preparation and analysis include the analysis of a method blank (BLK), a laboratory control spike (LCS), a matrix spike (MS), and a matrix duplicate (DUP) or matrix spike duplicate (MSD) in each analytical batch of 20 samples, and the addition of labeled internal standards and labeled C13 surrogates to each sample and QC sample.

10.G.2 The method blank must be shown to contain no analytes of interest >1/3 the LOQ for 537.1 and >½ LOQ for DoD (or >1/10th the amount in a sample or the regulatory method which ever is greater).

Corrective Action: If there is a detection >1/3 LOQ (or >½ LOQ for DoD) in the method blank the entire batch associated with the BLK will be re-extracted and reanalyzed. Sample data may not be reported if the method blank exceeds these minimum blank contamination requirements. If not enough sample volume exists for a re-extraction, the client will be notified for permission to report the sample as qualified with a 'B' flag for 'compound found in the associated blank. Samples associated with method blank concentrations <1/10 sample concentration or <1/10 regulatory limit do not require B-flags.

10.G.3 The laboratory control spike consists of an aliquot of control matrix similar to the sample matrix and of the sample weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike/matrix spike duplicate. See section 23 Tables in this SOP for compounds and control limits.

Corrective Action: If the LCS does not meet acceptance criteria (70-130% true amount for medium, high, and DoD; 50-150% true amount for low concentration), the data must be considered invalid. The entire batch associated with the LCS will be re-extracted and reanalyzed. For DoD if not enough sample volume exists for a re-extraction, the sample will be qualified with a 'Q' for the analyte not meeting acceptance criteria with client instruction.

For DoD projects, an LCS/LCSD is required if there is insufficient volume for MS/MSD.

10.G.4 The matrix spike and matrix spike duplicate will be included with each sample batch as per client. The client is to determine which sample is designated with MS/MSD. The %RPD limit for the MS/MSD is ≤30%. If the criteria is not met notify the project manager who will examine the DQOs and contact the client. For DoD projects, an LCSD is required, if there is insufficient volume for MS/MSD.

Corrective Action: If the MS does not meet acceptance criteria (70-130% true amount for medium, high, and DoD; 50-150% true amount for low concentration) which indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. For DoD, the sample will be qualified with a 'J' for the analyte not meeting acceptance criteria with client instruction.

10.G.5 For a matrix duplicate sample, any native hits associated in the sample and sample duplicate must have a %RPD limit of ≤30%.

10.G.6 All samples and quality control is fortified with labeled surrogates before being extracted. For 537.1, surrogates must recover between 70-130% of true amount. For DoD, surrogates must recover between 50-150% of true amount.

10.H On-going Quality Control for instrumentation –

10.H.1 A Calibration Curve is required at instrument set-up and after ICV or CCV failure, prior to sample analysis. Section 10 details how to prepare a calibration curve and Section 11 details how to analyze, quantitate, and evaluate a calibration curve, and Section 13 details the calculations used to evaluate the curve and subsequent calibration verifications.

Method-specific initial calibration criteria is discussed in more detail below for EPA 537.1 and DoD QSM Table B-15.

10.H.2 Initial Calibration Verification (ICV) – An ICV is a second source and must be analyzed after the calibration curve and prior to sample analysis. Analytes must recover within 70-130% of the true amount.

10.H.3 Continuing Calibration Verification (CCV) – A CCV must be analyzed at the beginning and the end of an analytical sequence, and after every tenth field sample during analysis.

For EPA 537.1, the CCV at the beginning of the sequence must be at or below the LOQ and must recover within 50-150% of the true amount; the subsequent CCVs are rotated between medium and high concentration calibration standards and must recover within 70-130% of the true amount.

For DoD, the CCV concentrations may range from the LOQ to the mid-level calibration range. The CCV must recover within 70-130% of the true amount. In addition, an LLCCV is prepared at the LOQ concentration and must recover within 70-130% of the true amount. The LLCCV is injected prior to analysis and at lease every 12 hours.

For DoD projects using QSM revision 5.1 (or later), if a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., trap column, mobile phase preparation, run blanks) requires that all samples since the last acceptable CCV be reanalyzed. Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis. If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed. Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The lab must notify the client prior to reporting data associated with a failed CCV.

Sample injection may continue for as long as all the calibration verification standard requirements listed above are met.

10.H.4 Instrument Blank (ICB/CCB) – An instrument blank must be analyzed immediately following the highest standard of the calibration curve and daily prior to sample analysis. The instrument blank must be shown to contain no analytes of interest >1/3 the LOQ for 537.1 and >½ LOQ for DoD.

10.H.5 All samples and quality control samples are spiked with labeled internal standards after extraction and before analysis. For 537.1, the internal standards must recover within 70-140% of the areas measured in the most recent CCV and within 50-150% of the average areas measured in the initial calibration. For DoD, the internal standards must recover within 50-150% of the daily initial CCV.

Note: All instrument quality control is fortified with labeled surrogates in addition to internal standards.

10.H.6 A second source (a spike source different from the calibration source) must be analyzed at least quarterly and when a new native intermediate is prepared. If a second vendor is not available, then a different lot of the standard should be used. The analytes must recover with 70-130% of the true amount.

10.I Quality control requirements specific to EPA 537.1 include:

10.I.1 Initial Demonstration of Peak Asymmetry Factor - the peak asymmetry factors must be calculated according to the formula in 13.0 for the first two eluting peaks using a mid-level calibration standard. The peak asymmetry factor must fall in the range of 0.8-1.5. The peak asymmetry factor must meet criteria every time a new calibration curve is generated. If it does not meet acceptance criteria, change the mobile phase conditions to higher aqueous content until the ratio for each peak is 0.8-1.5.The isotopically labeled compounds in this method may undergo suppression in the ESI source if the concentration of the co-eluting native analyte(s) is too high. The analyte concentration at which suppression may occur can vary depending on the instrument, LC conditions, ESI conditions, IS concentration, etc. See section 13 for the equation for calculating the %RPD between the high and low areas for each IS.

The %RPD calculated above must be <20% for each IS during calibration. If the calculated RPD is >20% for any IS, the analyst must recalibrate at lower analyte concentrations until the IS RPDs are <20%.

10.I.2 A field blank per every sample set. A sample set is composed of samples collected from the same sample site and at the same time.

10.I.3 The laboratory control spike must be rotated between low, medium, and high concentrations from batch to batch. The low spike concentration must be no more than two times the LOQ and must recover between 50-150% of the true amount. The high spike concentration must be near the high end of the calibration range. The medium and high spikes must recover between 70-130% of the true amount.

10.I.4 The matrix spike must be rotated between low, medium, and high concentrations from batch to batch. The low spike concentration must be no more than two times the LOQ and must recover between 50-150% of the true amount. The high spike concentration must be near the high end of the calibration range. The medium and high spikes must recover

10.J Quality control requirements specific to DoD QSM Table B-15 include:

10.J.1 The laboratory control spikes and matrix spikes must be spiked ≥ the LOQ and ≤ the mid-point of the calibration range and must recover between 70-130% of the true value.

10.J.2 An instrument sensitivity check (LLCCV) is required prior to sample analysis and once every 12 hrs. The LLCCV must be at the LOQ and recover within 70-130% of the true amount. The LLCCV can serve as the opening CCV for a sequence.

10.J.3 For any failures, check the following before moving forward with a corrective action:

10.J.4 Calculations to locate possible errors

10.J.5 Standard solutions for degradation

10.J.6 Contamination

10.J.7 Instrument performance

10.K Corrective Action: Errors, deficiencies, deviations, or laboratory events or data that fall outside of established acceptance criteria will be investigated. In some instances, corrective action may be needed to resolve the problem and restore proper functioning to the analytical system. The investigation of the problem and any subsequent corrective action taken is documented on a Nonconformance Work Report (NWR) and/or a Corrective Action Report (CAR).

10.L Deviations: Any activity not performed in accordance with laboratory procedures or Quality Assurance Project Plans is considered a deviation from plan. All deviations from plan will be documented as to the extent of, and reason for, the deviation.

11 Equipment/Apparatus

11.A Liquid chromatography (LC) / tandem mass spectrometer (MS/MS) with Data System

11.A.1 The TQ5500 LC MS/MS by SCIEX
11.A.2 Analyst® and MultiQuant™ Software by SCIEX
11.B LC Operational Settings:
11.B.1 Autosampler Temperature: 15 °C
11.B.2 Oven Temp: 40 °C
11.B.3 Injection Volumes: 10uL
11.C Mass Spec Operational Settings:
11.C.1 Scan Type: MRM (MRM)
11.C.2 Duration: 15 min
11.C.3 MRM Detection Window: 180 sec
11.C.4 Target Scan Time: 0.2 sec
11.D LC column
11.D.1 The suggested analytical column is a Cadenza CX-C18 VHP.
11.D.2 A second Cadenza Cx-C18 VHP column recommended for the removal of contamination introduced from instrument tubing and mobile phase reagents. The quality of this column is such that rejected analytical seperator columns may be used long after their normal lifespan in this position.
11.E Compressed nitrogen or nitrogen generator
11.F High Density Polyethylene (HDPE) bottles- 4mL and 10mL
11.G Centrifuge Tubes, 15 mL, polypropylene
11.H Injection vials with polyethylene caps:
11.I Polypropylene 300 µL vials
11.J Media 1 L Bottles
11.K Various Class A Glassware – not limited to volumetric flasks, graduated cylinders, and beakers.
11.L Adjustable Pipettes of various sizes

12 Reagents and Standards

12.A Reagent or pesticide grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All reagent and chemical lots will be documented properly for traceability. Certified reference Materials or stock standards must be purchased from a body that can provide ILAC-signatory (MRA) traceability and ISO/EC 9001, 17025, and 17034 certification.
12.A.1 Reagents:
12.A.2 Puchased Reagents:
All purchased reagents must be of high purity and demonstrated to be free of analytes and interferences.
TRIZMA® – Preset Crystals, pH 7.0 (Sigma cat# T-7193 or equivalent) –Reagent grade. A premixed blend of Tris [Tris(hydroxymethyl)aminomethane] and Tris HCL [Tris(hydroxymethyl)aminomethane hydrochloride. These blends are targeted to produce a pH near 7.0 at 25 °C in reagent water. Trizma® functions as a buffer, and removes free chlorine in chlorinated finished waters.
HPLC Grade Water – Purified water which does not contain any measurable quantities of any method analytes or interfering compounds greater than 1/3 the MRL for each method analyte of interest.
DI water from the laboratory system may be used in place of HPLC Grade water if it does not contain any measurable quantities of any method analytes or interfering compounds greater than 1/3 the MRL for each method analyte of interest. If DI water is to be used, 3 L must be flushed through the system prior to use.
Methanol HPLC Grade
Ammonium Acetate LCMS Grade
Glacial Acetic acid LCMS Grade
Ammonium Hydroxide LCMS Grade
12.A.3 Prepared Reagents:
Mobile Phase A: add 1.0mL of glacial acetic acid and 1.0mL ammonium hydroxide to 1L of HPLC Grade Water.
Mobile Phase B: add 1.0mL of glacial acetic acid and 1.0mL ammonium hydroxide 1 L of HPLC Grade Methanol
the standards above may be prepared in smaller and larger quantities as long as the ratios are kept the same.
12.B Standards:
12.B.1 All standards should be stored in polypropylene or HDPE containers at <4 °C to minimize being concentrated from the evaporation of methanol.
Note: When purchasing standards, the DoD requires the following when commercially available:
Both branched and linear isomers of the native compounds
Labeled analogs for each native analyte
12.B.2 Purchased Standards:
2000 ng/mL 24 Native Calibration Standard – Wellington, product number PFAC-24PAR; or equivalent.
Consists of 24 different PFAS compounds. Contains both branched and linear isomers for PFOS and PFHxS.
1000 ng/mL 24 Native Second Source – Absolute, product number 99207; or equivalent.
Consist of 24 different PFAS compounds. Contains both branched and linear isomers for PFOS and PFHxS.

1000-4000 ng/mL EPA 537.1 Surrogates – Wellington, product number EPA-537SS; or equivalent.
Consists of 3 different mass-labeled PFAS compounds. The isotopes used include ^{13}C and ^{2}H.
1000 ng/mL DOD 537 Surrogates – Wellington, product number MPFAC-24ES; or equivalent.
Consists of 19 different mass-labeled PFAS compounds. The isotopes used include ^{13}C and ^{2}H.
1000-4000 ng/mL EPA 537.1 Internal Standards – Wellington, product number EPA-537IS; or equivalent.
Consists of 3 different mass labeled PFAS compounds. The isotopes used include ^{13}C and ^{2}H.
2000 ng/mL DOD 537 Internal Standards – Wellington, product number MPFAC-C-IS; or equivalent.
Consists of 4 different mass labeled PFAS compounds. The isotope used is limited to ^{13}C.

12.B.3 Prepared Standards:

Note: When preparing standards…
All QSM TB-15 and 533 standards are to be prepared for a final methanol: water ratio of 80%:20%.
All 537.1 standards are to be prepared for a final methanol: water ratio of 96%:4%.

The stock solutions listed above are diluted into intermediates. The intermediates are then used to spike the calibration curve and all quality control samples. Refer to the standard prep log book, H:\Saphira\Standard Prep Log Book, for instructions on preparation of the intermediate standards.

12.B.4 The preparation of all standards is to be recorded and saved in H:\Saphira\Standard Prep Log Book.

12.B.5 The expiration date for analytical standards may be extended by following re-certification procedures below:

Contact the standard supplier and request an expiration date extension. If the supplier is not able to extend the date, then in-house re-certification may be performed as follows:

Analyze the expired standard and quantitate it against an unexpired standard from a different supplier or different lot # from same supplier.
For organic standards, the acceptance criteria is 20%D.
If the expired standard meets the above acceptance criteria, then extend the date by 3 months for organics.
The re-certification with extended expiration date is documented in the standard prep log book, along with the analysis date, instrument name, data file ID, and name of standard supplier with current unexpired lot number that was used to re-certify the expired standard.

Email this information to QAU.

13 Calibration and Standardization

13.A Prior to use and after major maintenance is performed, a mass calibration will be performed by a certified technician from SCIEX.

13.B Before Calibrating, perform a Tune Check by running a mid-point standard using the PFOS Tune Check method. See section 12.0 for details on how to inject. Verify that the mass of PFOS is ±0.5 amu of the true value (80 amu) by:

13.B.1 Opening the data for the tune check by double clicking the "Open Data File" Icon located underneath the Explore Icon on the left hand side of the Analyst® software.

13.B.2 Select the sample being used for the Tune Check and hit OK.

13.B.3 This will open a screen with peaks on it. Highlight the peaks then double click on the highlighted portion. This will zoom onto the PFOS peak. If the mass is within 79.5-80.5 amu, the tune check is acceptable.

13.B.4 If the Tune Check fails, SCIEX must be contacted and a certified technician will come out and perform an instrument tune. If the instrument tune does not resolve the problem, the SCIEX technician will perform a mass calibration.

13.C For details on how to prepare a calibration curve, reference section 10 and the most recent standard prep page located in H:\Saphira\Standard Prep Log Book.

13.D Once the calibration standards are prepared, analyze the standards and process the data according to the procedure in Section 12.0.

13.E The following applies to all calibration curves:

13.E.1 One of the concentrations will be at the quantitation limit. The analyst must refer to the incoming sample notice for the lab works code and look at the detection limits listed on the appropriate form 1 to determine the quantitation limit standard. The initial calibration curve is a reflection of the performance of the instrument at any given time. Compounds react to the changing dynamic of the instrument. Therefore it is sometimes necessary to delete levels for compounds in an initial calibration curve. When this occurs the following rules are followed to ensure integrity of the data:

13.E.2 A standard must be included in the curve for each compound, which is less than or equal to the reporting limit. If the responses of a sample peak exceed the calibration range of the system, dilute the extract and reanalyze.

13.E.3 The deletion of discrete points must never result in a calibration curve consisting of less than five points for each analyte of interest.

13.E.4 Points for an individual analyte in the middle of the curve may not be deleted, however unforeseen circumstances may occur such as a miss injection by the autosampler, a loose cap on an injection vial, etc. In this situation the entire level is deleted for all compounds and the reason for deletion is noted on the multilevel form. If this results in a calibration curve that consists of less than five points, another level may be run before the analysis of samples begin.

13.E.5 Points at the low end and high end of the curve may be deleted if it is determined the compound ceases to be linear at either end. Any positive findings in the samples will be analyzed so as to fall within the linear range of that particular compound.

13.F For a calibration curve to be considered acceptable for QSM TB-15 and meet DoD criteria, the following conditions must be met:

13.F.1 Note: secondary transitions do not need to meet acceptance criteria since they are only used to confirm the existence of the analyte and not the concentration. The only secondary transition requirement is the S/N ratio must be ≥ 3:1 for analytes containing branched analytes in the standard. The ion ratio must be monitored and also documented using the instrument software for qualitative purposes. For a sample containing an analyte in the primary transition, it must be

documented on the multilevel (Table 5) that the secondary transition was also present in order for positive detection to be confirmed.

13.F.2 Calibrations must contain a minimum of 5 points for linear regression or 6 points for quadratic regression; weighting is allowed.

13.F.3 If a labeled analog is not commercially available, then the surrogate with the closest retention time is used for quantitation.

13.F.4 $r^2 \geq 0.99$ for each analyte.

13.F.5 All analytes must recover within 70-130% of their true amount.

13.F.6 The RSD% of the RFs for all analytes must be <20%. This is calculated using the Query described in section 12.0.

13.F.7 Internal standards must recover within 50-150% of the midpoint of the curve, and surrogates must recover within 50-150% of the true amount.

13.G For a calibration curve to be considered acceptable for EPA 537.1, the following conditions must be met:

13.G.1 Note: secondary transitions do not need to meet acceptance criteria since they are only used to confirm the existence of the analyte and not the concentration.

13.G.2 Peak Asymmetry Factor must fall in the range of 0.8-1.5 in the mid-level standard for the first two eluting peaks.

13.G.3 Calibrations must contain a minimum of 5 points spanning a 20-fold range.

13.G.4 Curves must always be forced through zero according to the EPA 537.1 method. Weighting is allowed.

13.G.5 All analytes must recover within 70-130% of their true amount; except the lowest point must recover with 50-150% of their true amount.

13.G.6 Internal standards must recover with 70-140% of the midpoint of the curve, surrogates must recover within 70-130% of the true amount.

13.G.7 The RPD for the mass-labeled internal standard area between the low and high calibration standard must be <20% for each internal standard. See section 13 for the calculation.

13.H An instrument blank, CCV, and ICV are to be injected after the completion of a curve. For more information and acceptance criteria, see section 8.

13.I Each sample analysis sequence must include an acceptable initial calibration; calibration verification standards. When a CCV or Resolution Check fails to meet the acceptance criteria, all samples that were injected after the last acceptable check must be re-injected.

14 Procedure

14.A See SOP PRE537 for the extraction procedure of aqueous and solid samples. The extracts will be analyzed according to the procedure listed below. The analyst is given a copy of the final extraction sheet from the extraction technician for each preparatory batch. The analyst uses the information on extraction sheet to enter sample weights and final volumes into the instrument software for quantitation purposes. In addition, the analyst uses the spike and surrogate information from the extraction sheet to calculate the spike and surrogate recoveries in the samples and associated QC samples. See section 13 of this SOP for calculations.

14.B Receipt of extracts:

14.B.1 Extracts are received and ensured they have been brought to the appropriate pH, final volume, and internal standards concentration. This is noted on the extraction sheet with a date and initial.

EPA 537.1 extracts require the 10µL/mL EPA537 IS solution

EPA 533 extracts require 10µL/mL IIS solution.

DoD/QSM Table B-15 samples do not require IS or IIS but can be spiked with 10µL/mL IIS solution to comply with legacy calculation methods. Extracts are sometimes received in alkaline conditions when ammonia fails to evaporate in the concentration step. These samples may be acidified with 20µL of acetic acid to ensure that PFBA and PFMBA do no split.

14.C Vialing

14.C.1 100µL of sample is transferred to a 300 µL conical injection vial and capped with a polyethylene cap.

Or 500µL of sample is transferred to a 1mL flat bottom injection vial and capped with a polyethylene cap.

14.C.2 For samples requiring small dilutions:

The injection volume may be modified to provide up to a 100x dilution factor (0.1µL/10µL) without any change in sample provide sufficient EIS sensitivity exists. S/N should aways be greater than 10 for EIS.

Due to difficulties with sulfur isotopes and compounds labeled M+2, some great bias may be seen when diluting this way for 4:2, 6:2, and 8:2 FTS. Due to this D4-13C2 isotoplologues are employed instead.

14.C.3 For samples requiring large dilutions (where the internal standard signal to noise cannot be maintained above 10)

Sample are diluted with a stock solution containing all relevant IS/EIS/IIS and no native present.

EPA 533 does not support dilutions in any way, instead re-extraction must be performed with a subsampled aliquot.

DoD/QSM Table B-15 samples are best reacquired as dilutions of the unextracted sample.

Samples prepared this way are required to be prepared and analyzed in duplicate and with PS samples every batch.

14.D Creating analysis batches and sequences and submitting them to the insturment.

14.D.1 Open the Analyst® software and connect to the mass spec by going to the Hardware Configuration Tab on the left side of the screen, double click to open. Select LCMS System and “Activate Profile.” This should result in an audible beep from the instrument and a green check mark indicating that the system has been connected to the computer.

14.D.2 If the system does not connect, power cycle the LC units, autosampler, and controller box and try to connect again.

14.D.3 Make sure the correct project folder is open, it should correlate to the date of the curve the samples are to be acquired under.

14.D.4 Under the navigation plane select acquire and create aquision batch.

14.D.5 Generate a set by typing the Date in the format YYYY-MM-DD in the set field and select create batch

14.D.6 Add samples to be the batch by selecting add samples
14.D.7 Name the samples relevantly to what you are injecting
14.D.8 Add the correct vial locations
14.D.9 Make sure they are being submitted under a data file of the format YYYY-MM-DD
14.D.10 Add the following information to the sample field in the following format
FA,units,IA,UCF,other
where:
FA= Final Amount with units
units= units being reported (ng/mL for CCV's , ng/L, ng/g, ug/L, or ng/mL for AFFF)
IA= Initial Amounts with units
UCF= Unit Conversion Factor (multiples of 1000)
14.D.11 Change to the submit tab and submit samples to the que in the correct order.
If the instrument is going from IDLE into the queue ensure that a MeOH cycle is run before analysis. The instrument uses the end of one injection to prepare for the next and the column oven needs to reach temperature before analysis can begin. A single cycle is sufficient to ensure this happens.
14.D.12 Press the ready button the bring the mass spectrometer online and press start queue to being the analysis.

14.E Analysis of data usning multiquant

14.E.1 Open the MultiQuant™ software in order to process the data. If a new calibration curve has been analyzed, go to file then "New Results Table." Select the data to be processed.
14.E.2 Hit "Next", choose an existing processing method (PFAS 537 for 537.1 or PFAS DOD for QSM TB-15) then hit "Finish." Save the file with the QCG name.
14.E.3 If the data will be processed from an existing calibration curve, go to file then "Open Results Table." Open the most recent ICAL file. Save the file with the QCG name for the samples that will be processed and continue to the steps below.
14.E.4 This will lead to screen where every analyte is listed for every sample. One analyte can be viewed by selecting it in the list on the left hand side of the screen.
14.E.5 The chromatograms and calibration curves can be viewed by selecting the appropriate symbols and the top of the screen.
14.E.6 The data set can then by processed using a Query which was developed, controlled, and provided by the manufacturer. This query calculate asymmetry factors, IS/surr recovery, and various other requirements. To process with the query, go to "Process" at the top of the screen, select Query. This lead to another screen. Select the correct query (PFAS_QUERY 2 for DOD/537M and PFAS_QUERY 537 for 537.1)
14.E.7 Click OK, fill out the acceptance criteria for the IS, the amount of sample added to the injection vial, the final volume in the injection vial, and the final volume of the extract. Click OK.
14.E.8 The Query will flag failures with a reason listed in the "Failure Reason" column. These failures include but are not limited to accuracy (CCV/linearity check failures), IS/Surr failures, potential problems with Ion ratio or signal/noise ratio. All of the flags should be thoroughly checked by the analyst and are not to be assumed as final until verified by the analyst.
14.E.9 Peaks may be manually integrated if the software did not properly integrate a specific peak, by using the mouse to accurately draw the baseline integration. Chromatographic noise peaks may be removed by setting the integration to "Not Found." For analytes with multiple peaks (branched and linear), all of the peaks associated with the analyte should be integrated and reported as the summation of the peaks for that particular analyte. This is applicable to the calibration standards as well as samples.
14.E.10 After any changes are made, save the file. To export or to pdf the data, go to file then Create Report select the correct report template: Sample Report APPL for PDF and printing data, ICAL Report 2 for PDF and printing a calibration curve, and CSV LIMS Report APPL for exporting the data. Save the PDF or export file into the HPLC Server by hitting the "Set… " button to the right of Generate report file. This will allow the data to be named by the QCG name and the data to be saved in the correct location.
14.E.11 Select the output format (PDF, word, csv) then hit ok.
14.E.12 The data is now ready to be uploaded into the LIMS System.

15 Data Analysis and Calculations

There exists many methods of quantification for PFAS analysis. EPA 8327, which is not explored within, relies on external standard quantification. EPA 537.1 and its sister methods rely on an internal standard approach. The QSM suggests Isotope dilution without Isotope performance standards, while EPA 533 requires isotope dilution with isotope performance standards. APPL takes great care in providing exactly as stated for all methods performed. The multiquant software employed by APPL is both powerful and versatile, but should not be used without caution. Anyone using the software is fully responsible to understand the math it's performing, the requirements of the methods performed, and to endure they are congruous.

15.B Multiquant generated equations

Mean response factor, Linear regressions and Quadratic regressions are generated by Multiqaunt software, may be unwighted, weighted $1/x$ or weighted $1/x^2$ as necessary.
They all take the form of $y = f(x)$
Where:

$$x = \frac{Concentration_{Analyte}}{Concentration_{InternalStandard}}, \quad y = \frac{Area_{Analyte}}{Area_{InternalStandard}}$$

The generation of these functions is not described herein, but their aplication is.

15.C Single-point external standard quantification (DoD/DoE QSM 5.3 – Table B-15 - Extracted Internal Standard)

"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."
-DoD/DoE QSM 5.3 Table B-15

In order to best represent the quoted requirement, multiquant is programmed to present EIS recoveries as a percentage of the most recent QC sample.

$$\%EIS = \frac{Area_{sample}}{Area_{CCV}} * 100$$

This forces two things, the concentration of EIS cannot vary between injections and the EIS may not be corrected by an Injected internal standard.

15.D Multi-point external standard quantification (EPA 8327)

Regression equations from 17.A are applied and the concentration and area of internal standards is assumed to be 1.

15.E Internal standard quantification (EPA 537.1)

Regression equations from 17.A are applied and the isotopologues added after the sample extract concentration step are used for the internal standards; in this way samples are correct for instrument variation and final volume variation.

15.F Isotope dilution quantification (EPA 533 and DoD/DoE QSM 5.3 – Table B-15)

Regression equations from 17.A are applied and the isotopologues added before the sample concentration concentration step are used as internal standards; in this way analytes are correct for extraction deficiencies, matrix effects, instrument variation and final volume variation.

15.G Isotope performance quantification (EPA 533 - extracted internal standard)

Regression equations from 17.A are applied and the isotopologues added before the sample concentration concentration step are treated as analytes and isotopoglues added after the sample concentration step are used as internal standards; in this way extracted isotopologues are correct for instrument variation and final volume variation.

15.H Conversion from in extract to in sample concentration

$$Concentration_{Sample} = Concentration_{Extract} * \frac{Volume_{Final}}{Amount_{Initial}} * UnitConversionFactor$$

15.I Correction of data to the protonated acid state.

All results are reported in the protonated acid form HA, where as some of our analytes come in stock solutions in the form KA or NaA. Correction of results is performed as such:

$$Concentration_{Acid} = Concentration_{Salt} * \frac{MolecularWeight_{Acid}}{MolecularWeight_{Salt}}$$

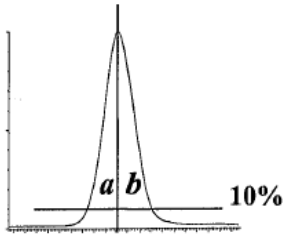
15.J Calibration standards, internal standards, surrogates, and spikes are prepared from dilutions of the purchased stock standards using the following equation:

$$C_1 * V_1 = C_2 * V_2$$

15.K Asymmetry Factor:

This calculation is performed by Multiquant and applies to the first two eluting peaks in EPA 537.1 which must be within 0.8-1.5.

$$A_s = \frac{b}{a}$$



where:

A_s = peak asymmetry factor

B = width of the back half of the peak measured (at 10% peak height) from the trailing edge of the peak to a line dropped perpendicularly from the peak apex

a = the width of the front half of the peak measured (at 10% peak height) from the leading edge of the peak to a line dropped perpendicularly from the apex.

15.L Manual integration: The MultiQuant™ software program is used for integration and quantitation of PFAS data. Manual integration of peaks should be consistent with the ICAL integrations for each standard level. If method acceptance criteria are not met for a particular analyte, then examine the software's integration for that analyte, and determine whether or not the integration is consistent with the ICAL integration at each level. If not, then perform the appropriate manual integration. It may be necessary to integrate other levels also to keep the integration consistent. If it is not possible to meet the criterion while maintaining the same baseline, make any adjustments necessary and recalibrate the instrument. If method acceptance criteria are met, but the software's integration for a peak is inconsistent with the ICAL integration at each level and performing a manual integration would not cause the ICAL to be unacceptable, do not manually integrate. It is the intent of the laboratory to minimize the amount of manual integrations performed by the chemist. Manual integrations should be signed and dated in the "Flag?" column such that they appear on the Raw Data. Manual integration review is performed at the time of final report review and the signature of such is included on the multilevel.

- 15.L.1 MI1) Integration does not follow baseline
- 15.L.2 MI2) Non-target peak interference
- 15.L.3 MI3) To split a peak that was integrated as one peak by the computer.
- 15.L.4 MI4) To integrate a split peak
- 15.L.5 MI5) The whole peak or part of the peak was not integrated.
- 15.L.6 MI6) Computer integrated wrong peak
- 15.L.7 MI7) Other – (See case narrative)

15.M After review by the section manager, the manager will date and initial. Upon client request, the integrations will be reviewed by the QAU or his/her designee initialed and dated. The hard copies will be filed with the raw data. See SOP QC044 Manual Integration for more information.

16 Data Assessment and Acceptance Criteria for QC

16.A The analyst first reviews data by checking the following against the acceptance criteria listed in section 8.0 Quality Control and section 11.0 Calibration. For DoD project acceptance criteria, consult the DoD QSM PFAS Table B-15, which is listed in this SOP as Table 4.

- 16.A.1 The initial calibration curve
- 16.A.2 The initial calibration verification and continuing calibration verification %D
- 16.A.3 The LLCCV recovery
- 16.A.4 The instrument (initial and continuing) blanks
- 16.A.5 The method blank
- 16.A.6 The lab spike recovery and precision
- 16.A.7 The matrix spike recovery and precision
- 16.A.8 Surrogate and Internal Standard %R
- 16.A.9 S/N Ratios
- 16.A.10 Asymmetry Factor
- 16.A.11 Ion Ratios

16.B The MultiQuant™ quantitation software automatically flags any data point that does not meet acceptance criteria, such as: ICAL, CCV, I.S., Surrogate, S/N Ratio, Isomer Ratios etc.

16.C The technical reason for flagging the data point will be listed under the "Failure Reason" column in the software.

16.D All software flags should be critically reviewed by the analyst to prevent errors resulting from inaccurate software integrations or determinations.

16.E The analyst reviews data and assembles the final report, in accordance with the Multilevel Quality Control Sign Off Worksheet, as discussed in APPL's SOP QC045 Data Integrity and Final Review. The analyst signs the MQCS sheet before secondary peer review. See Table 5 of this SOP for a copy of the PFAS MQCS.

16.F If at any point the review shows an out of control situation, complete an NWR form. See SOP QC033 Non-conforming Work Report. The analyst notifies the section manager, and the problem is investigated. The section manager notifies the project manager regarding any QC failures, so that the client is aware of any data integrity issues prior to receiving the final report.

16.G The corrective action for resolving QC issues may be one of several points considered; standard preparation, improper injection size, extraction technique, etc. The problem is potentially solved and reanalysis or re-extraction/reanalysis is performed if possible. If re-extraction is not possible due to limited volume etc, flag the reported data accordingly and notify the client.

16.H For DoD projects, consult Table 4 of this SOP for corrective actions and appropriate flagging criteria

16.I When QC parameters are exceeded, the following takes place:

- 16.I.1 For matrix spike criteria that are exceeded, contact the project manager who will contact the client as to additional measures to be taken. For the specific analyte(s) in the parent sample, apply appropriate flags if acceptance criteria are not met If the MS/MSD.
- 16.I.2 For Blank, LCS, IS, or Surrogate criteria that are exceeded, re-inject or re-extract as needed. Failing method blanks or LCS may require re-extraction of the entire sample batch, if the issue can not be resolved through re-injection or client approval, based on the project DQOs.
- 16.I.3 If the instrument calibration parameters are not met, the standards are re-prepared and re-analyzed. No sample analysis may take place with an out of compliance ICAL or ICV, based on the criteria listed in section 11 of this SOP.

16.J All pertinent information on the NWR and MQCS is included in the case narrative.

17 Corrective Actions and Contingencies for Out of Control Data or Unacceptable Data

18 In the event that an out of control situation occurs, the project manager will be notified immediately. The affect of the out of control situation will be assessed according to the project DQO. If sufficient sample remains, and the situation will significantly affect the quality of the results, the analysis will be repeated. If the situation does not significantly affect the quality of the data, the project manager will notify the client and instructions from the client will be followed. In the event no sample remains, the client will be notified immediately. All situations will be

documented on the multi level sheet and initialed by the project manager. All out of control situations will be brought to the attention of the QAU in the form of a NWR. The QAU has the final authority to approve the actions taken

19 Method Deviations

19.A This SOP was compared to EPA method 537.1. The following deviation were made:

19.A.1 EPA 537.1 requires that an initial demonstration of low background is performed whenever a new lot of cartridges, solvents, centrifuge tubes, pipettes, and injection vials are used.

APPL, Inc. procedure and justification: The lab will analyze a method blank with each preparatory batch in order to demonstrate that the procedure is free from contaminants. This will continuously monitor contamination within each new lot, rather than one IDOC study at the start of a new lot. All samples in the batch will be re-extracted if method blank contamination is present.

19.A.2 EPA 537.1 requires an initial demonstration of accuracy and precision to be performed using 4-7 lab control spikes spiked near the mid-point of the curve. The accuracy must be within 70-130% of the true amount and the RSD% less than 20%.

APPL, Inc. procedure and justification: The lab's standard practice incorporates accuracy and precision checks as a part of the initial demonstration of capability, which is done in accordance with DoD QSM Four spikes are used which are spiked at 1-4X the LOQ.

19.A.3 EPA 537.1 allows for an RPD between a sample and sample duplicate to be ≤50%, if the concentration of the detected analyte is below 2X the LOQ. For the low spiked MS/MSD, an RPD of ≤50% is acceptable.

APPL Inc procedure and justification: Due to limited LIMS capability for only one set of acceptance criteria, the %RPD will be flagged for any RPD >30%. This criteria is more stringent than the method requirement.

20 Pollution Prevention

20.A All hazardous materials that are generated during the testing of samples must be properly collected and stored. Drums are available in the storage room for the following types of wastes- acidic, basic and solvents.

21 Waste Management

21.A Spent sample extracts and standards / surrogates / spikes are disposed in the LC Waste Drum (Profile ID 312936 Acetonitrile/water/methanol).

21.B It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and lands disposal restrictions. The laboratory has the responsibility to protect the environment by minimizing and controlling all releases from fume hoods and bench operations.

22 Method Performance

22.A Continuing method performance is monitored by analysis of LCS samples with each batch and control charting the results as per SOP# QC016.

22.B A method detection Limit (MDL) study is run to ensure the performance of the instrumentation is able to satisfy data quality objectives of the client by reaching the reporting limits necessary. An MDL study is performed for each matrix per instrument after major instrument changes take place, such as a column change and is performed in accordance with SOP# QC018.

22.C The method is not performed by any analyst until a Demonstration of Capability (DOC) is completed. Every analyst who performs this method has demonstrated acceptable accuracy and precision by passing a Demonstration of Capability study.

23 Equipment / Instrument Maintenance and Troubleshooting

23.A The LCMSMS is under service contract with SCIEX. For any major problems, it is best to allow certified technicians to work on the instrument. Technical Support Telephone 1-877-740-2129.

23.B If the Tune Check fails, SCIEX must be contacted and a certified technician will come out and perform an instrument tune. If the instrument tune does not resolve the problem, the SCIEX technician will perform a mass calibration. At a minimum the mass calibration is performed by the instrument manufacturer during the annual periodic maintenance schedule.

23.C More detailed maintenance and instructions for the software may be found at https://sciex.com/support/training or using the help menu in the instrument software.

23.D For reoccurring problems, the instrument Log Book located in H:\Saphira may be referenced for troubleshooting help.

23.E The pumps on the instrument must be purged of air bubbles whenever the mobile phases are changed out. To purge the pump, open the pump by twisting the knob on the door counter-clockwise. Select the purge option. This will purge for about 2 minutes. The knob can then be tightened.

23.F If there appears to be problems with the pump seals, the pumps can be purged with isopropyl alcohol and then purged with the mobile phases.

23.G The ion source, curtain plate, orifice plate on the mass spec may be wiped down gently with isopropyl while the system is under vacuum.

23.H For further cleaning of the mass spec, the vacuum must be released by holding the vent button on the side of the instrument down for 3 secs. Allow the system to vent for 15-20 mins.

23.I To pump back down, hold the reset button for 5 secs and allow the vacuum to run for about 30 mins.

23.J For any problems that are not resolved with the maintenance listed above need to be reported to SCIEX for further support.

24 Computer Hardware and Software

24.A Analyst®

24.A.1 This software is used in acquiring the data from the LCMSMS.

24.A.2 Most questions can be answered by accessing the 'Help' section of the software.

24.B MultiQuant™

24.B.1 This software is used in processing the data from the LCMSMS.

24.B.2 Most questions can be answered by accessing the 'Help' section of the software.

24.C The following Computer capabilities are needed to run the Analyst® and MultiQuant™ software are at minimum

25 References

25.A Method 537 Revision 1.11 Determination of Selected Perfluorinated Alkyl Acids in Drinking Water By Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry, Version 1.1, 2009.
25.B Method 537.1 Version 1.0 November 2018, Determiniation of Selected PFAS substances in Drinking water by SPE and LCMS-MS
25.C Water quality – Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry, ISO25101, 1st Edition, 2009.
25.D DoD /DoE QSM, Version 5.3, May 2019
25.E ISO/IEC 17025:2017
25.F https://sciex.com/support/training
25.G www.waterboards.ca.gov/drinking_water/certlic/drinkingwater/PFOA_PFOS.html

26 Tables, Diagrams, Flowcharts, Validation Data

Table 1: LOQ, LOD, MDL, CLs for 537.1 samples
Table 2: LOQ, LOD, MDL, CLs for QSM TB-15 water samples
Table 3: LOQ, LOD, MDL, CLs for QSM TB-15 solid samples
Table 4 : LCMSMS Transition list
Table 5: DoD QSM PFAS Table B-15

Section Manager Name: Hart Hedgpeth

Section Manager Signature:  Date: 08/31/2022

QAU Director Name: Pamela Cooper

QAU Director Signature: _____ Date: 08/31/2022

Standard Operating Procedure

Given Short Name	Known Synonyms	Given long name	Cas	EPA 537.1	EPA 533	QSM 5.3 B-15
PFBA	HFBA, PFBuA	Perfluorobutanoic acid	375-22-4		IIS/EIS	EIS
PFPeA		Perfluoropentanoic acid	2706-90-3		EIS	EIS
PFHxA		Perfluorohexanoic acid	307-24-4	SUR	EIS	EIS
PFHpA		Perfluoroheptanoic acid	375-85-9	x	EIS	EIS
PFOA		Perfluorooctanoic acid	335-67-1	IS	IIS/EIS	EIS
PFNA		Perfluorononanoic acid	375-95-1	x	EIS	EIS
PFDA		Perfluorodecanoic acid	335-76-2	SUR	EIS	EIS
PFUnA	PFUA	Perfluoroundecanoic acid	2058-94-8	x	EIS	EIS
PFDoA	PFDoDA	Perfluorododecanoic acid	307-55-1	x	EIS	EIS
PFTrDA	PFTriDA	Perfluorotridecanoic acid	72629-94-8	x		x
PFTeDA	PFTA	Perfluorotetradecanoic acid	376-06-7	x		EIS
PFHxDA		Perfluorohexadecanoic acid	67905-19-5			x
PFODA		Perfluorooctadecanoic acid	16517-11-6			EIS
PFBS	PFBuS	Perfluorobutanesulfonic acid	375-73-5	x	EIS	EIS
PFPeS		Perfluoropentanesulfonic acid	2706-91-4		x	x
PFHxS		Perfluorohexanesulfonic acid	355-46-4	x	EIS	EIS
PFHpS		Perfluoroheptanesulfonic acid	375-92-8		x	x
PFOS		Perfluorooctanesulfonic acid	1763-23-1	IS	IIS/EIS	EIS
PFNS		Perfluorononanesulfonic acid	68259-12-1			x
PFDS		Perfluorodecanesulfonic acid	335-77-3			x
PFDoS	PFDoDS	Perfluorododecanesulfonic acid	79780-39-5			x
PFBSA	FBSA	Perfluorobutanesulfonamide	30334-69-1			x
PFHxSA	FHxSA	Perfluorohexanesulfonamide	41997-13-1			x
PFOSA	FOSA	Perfluorooctanesulfonamide	754-91-6			EIS
NMeFOSA		N-Methyl perfluorooctanesulfonamide	31506-32-8			EIS
NEtFOSA		N-Ethyl perfluorooctanesulfonamide	4151-50-2			EIS
NMeFOSAA		N-Methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	IS		EIS
NEtFOSAA		N-Ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	SUR		EIS
NMeFOSE		N-Methyl perfluorooctanesulfonamidoethanol	24448-09-7			EIS
NEtFOSE		N-Ethyl perfluorooctanesulfonamidoethanol	1691-99-2			EIS
3:3FTcA	FPrPA; 33FTA; 3:3FTCA	2H,2H,3H,3H-Perflurohexanoic acid	356-02-5			x
5:3FTcA	FPePA; 53FTA; 5:3FTCA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3			x
7:3FTcA	FHpPA; 73FTA; 8:3FTCA	2H,2H,3H,3H-Perflurodecanoic acid	812-70-4			x
4:2FTS	42FTSA	1H,1H,2H,2H-Perfluorohexanesulfonic acid	757124-72-4		EIS	EIS
6:2FTS	62FTSA	1H,1H,2H,2H-Perfluorooctanesulfonic acid	27619-97-2		EIS	EIS
8:2FTS	82FTSA	1H,1H,2H,2H-Perfluorodecanesulfonic acid	39108-34-4		EIS	EIS
10:2FTS	102FTSA	1H,1H,2H,2H-Perfluorododecanesulfonic acid	120226-60-0			EIS
PFMPA (533)	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1		x	x
PFMBA (533)	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5		x	x
NFDHA (533)	PF3,6OHpA	Nonaflluoro-3,6-dioxaheptanoic acid	151772-58-6		x	x
HFPO-DA		Hexafluoropropylene oxide dimer acid	13252-13-6	SUR	EIS	EIS
ADONA		4,8-dioxa-3H-perfluorononanoic acid	919005-14-4	x	x	x
PFEESA (533)	PF3OHxS	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7		x	x
9Cl-PF3ONS		9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	x	x	x
11Cl-PF3OUdS		11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	x	x	x

Standard Operating Procedure

TABLE 1
LOQ, LOD, MDL, CLs for 537.1 samples (50mL)

Analyte	LOQ (ng/L)	LOD (ng/L)	MDL (ng/L)	CLs (%)
Perfluoro-n-hexanoic acid (PFHxA)[A]	2	1.2	0.46	70-130
Perfluoro-n-heptanoic acid (PFHpA)[A]	2	1.7	0.69	70-130
Perfluoro-n-octanoic acid (PFOA)[A]	2	1.2	0.47	70-130
Perfluoro-n-nonanoic acid (PFNA)[B]	2	1.5	0.59	70-130
Perfluoro-n-decanoic acid (PFDA)[B]	2	1	0.37	70-130
Perfluoro-n-undecanoic acid (PFUdA)[B]	2	1	0.38	70-130
Perfluoro-n-dodecanoic acid (PFDoA)[B]	2	1	0.23	70-130
Perfluoro-n-tridecanoic acid (PFTrDA)[B]	2	1	0.27	70-130
Perfluoro-n-tetradecanoic acid (PFTeDA)[B]	2	1	0.19	70-130
N-methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)[C]	2	1.1	0.43	70-130
N-ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)[C]	2	1.2	0.46	70-130
Perfluoro-1-butanesulfonate (PFBS)[A]	2	1	0.27	70-130
Perfluorohexanesulfonate (PFHxS)[A]	2	1	0.29	70-130
Perfluorooctanesulfonate (PFOS)[B]	2	1	0.27	70-130
Hexafluoropropylene oxide dimer acid (HFPO-DA)	6	1	0.27	70-130
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid(11-Cl-PF3OudS)	4	1	0.36	70-130
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid(9-Cl-PF3ONS)	4	1	0.35	70-130
4,8-Dioxa-3H-perfluorononanoic acid (NaDONA)	4	1.3	0.50	70-130
Surrogates				
13C2-PFHxA[D]	NA	NA	NA	70-130
13C2-PFDA[E]	NA	NA	NA	70-130
d5_EtFOSAA[E]	NA	NA	NA	70-130
Internal Standards				
13C2_PFOA	NA	NA	NA	Section 8
d3_MeFOSAA	NA	NA	NA	Section 8

A=the mass-labeled surrogate for these analytes is 13C2-PFHxA

B=the mass-labeled surrogate for these analytes is 13C2-PFDA

C=the mass-labeled surrogate for these analytes is d5_EtFOSAA

D=the mass-labeled internal standard for this surrogate is 13C2_PFOA

E=the mass-labeled internal standard for these surrogates is d3_MeFOSAA

TABLE 2
LOQ, LOD, MDL, CLs for QSM B-15 (537.1M) water samples (50mL)

Analyte	LOQ (ng/L)	LOD (ng/L)	MDL (ng/L)	CLs (%)
Perfluoro-n-butanoic acid (PFBA)	16	1	0.25	70-130
Perfluoro-n-pentanoic acid (PFPeA)	8	1	0.31	70-130
Perfluoro-n-hexanoic acid (PFHxA)	4	1	0.32	70-130
Perfluoro-n-heptanoic acid (PFHpA)	4	1	0.25	70-130
Perfluoro-n-octanoic acid (PFOA)	4	1	0.41	70-130
Perfluoro-n-nonanoic acid (PFNA)	4	1	0.25	70-130
Perfluoro-n-decanoic acid (PFDA)	4	1	0.25	70-130
Perfluoro-n-undecanoic acid (PFUdA)	4	1	0.40	70-130
Perfluoro-n-dodecanoic acid (PFDoA)	4	1	0.25	70-130
Perfluoro-n-tridecanoic acid (PFTrDA)[E]	4	1	0.29	70-130
Perfluoro-n-tetradecanoic acid (PFTeDA)	4	1	0.43	70-130
Perfluoro-1-octanesulfonamide (PFOSA)	16	1	0.25	70-130
N-methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)	16	10	4.9	70-130
N-ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)	16	10	4.9	70-130
Perfluoro-1-butanesulfonate (PFBS)	4	1	0.25	70-130
Perfluoro-1-pentanesulfonate (PFPeS)[A]	4	1	0.29	70-130
Perfluorohexanesulfonate (PFHxS)	4	1	0.25	70-130
Perfluoro-1-heptanesulfonate (PFHpS)[B]	4	1	0.28	70-130
Perfluorooctanesulfonate (PFOS)	4	1	0.25	70-130
Perfluoro-1-nonanesulfonate (PFNS)[C]	4	2	1.2	70-130
Perfluoro-1-decanesulfonate (PFDS)[D]	4	1	0.32	70-130
4:2 Fluorotelomer sulfonate (4:2 FTS)	16	2	0.54	70-130
6:2 Fluorotelomer sulfonate (6:2 FTS)	16	1	0.45	70-130
8:2 Fluorotelomer sulfonate (8:2 FTS)	16	3	1	70-130
Hexafluoropropylene oxide dimer acid (HFPO-DA)	8	5	2.4	70-130
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid(11-Cl-PF3OudS)	8	3	1.2	70-130
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid(9-Cl-PF3ONS)	8	3	1.2	70-130
1.3	8	3	1.3	70-130
Surrogates				
13C4_PFBA[F]	NA	NA	NA	50-150
13C5_PFPeA[F]	NA	NA	NA	50-150
13C3_PFBS[F]	NA	NA	NA	50-150
13C5_PFHxA[F]	NA	NA	NA	50-150
13C2_4:2 FTS[F]	NA	NA	NA	50-150
13C4_PFHpA[G]	NA	NA	NA	50-150
13C3_PFHxS[G]	NA	NA	NA	50-150
13C8_PFOA[G]	NA	NA	NA	50-150
13C2_6:2FTS[G]	NA	NA	NA	50-150
13C9_PFNA[G]	NA	NA	NA	50-150
13C8_PFOSA[G]	NA	NA	NA	50-150
13C8_PFOS[H]	NA	NA	NA	50-150

TABLE 2 - Continued
LOQ, LOD, MDL, CLs for QSM TB-15 water samples

Analyte	LOQ (ug/Kg)	LOD (ug/Kg)	MDL (ug/Kg)	CLs (%)
13C6_PFDA[J]	NA	NA	NA	50-150
13C2_8:2FTS[J]	NA	NA	NA	50-150
13C7_PFUdA[J]	NA	NA	NA	50-150
d3_MeFOSAA[J]	NA	NA	NA	50-150
d5_EtFOSAA[J]	NA	NA	NA	50-150
13C2_PFDoA[J]	NA	NA	NA	50-150
13C2_PFTeDA[J]	NA	NA	NA	50-150
Internal Standards				
13C3_PFBA	NA	NA	NA	Section 8
13C2_PFOA	NA	NA	NA	Section 8
13C4_PFOS	NA	NA	NA	Section 8
13C2_PFDA	NA	NA	NA	Section 8

A=the mass-labeled surrogate for this analyte is 13C2_4:2 FTS

B=the mass-labeled surrogate for this analyte is 13C2_6:2 FTS

C=the mass-labeled surrogate for this analyte is 13C2_8:2 FTS

D=the mass-labeled surrogate for this analyte is d5_EtFOSAA

E=the mass-labeled surrogate for this analyte is 13C2_PFTeDA

F=the mass-labeled internal standard for these surrogates is 13C3_PFBA

G=the mass-labeled internal standard for these surrogates is 13C2_PFOA

H=the mass-labeled internal standard for this surrogate is 13C4_PFOS

J=the mass-labeled internal standard for these surrogates is 13C2_PFDA

TABLE 3
LOQ, LOD, MDL, CLs for QSM B-15 soil samples (1g)

Analyte	LOQ (ug/Kg)	LOD (ug/Kg)	MDL (ug/Kg)	CLs (%)
Perfluoro-n-butanoic acid (PFBA)	1	0.4	0.1	70-130
Perfluoro-n-pentanoic acid (PFPeA)	1	0.4	0.15	70-130
Perfluoro-n-hexanoic acid (PFHxA)	1	0.4	0.1	70-130
Perfluoro-n-heptanoic acid (PFHpA)	1	0.4	0.1	70-130
Perfluoro-n-octanoic acid (PFOA)	1	0.4	0.15	70-130
Perfluoro-n-nonanoic acid (PFNA)	1	0.4	0.1	70-130
Perfluoro-n-decanoic acid (PFDA)	1	0.4	0.15	70-130
Perfluoro-n-undecanoic acid (PFUdA)	1	0.4	0.1	70-130
Perfluoro-n-dodecanoic acid (PFDoA)	1	0.4	0.15	70-130
Perfluoro-n-tridecanoic acid (PFTrDA)[E]	1	0.4	0.1	70-130
Perfluoro-n-tetradecanoic acid (PFTeDA)	1	0.4	0.2	70-130
Perfluoro-1-octanesulfonamide (PFOSA)	1	0.4	0.1	70-130
N-methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)	1	0.4	0.2	70-130
N-ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)	1	0.4	0.2	70-130
Perfluoro-1-butanesulfonate (PFBS)	1	0.4	0.1	70-130
Perfluoro-1-pentanesulfonate (PFPeS)[A]	1	0.9	0.41	70-130
Perfluorohexanesulfonate (PFHxS)	1	0.4	0.15	70-130
Perfluoro-1-heptanesulfonate (PFHpS)[B]	1	0.4	0.15	70-130
Perfluorooctanesulfonate (PFOS)	1	0.4	0.1	70-130
Perfluoro-1-nonanesulfonate (PFNS)[C]	1	0.8	0.39	70-130
Perfluoro-1-decanesulfonate (PFDS)[D]	1	0.4	0.2	70-130
4:2 Fluorotelomer sulfonate (4:2 FTS)	1	0.4	0.2	70-130
6:2 Fluorotelomer sulfonate (6:2 FTS)	1	0.4	0.2	70-130
8:2 Fluorotelomer sulfonate (8:2 FTS)	1	0.4	0.15	70-130
Hexafluoropropylene oxide dimer acid (HFPO-DA)	1	0.4	0.2	70-130
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid(11-Cl-PF3OudS)	1	0.4	0.2	70-130
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid(9-Cl-PF3ONS)	1	0.4	0.2	70-130
4,8-Dioxa-3H-perfluorononanoic acid (NaDONA)	1	0.4	0.2	70-130
Surrogates				
13C4_PFBA[F]	NA	NA	NA	50-150
13C5_PFPeA[F]	NA	NA	NA	50-150
13C3_PFBS[F]	NA	NA	NA	50-150
13C5_PFHxA[F]	NA	NA	NA	50-150
13C2_4:2 FTS[F]	NA	NA	NA	50-150
13C4_PFHpA[G]	NA	NA	NA	50-150
13C3_PFHxS[G]	NA	NA	NA	50-150
13C8_PFOA[G]	NA	NA	NA	50-150
13C2_6:2FTS[G]	NA	NA	NA	50-150
13C9_PFNA[G]	NA	NA	NA	50-150

TABLE 3 - Continued
LOQ, LOD, MDL, CLs for QSM B-15soil samples (1g)

Analyte	LOQ (ug/Kg)	LOD (ug/Kg)	MDL (ug/Kg)	CLs (%)
13C8_PFOSA[G]	NA	NA	NA	50-150
13C8_PFOS[H]	NA	NA	NA	50-150
13C6_PFDA[J]	NA	NA	NA	50-150
13C2_8:2FTS[J]	NA	NA	NA	50-150
13C7_PFUdA[J]	NA	NA	NA	50-150
d3_MeFOSAA[J]	NA	NA	NA	50-150
d5_EtFOSAA[J]	NA	NA	NA	50-150
13C2_PFDoA[J]	NA	NA	NA	50-150
13C2_PFTeDA[J]	NA	NA	NA	50-150
Internal Standards				
13C3_PFBA	NA	NA	NA	Section 8
13C2_PFOA	NA	NA	NA	Section 8
13C4_PFOS	NA	NA	NA	Section 8
13C2_PFDA	NA	NA	NA	Section 8

A=the mass-labeled surrogate for this analyte is 13C2_4:2 FTS

B=the mass-labeled surrogate for this analyte is 13C2_6:2 FTS

C=the mass-labeled surrogate for this analyte is 13C2_8:2 FTS

D=the mass-labeled surrogate for this analyte is d5_EtFOSAA

E=the mass-labeled surrogate for this analyte is 13C2_PFTeDA

F=the mass-labeled internal standard for these surrogates is 13C3_PFBA

G=the mass-labeled internal standard for these surrogates is 13C2_PFOA

H=the mass-labeled internal standard for this surrogate is 13C4_PFOS

J=the mass-labeled internal standard for these surrogates is 13C2_PFDA

Table 4 : LCMSMS Transition list

	Quant. Q1->Q3	Conf. Q1-Q3
PFBA	212.9->169	N/A
PFPeA	262.9->219	N/A
PFHxA	313->269	313->119
PFHpA	363->319	363->169
PFHpA	363->319	363->169
PFOA	413->369 [1]	413->169
PFNA	463->419	463->169
PFDA	513->469	513->169
PFUdA	563->519	563->169
PFDoA	613->569	613->169
PFTrDA	663->619	663->169
PFTeDA	713->669	713->169
PFHxDA	813->769	813->169
PFODA	913->869	913->169
3:3FTA	241->117	241->63
5:3FTA	341->236.7	341->217
7:3FTA	441->317	441->337
PFBS	298.9->80 [1,2]	298.9->99
PFPeS	349->80	349->99
PFHxS	399->80 [1,2]	399->99
PFHpS	449->80	449->99
PFOS	499->80 [1,2]	499->99
PFNS	549->80	549->99
PFDS	599->80	599->99
PFDoS	698.9->80	698.9->99
4:2 FTS	327->307 [1]	327->81
6:2 FTS	427->407 [1]	427->81
8:2 FTS	527->507 [1]	527->81
10:2FTS	628->608	628->81

[1]Quant transitions listed in the DoD QSM as required transitions. If they are not to be used, technical justification must be given (e.g. alternative transition was used due to observed interferences).
[2]These transitions must be used for EPA 537.1

	Quant. Q1->Q3	Conf. Q1-Q3
PFMPA	229->85	N/A
PFMBA	279->85	N/A
PFSBA	298->78	N/A
PFEESA	315->135	N/A
NFDHA	295->201	201->85
FHxSA	398->78	N/A
HFPO-DA	285->169	329->185
ADONA	377->85	377->251
9Cl-Pf3ONS	531->351	533->353
11Cl-PF3OUDS	631->451	633->453
PFOSA	498->78	N/A
N-MeFOSA	511.9->219	511.9->169
N-EtFOSA	526->219	526->169
N-MeFOSAA	570->419 [1]	570->483
N-EtFOSAA	584->419 [1]	584->526
N-MeFOSE	616.1->59	N/A
N-EtFOSE	630->59	N/A

[1]Quant transitions listed in the DoD QSM as required transitions. If they are not to be used, technical justification must be given (e.g. alternative transition was used due to observed interferences).

IS	Q1->Q3
13C3_PFBA_IIS	216->172
13C4_PFBA_EIS	217->172
13C5_PFPeA_EIS	267.9->223
13C3_PFBS_EIS	302->80
13C2_4:2 FTS_EIS	329->81
13C3_HFPODA_EIS	287->169
13C2_PFHxA_IIS	315.1->270
13C5_PFHxA_EIS	318->273
13C4_PFHpA_EIS	367->322
13C3_PFHxS_EIS	402->80
18O2_PFHxS_IIS	403->83.9
13C2_6:2FTS_EIS	429->81
13C2D4_6:2FTS_EIS	433->412
13C2_PFOA_IIS	415->370
13C8_PFOA_EIS	421->376
13C5_PFNA_IIS	468->423
13C9_PFNA_EIS	472->427
13C4_PFOS_IIS	502.8->79.9
13C8_PFOS_EIS	507->80
13C2_8:2FTS_EIS	529->81
13C2_PFDA_IIS	515.1->470.1
13C6_PFDA_EIS	519->474
13C8_PFOSA_EIS	506->78
D3_MeFOSAA_EIS	573->419
13C7_PFUdA_EIS	570->525
D5_EtFOSAA_EIS	589->419
13C2D4_10:2FTS_EIS	633->612
D7_NMeFOSE_EIS	623.2->58.9
D3_NMeFOSA_EIS	515->169
13C2_PFDoA_EIS	615->570
D9_NEtFOSE_EIS	639.2->58.9
D5_NEtFOSA_EIS	531.1->169
13C2_PFTeDA_EIS	715->670
13C2_PFHxDA	815->770

Standard Operating Procedure

TABLE 5

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
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.
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.
Biota Sample Preparation	Each sample and associated batch QC samples.	Sample prepared as defined by the project (e.g., whole fish versus fileted fish).	NA.	NA.	NA.

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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.
Sample Cleanup Procedure	Each sample and associated batch QC samples. Not applicable to AFFF and AFFF Mixture Samples.	Removal of interferences from matrix. ENVI-CarbTM or equivalent must be used on each sample and batch QC sample.	NA.	Flagging is not appropriate.	Cleanup should reduce bias from matrix background.
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).

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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.
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 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	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.

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
standard.					
Sample PFAS Identification	Identify all positive sample detections per method.	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%. S/N ≥ 10:1 for all ions used for quantification and S/N ≥ 3:1 for all ions used for confirmation. Quant ion and confirmation ion must be present and must maximize simultaneously (±2 seconds).	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.

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Ion Transitions (Precursor-> Product)	Identify all positive sample detections per method.	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.
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	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ICAL has passed. e.g., PFHxS and PFOS are not available as

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		(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 Extracted Internal Standard Analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (Internal Standard Quantitation) 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. Analytes must be within 70-130% of their true value for each calibration standard.			sulfonates but as their corresponding salts, such as Na+ and K+. Isotope Dilution is required for all analytes. External Calibration is not allowed. Calibration can be linear (minimum of 5 standards) or quadratic (minimum of 6 standards); weighting is allowed.
Retention Time 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

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time	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.
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.
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.

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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.
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 carry over still does not occur.

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Extracted Internal Standard Analytes (EIS)	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.
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.

TABLE 5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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.
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 Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	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).

TABLE5, continued

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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 saple is ≥LOQ. The MD is a second aliquot of the field sample that has been prepared by serial dilution.
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 70-130% 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.

Extractions for Aqueous, Solid, AFFF and Biota PFAS Samples
By EPA METHOD 537.1 and 533, 1633 and DoD/DoE QSM Table B-15

1 Purpose

1.A The purpose of this SOP is to describe the procedure for extracting PFAS analytes from aqueous samples using solid phase extraction (SPE) media or serial dilution and solid sample using solid liquid extraction (SLE), prior to instrumental analysis using EPA method 537.1, 533, or APPL's HPLC-B-15.

2 Scope and Application

2.A This SOP applies to all personnel involved in the cartridge solid phase extraction of PFAS in aqueous samples according to EPA 537.1, EPA 533, EPA 1633, as well as, the solid sample liquid extraction according to, and in compliance with DoD QSM Table B-15.

2.B This method is restricted to use by or under the supervision of personnel who have been trained according to this SOP as PFAS extraction technicians for the purpose of preparing extracts from environmental water and soil samples. The extracts are analyzed by LCMS-MS according to SOP HPL537 by a trained instrument analyst. Each preparatory technician must demonstrate the ability to generate acceptable results with this method.

3 Method Summary

3.A EPA 537.1**:** A water sample of approximately 250mL (unless a clients project requirements specify otherwise) is fortified with isotopologues (surrogates) and passed through a solid phase extraction (SPE) cartridge. The compounds are eluted from the solid phase media and then the extract is concentrated and brought to a final volume of 5mL at 96:4 methanol:water with more isotopologues (internal standards).

3.B EPA 533**:** A water sample of approximately 250mL (unless a clients project requirements specify otherwise) is fortified with isotopologues (Isotope Dilution Analogue) and passed through a solid phase extraction (SPE) cartridge. The compounds are eluted from the solid phase media and then the extract is concentrated and brought to a final volume of 5mL at 80:20 methanol:water with more isotopologues (Isotope Performance Standards).

3.C EPA 1633 and B-15**:** (aqueous) A water sample of approximately 50mL or 250mL is fortified with isotopologues (Extracted internal standards) and passed through a solid phase extraction (SPE) cartridge. The compounds are eluted from the solid phase media and then the extract is concentrated and brought to a final volume of 2mL at 80:20 methanol:water.

3.D EPA 1633: (solid) A soil (5g dry weight) or biota (0.5g dry weight) is fortified with isotopologues (Extracted Internal Standards), submerged in methanol, vortexed, then shaken. This solvent is exchanged into water, cleaned using a WAX SPE procedure. The compounds are eluted from the solid phase media and then the extract is concentrated and brought to a final volume of 2mL at 80:20 methanol:water.

3.E B-15**:** (solid) A soil or biota sample of greater than 0.5g is fortified with isotopologues (Extracted Internal Standards), submerged in methanol, vortexed, then sonicated. The extract is passed through a carbon cartridge and then concentrated and brought to a final volume of 2mL with 80:20 methanol.

3.F B-15: (AFFF and AFFF formulations) A water sample of approximately 0.2mL fortified with isotopologues (Extracted internal standards). The sample is brought to a final volume of 1mL and analyzed; the sample is further diluted with methanol and isotopologues as necessary.

3.G AFFF with SPE: A 0.02mL subsumable is dissolved into 40mL of water, fortified with isotopologues (Extracted internal standards) passed through a solid phase extraction (SPE) cartridge, eluted from the cartridge ammonium hydroxide in methanol. This eluent is cleaned up with activated carbon and neutralized with of glacial acetic acid, centrifuged, and syringe filtered before analysis.

4 **Detection Limits** - NA

5 **Definitions** - See Table 1 below for a crosswalk between APPL SOP terminology and DoD QSM Table B-15 Terminology.

Table 1 Definitions Crosswalk

DoD QSM Table B-15 Terminology	APPL SOP Terminology
IIS (Injection Internal Standard)	IIS
EIS (Extracted Internal Standard)	EIS
EPA 537.1 Terminology	
PDS (Primary Dilution Standard)	Native Standard
IS PDS	IS
LRB	BLK
LFB	LCS
LFSM	MS
LFSMD	MSD
EPA 533 Terminology	
Isotope Performance Standard	IIS
Isotope Dilution Analogue	EIS
EPA 537.1 and 533 Terminology	
LRB	BLK
LFB	LCS
LFSM	MS
LFSMD	MSD
LD	DUP

5.A BLANK: see Equipment Rinse, Method Blank, Trip Blank.

5.B CONTROL SAMPLE: A QC sample introduced into a process to monitor the performance of the system.

5.C FIELD REAGENT BLANK (FRB) – Required for EPA 537.1 and 533 Drinking Water samples. An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment. The State of California, Division of Drinking Water requires a FRB per field site.

5.D LABORATORY CONTROL SAMPLE: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

5.E MATRIX: The component or substrate (e.g., surface water, and drinking water) which contains the analyte of interest.

5.F Matrix duplicate (MD) - Two entire 50 mL containers (for waters) or two aliquots (for soils) of the same sample are prepared and analyzed separately with identical procedures. Analysis of a matrix sample and matrix sample duplicate, indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

5.G Matrix spike (MS) - An entire 50 mL container (for water) or aliquot (for soil) of an environmental sample to which known quantities of the method analytes are added in the laboratory. The matrix spike is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the matrix spike corrected for background concentrations.

5.H Matrix spike duplicate (MSD) - Two entire 50 mL containers (for waters) or two aliquots (for soils) of the same sample to which known quantities of the method analytes are added in the laboratory are prepared and analyzed separately with identical procedures. Analysis of a matrix spike and matrix spike duplicate, indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

5.I METHOD BLANK: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete

sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

5.J MRL (Method Reporting Limit): Practical reporting limit based on the lowest calibration point in the ICAL

5.K PPB (Parts Per Billion): The references to "ppb" in this SOP correspond to internal standard, surrogate, spike mix, or calibration mix concentrations and are equivalent to the units ng/mL or ug/L.

5.L PSS (Post Spike): Required for DoD projects when aqueous samples are prepared by serial dilution instead of SPE and have reported analyte concentrations <LOQ. A known amount of target analyte(s) is added to the sample at the LOQ concentration and the % recovery is evaluated.

5.M Surrogate(s) (Surr): Isotopically labeled analyte(s) that are added to a field samples or QC samples prior to extraction. Surrogate recovery is used to confirm the effectiveness of extraction, but not to correct for losses.

5.N Extracted Internal Standard(s) (EIS)- Isotopically labeled analyte(s) that are added to a field samples or QC samples prior to extraction. These standards are used to perform isotope dilution, or extracted internal standard quantification when an exact isotopologue is not available.

5.O Injected Internal Standard(s)- Isotopically labeled analyte(s) that are added after concentration and before brining samples to final volume. These are used for quantification of either native analytes (537.1) or EIS.

5.P TB (Trip Blank): Required by the State of California Division of Drinking Water per ice chest. An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the TB is to determine if method analytes or other interferences are present during shipping and handling.

6 Interferences and Potential Problems

6.A All labware must be meticulously cleaned. Wash labware with detergent and tap water, rinse with tap water, followed by a reagent water rinse. If the glassware is to be used for methanol, a final rinse with HPLC grade methanol is recommended Do not cover with aluminum foil because PFAAs can be potentially transferred from the aluminum foil to the glassware.

6.B Method interference's 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 target 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, SPE sample transfer lines, etc. All items such as these must be routinely demonstrated to be free from interferences (less than 1/3 the MRL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent blanks.

6.C Matrix interference may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from source to source, depending upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent.4-5 Total organic carbon (TOC) is a good indicator of humic content of the sample. Under the LC conditions used during method development, matrix effects due to total organic carbon (TOC) were not observed.

6.D Relatively large quantities of the preservative are added to sample bottles for EPA 537.1 drinking water analysis. The potential exists for trace-level organic contaminants in these reagents. Interference from these sources should be monitored by analysis of laboratory reagent blanks with each preparatory batch, particularly when new lots of reagents are acquired.

6.E SPE cartridges can be a source of interference. The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

6.E.1 NOTE: PFAA standards, extracts and samples should not come in contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAA analyte, IS (Internal Standards) and SUR (Surrogate) standards commercially purchased in glass ampules are

acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers.

6.F APPL understands that some compounds are liable at higher temperatures and without the protection of Water and or/ MeOH. Notably, this includes the sulfonamidoacetic acid, sulfonamides, sulfonamido ethanol, and fluorotelomer carboxylates. Until it is established exactly how best to concentrate extracts, a lower temperature is employed and samples are not brought to/near dryness, instead stopping at or just below final volume.

7 **Health and Safety** - Latex or rubber gloves, lab coat, and safety glasses must be worn for this extraction procedure.

8 Sample Preservation, Containers, Handling and Storage

8.A QSM Table B-15, non-preserved non-potable water samples are collected in a polypropylene 50mL container and stored at 4 °C ± 2 °C.

8.A.1 The samples must be extracted within 28 days of collection and analyzed within 90 days of extraction.

8.A.2 For WWTP samples in the State of California, field blanks and equipment blanks are required, using PFAS-free reagent water.

8.B QSM Table B-15, soil samples are collected in a 50mL unpreserved polypropylene container and stored at 4 °C ± 2 °C.

8.B.1 The samples must be extracted within 28 days of collection and analyzed within 90 days of extraction.

8.C For 537.1, the drinking water samples are collected in a polypropylene 250 mL container containing approximately 1.25g of Tris. The 250mL sample containers with pre-weighed Tris will be purchased from ESS Catalogue 0250-1901-QC-1.25G TRZ, or equivalent. Samples must be stored at or below 6 °C upon arrival. Samples should never be frozen.

8.C.1 The samples must not exceed 10 °C during the first 48 hrs after collection. Upon arrival, the temperature must be confirmed to be below 10 °C. Notify the project manager, who will notify the client if the temperature has been exceeded upon arrival.

8.C.2 The drinking water samples must be extracted within 14 days of collection. Extracts must be stored at room temperature and analyzed within 28 days of extraction.

8.C.3 A Field Reagent Blank (FRB) is required for each sample site at the same time samples are collected. FRB's are required for EPA 537.1 drinking water. Consult the client QAPP for more information regarding the use of FRB for a particular project.

8.C.4 For bottle orders, the lab must send a FRB kit with two 250 mL polypropylene containers: one container will contain the appropriate Tris preservative and filled with reagent water, the other container will be empty. At the sampling site, the contents of the full bottle will be transferred to the empty bottle and the bottle will be labeled as a FRB. This will be sent back to the lab and analyzed with the samples to ensure that PFAS were not introduced into the sample during the collection and handling process. See Appendix A of this SOP for a field instruction sheet, which is included with the FRB kit prepared by APPL.

8.C.5 The Tris preservative used for the FRB must be the same batch or lot number used for the field samples.

8.D The reagent water used for the field blanks must be initially analyzed as a method blank and proven to meet the blank acceptance criteria in SOP HPL537. This ensures that PFAS is not being detected in field blanks because of contaminated reagent water.

8.E For 533, the drinking water samples are collected in a polypropylene 250mL container containing 0.5mL of 0.5g/mL ammonium acetate. Sample bottles are prepared the week of bottle order using a solution prepared by the LCMS annalist stored in the LCSM solvent cabinet. The samples must not exceed 10 °C during the first 48 hrs after collection. Upon arrival, the temperature must be confirmed to be below 10 °C. Notify the project manager, who will notify the client if the temperature has been exceeded upon arrival.

8.E.1 The drinking water samples must be extracted within 28 days of collection. Extracts must be stored at room temperature and analyzed within 28 days of extraction.

8.E.2 A Field Reagent Blank (FRB) is required for each sample site at the same time samples are collected. FRB's are required for EPA 533 drinking water.

8.E.3 For bottle orders, the lab must send a FRB kit with two 250 mL polypropylene containers: one container will contain the appropriate ammonium acetate preservative and, the other container will contain reagent water. At the sampling site, the contents of the full bottle will be transferred to the preserved bottle and the bottle will be labeled as a FRB. This will be sent back to the lab and analyzed with the samples to ensure that PFAS were not introduced into the sample during the collection and handling process. See Appendix B of this SOP for a field instruction sheet, which is included with the FRB kit prepared by APPL.

8.F For TCLP requests on solid samples for PFAS, the hold time is as follows: From collection to TCLP extraction and finishing SPE extraction 28 days. From SPE extraction to instrument analysis 90 days.

9 Quality Control

9.A One method blank, one lab control spike a matrix spike and a duplicate must accompany each set of twenty field samples (or fewer) unless insufficient sample is provided.

9.B If there is insufficient volume to perform a duplicate analysis, then extract an LCSD . For DoD projects, an LCSD is required, if there is insufficient volume for MS/MSD.

10 Equipment/Apparatus

10.A Sample containers, 250mL high density polyethylene or polyproplyene with screw caps
10.B Centrifuge Tubes, 15 mL, polypropylene
10.C Centrifuge Tubes, 50 mL, polypropylene Plug Caps
For use by lab and as sample containers.
10.D 2mL polyproypylene injection vials VWR: 89524-188
10.E 2 mL Screw Top Caps --- Agilent PN: 5191-8151
10.F Water Bath Sonicator Branson 3510 or equivalent
10.G Mini Laboratory Vortex Mixer
10.H Vacuum manifold with bleed valve and tube rack insert
10.I Vacuum trap (large Carboy fitted with stopper and appropriate tubing)
10.J Strata™-XL 100 µm Polymeric Reversed Phase 500mg /6mL Cartridges from Phenomenex
10.K Strata™-X-AW 33 µm Polymeric Weak Anion 200mg/3mL from Phenomenex are currently in use.
10.L Supelco® Supelclean™ ENVI-Carb™ Cartridges from Sigma; or equivalent. 0.2g/3mL preferred.
10.M bulk Supelco® Supelclean™ ENVI-Carb™.
10.N Sample reservoirs, 60mL, polypropylene, luer slip
10.O Adapter cap for Sample Transfer lines
10.P Various Class A Glassware – not limited to volumetric flasks, graduated cylinders, beakers.
10.Q Adjustable Pipettes of various sizes
10.R LC solvent bottle
10.S Dry bath and nitrogen evaporator

11 Reagents and Standards - Reagent grade chemicals are used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the analysis. All reagent and chemical lots will be documented properly for traceability. Certified reference Material standards must be purchased from a body that can provide ILAC-signatory (MRA) traceability and ISO/EC 9001, 17025, and 17034 certification.

11.A Reagents:

11.A.1 Purchased: All purchased reagents must be of high purity and demonstrated to be free of analytes and interference.

[**Tris**]	Trizma®– Preset Crystals, pH 7.0 (Sigma cat# T-7193 or equivalent)
[H_2O]	HPLC Grade Water
[DI]	Deionized Water
[MeOH]	HPLC Grade Methanol
[AmAc]	Anhydrous Ammonium Acetate
[GAA]	Glacial Acetic Acid
[AmOH]	Ammonium Hydroxide (25-30 % ammonia, by mass)
[Na_2HPO_4]	Sodium Phosphate Dibasic
[NaH_2PO_4]	Sodium Phosphate Monobasic

[FA] Formic Acid >99%

11.A.2 Prepared:

[AmAc-H_2O] 25.0 g AmAc, H_2O dissolve to 50-mL.
Store in a 50mL plug cap centrifuge tube at room temperature.

[AmAc-H_2O-d] 2.0mL AmAc-H_2O diluted to 1L with H_2O.

[AmAc buffer] Acetate Buffer- Combine 1.0-mL GAA, 0.7732-mL AmAc-H_2O bring to 1L with H_2O.
Store in clear glass LC solvent bottle at room temperature.

[AmOH-MeOH] Bring 4.0 mL of AmOH to 1L with MeOH.
Store in clear glass LC solvent bottle at room temperature.

[AmOH@0.5-MeOH] Bring 2mL AmOH to 100mL with MeOH.

[PO_4 buffer] 7.098g Na_2HPO_4 and 2.9995g NaH_2PO_4 to 750mL H_2O
Store in clear glass LC solvent bottle at room temperature.

[FA-MeOH:H2O] Dissolve 2.3g FA in 500mL of H_2O, bring to 1L with MeOH

11.A.3 Standards - All spike and surrogates required for this method are to be prepared by the instrument analyst and documented on the Extraction Sheet.

12 Calibration and Standardization - NA

13 Procedures

13.A General

13.A.1 Note: Before starting the procedure for all water samples, the full polypropylene sample container (including the water sample) must be weighed, and the weight recorded on the extraction sheet to the nearest 1g. After performing the SPE extraction, the empty sample containers must be weighed, and the weight recorded as above. The final weight of the sample will be the difference between the full and empty container. This weight is to be recorded on the extraction sheet in grams of water extracted. The density of water 1g / mL.

13.A.2 Note: All applicable information (extraction start date/time, end date/time, lot numbers, spikes, spike amounts, prep dates, reagents, etc.) must be documented on an extraction sheet.

13.A.3 The procedures that the instrument analyst uses for preparing the spike and surrogate mixes are listed in SOP HPL537 section 10.0. The calculations required for spike and surrogate % recovery are listed in sect 13.0 of this SOP. The % recoveries are automatically calculated from the instrument software and LIMS.

13.A.4 A draft hard copy of the extraction sheet is prepared prior to extracting the samples, and it is used by the extraction technician for information and documentation purposes during the extraction process.

13.A.5 The draft sheet documents the spike and surrogate amounts to be used during the extraction process, which corresponds to the spike and surrogate amounts listed in the sample extraction procedures below.

13.A.6 The draft sheet also documents the name, concentration, and prep date of the spike / surrogate mixes, in order to cross reference the mixes used for each preparatory batch with the standard prep log book, located in H:\Saphira\Standard Prep Log Book.

13.A.7 The extraction technician uses the draft sheet as a guide for adding spikes and surrogates and for documenting date and time of extraction, sample weights, reagents and lot numbers etc. during the extraction process.

Once the draft extraction sheet is complete, it is reviewed and a final hard copy printed to be given to the instrument analyst for reference.

13.A.8 Sample extracts prepared using this SOP are analyzed for PFAS compounds by LCMS-MS (Saphira instrument) by EPA 537.1 or EPA 533, and in accordance with DoD QSM Table B-15.

13.A.9 The acceptance criteria for spikes and surrogates are listed in HPL537. The instrument analyst will notify the extraction technician if re-extraction is required for a particular set of samples, based on the information listed in HPL537 regarding quality control failures pertaining to the extraction process.

13.B EPA 537.1 Drinking Water Sample Extraction

Table 2 EPA 537.1 QC Preparation Summary

	Each extraction batch contains	537 100 ng/mL Surrogate Spike	PFAS 537 50 ng/mLSpike
Samples	≤20	0.1mL	
BLK	1	0.1mL	
LCS	1	0.1mL	0.01mL or 0.1mL or 0.2mL
LCSD	If insufficient volume for MS/DUP	0.1mL	0.01mL or 0.1mL or 0.2mL
MS	1	0.1mL	0.01mL or 0.1mL or 0.2mL
DUP	If samples expected to be greater than MRL	0.1mL	
MSD	If samples expected to be less than MRL	0.1mL	0.01mL or 0.1mL or 0.2mL

13.B.1 Before extraction perform these tasks:

Record Trizma lot numbers in the pH field of the Extraction DB, Note: that FRB are required to have the same Trizma lot numbers as their samples.

Record Initial weights to 1g precision in the sample comments section

Spike all samples according to Table 1

Cap and invert all QC and samples. Allow settling before extraction if necessary.

13.B.2 Connect the vacuum manifold first to waste carboy and then to the vacuum port in series. Set up the appropriate number of Strata –XL 100 µm Polymeric Reversed Phase SPE Cartridges (or equivalent) onto the manifold.

13.B.3 Clean and condition the cartridges by rinsing first with 3x5mL of methanol then rinse with 4x5 mL of reagent water without allowing the solutions to drop below the top edge of the packing. Ensure the SPE cartridge is full of water before proceeding to the loading step.

13.B.4 Attach the sample transfer tube on top of the SPE Cartridge and set up each sample.

13.B.5 Adjust the vacuum flow rate to 10-15 mL/min with unused ports on the top of the manifold and the needle valve on the port to the vacuum trap carboy. Do not allow the cartridge to go dry before the entire sample has passed through.

13.B.6 After the entire sample has passed through, rinse the polypropylene sample bottle with 4x5 mL aliquots of reagent water pull the rinse through the cartridge. Draw air through the cartridge for 5 min at high vacuum (10-15 in Hg).

13.B.7 Turn off the vacuum and release the pressure. Lift the top off the manifold and insert a rack with collection tubes into the tank of the manifold. Place the top back onto the manifold.

Note: Make sure the tips on the top of the manifold slide into the collection tube such that the sample will elute into the proper tube.

13.B.8 Rinse the polypropylene sample bottle with 5 mL of methanol and elute the analytes from the cartridge by pulling the methanol through the transfer tube and cartridge in a dropwise rate with a low vacuum. Repeat sample bottle rinse and cartridge elution with a second 5mL aliquot of methanol.

Alternatively, remove sample transfer tubes and rinse them into the sample bottle. The use this to rinse the sample bottle then finally elute with this rinse. This is mechanically easier to rinse the sample transfer tube, and in either way equivalent.

13.B.9 Pull all of the methanol through the cartridge into the collection tube.

While the samples are still under vacuum, it is ill advised to remove the SPE cartridges while the stopcock is open. Doing so will pull sufficient air to force the sample extract from the centrifuge tube.

13.B.10 Shut off the vacuum and cap the collection tubes.

13.B.11 The extracts are concentrated to with a gentle stream of nitrogen and a dry bath at 65°C.

13.B.12 The extracts are brought up to 2mL at 96%methanol, 4%water, and IIS solution. The entire extract in transferred to a polypropylene injection vial fitted with silicone septa.

13.B.13 The extracts are now ready for analysis according to SOP HPL537.

13.C EPA 533 Drinking Water Sample Extraction

Table 3 EPA 533 QC Preparation Summary

	Each extraction batch contains	EIS Spike mix A	PFAS 200ng/mLSpike
Samples	≤20	0.02mL	
BLK	1	0.02mL	
LCS	1	0.02mL	0.01mL or 0.1mL or 0.2mL
LCSD	If insufficient volume for MS/DUP	0.02mL	0.01mL or 0.1mL or 0.2mL
MS	1	0.02mL	0.01mL or 0.1mL or 0.2mL
DUP	If samples expected to be greater than MRL	0.02mL	
MSD	If samples expected to be less than MRL	0.02mL	0.01mL or 0.1mL or 0.2mL

11.A.1 Before extraction perform these tasks:

Record Initial weights to 1g precision in the sample comments section

Spike all samples according to Table 3

All samples must contain 1g/L ammonium acetate and have a pH between 6 and 8.

To QC, ensure 0.5mL of AmAc-H_2O is added be for continuing.

Measure pH simply by diping a pH strip into the sample.

Adjust pH with GAA or AmOH as necessary.

Cap and invert all QC and samples. Allow settling before extraction if necessary.

11.A.2 Connect the vacuum manifold first to waste carboy and then to the vacuum in series. Set up the appropriate number of Weak Anion SPE Cartridges onto the manifold.

11.A.3 Clean and condition the cartridge by rinsing first with 10 mL MeOH, then 10 mL of PO_4 buffer. Close the valve and add 2–3 mL of phosphate buffer to the cartridge reservoir and fill the remaining volume with H_2O.

Do not allow the cartridge to go dry at any step before the sample has fully passed through the SPE cartridge. If sample loading has not started, and the cartridge is allowed to go dry, return to the beginning of this step.

11.A.4 After the addition of H_2O - Attach the sample transfer tube on top of the SPE Cartridge. Allow the solvent to reach the midpoint of the SPE tube and add the sample into the sample transfer tube.

11.A.5 Apply very weak vacuum, less than 5”Hg, such that the flow rate, as measured from the sample transfer tube to the SPE cartridge, of each sample is less than two drops per second, 6mL/min.

As samples proceed the vacuum should be increased to maintain this flow rate, at maximum, for the fastest sample. As samples finish they will be allowed to pull air, or stopped with a stopcock, for the duration of the remaining samples allowing higher vacuum for the remaining slower samples.

Do not allow the cartridge to dry at any step before the sample has fully passed through the SPE cartridge. If sample loading has started then add sample to the SPE cartridge up to the mid point of the cartridge, proceed with the rest of the sample, and note in the comment section of the extraction sheet with “Dried SPE”.

It may be necessary to re-extract if this occurs. Until that is determined samples will be analyzed to determine if re-extraction is necessary.

11.A.6 After the entire sample has passed through the cartridge, rinse the sample bottle with a 10 mL aliquot of AmAc-H_2O-d. Transfer this rinse through the sample tranfer tube and cartridge. Rinse the sample transfer tube and cartridge with 1mL MeOH.

11.A.7 Apply a high vacuum for about 5 minutes, until the cartridge is visibly dry, to remove residual solution from the cartridge.

Alternatively, employ a centrifuge to remove residual solution. Use sample stickers to mark each SPE cartridge and centrifuge at 3500 for 10 minutes or 4500 for 5 minutes using the 15mL centrifuge tube rotor.

11.A.8 Turn off the vacuum and release the pressure. Lift the top off the manifold and insert a rack with collection tubes into the tank of the manifold. Place the top back onto the manifold.

11.A.9 Rinse the sample transfer tube with 5 mL of AmOH@0.5-MeOH into the sample bottle. Cap and shake the sample bottle.

This step may be performed proactively, as samples finish.

11.A.10 Elute the SPE cartridge using this sample bottle rinse, either careful decanting or disposable polyethylene transfer pipettes may be employed.

11.A.11 Repeat the rinse and cartridge elution using 5 mL of AmOH-MeOH.

The sample transfer tube should be rinsed again here, into the bottle, as in step 13.C.9 including the caping and shaking.

11.A.12 Pull all of the eluents through the cartridge into the collection tube.

While the samples are still under vacuum, it is ill advised to remove the SPE cartridges while the stopcock is open. Doing so will pull sufficient air to force the sample extract from the centrifuge tube.

11.A.13 Shut off the vacuum and cap the collection tubes.

11.A.14 The extracts are concentrated to 2mL final volume with a gentle stream of nitrogen and a dry bath at 65°C.

11.A.15 The extracts are now ready for analysis according to SOP HPL537.

13.D Table B-15 Aqueous Sample Extraction

Table 4 QSM Table B-15 Aqueous Sample- QC Preparation Summary

	Each extraction batch contains	EIS Spike mix(s)	PFAS 200ng/mLSpike
Samples	≤20	0.02mL each	
BLK	1	0.02mL each	
LCS	1	0.02mL each	0.1mL
LCSD	If unable to perform MS/MSD	0.02mL each	0.1mL
MS	1	0.02mL each	0.1mL
DUP	None	0.02mL each	
MSD	1	0.02mL each	0.1mL

13.D.1 Before extraction perform these tasks:

Record Initial weights to 0.1g precision on the extraction sheet

Add 0.1mL AmAc to each sample and QC; check the pH is 6-7. If necessary adjust with GAA or AmOH.

Spike all samples according to Table 1

Alternative spiking may be employed so long as its documented on the extraction sheet.

Cap and invert all QC and samples.

For samples with colloidal particles and emulsions, treat the sample as normal by filtering the entire sample container through the SPE cartridge.

For all other sample problems (multiphasic for example), the client will be contacted for further instruction.

13.D.2 Connect the vacuum manifold first to waste carboy and then to the vacuum in series. Set up the appropriate number of Weak Anion SPE Cartridges onto the manifold.

13.D.3 Clean and condition the cartridge by rinsing first with 10mL of AmOH-MeOH, then 10mL of MeOH, and then 10mL of PO4 buffer.

This is done most easily by adding 3mL AmOH-MeOH to the SPE cartridge, connceting the sample reservoir, then adding the remaining 7mL of AmOH-MeOH. When one solution finishes leaving the sample reservoir, add the next.

Do not allow the cartridge to go dry at any step before the sample has fully passed through the SPE cartridge. If sample loading has not started, and the cartridge is allowed to go dry, return to the beginning of this step.

13.D.4 Before loading the sample – Ensure the solvent is at the midpoint of the SPE tube and add the sample into the sample transfer tube. Employ more PO4 buffer as necessary.

13.D.5 Apply very weak vacuum, less than 5”Hg, such that the flow rate, as measured from the sample transfer tube to the SPE cartridge, of each sample is less than two drops per second, 6mL/min.

As samples proceed the vacuum should be increased to maintain this flow rate, at maximum, for the fastest sample. As samples finish they will be allowed to pull air, or stopped with a stopcock, for the duration of the remaining samples allowing higher vacuum for the remaining slower samples.

Do not allow the cartridge to dry at any step before the sample has fully passed through the SPE cartridge. If sample loading has started then add sample to the SPE cartridge up to the mid point of the cartridge, proceed with the rest of the sample, and note in the comment section of the extraction sheet with “Dried SPE”.

It may be necessary to re-extract if this occurs. Until that is determined samples will be analyzed to determine if re-extraction is necessary.

13.D.6 After the entire sample has passed through the cartridge, add 10mL of deionized water to the sample reservoir, when that has finished passing through the cartridge, add to the reservoir 5mL FA-MeOH:H2O.

13.D.7 Apply a high vacuum for about 2 minutes, until the cartridge is visibly dry, to remove residual solution from the cartridge.

13.D.8 Turn off the vacuum and release the pressure. Lift the top off the manifold and insert a rack with collection tubes into the tank of the manifold. Place the top back onto the manifold.

13.D.9 Add 5mL of AmOH-MeOH to the origional sample bottle, and use a glass disposable pipette to rinse the sides of the bottle with the AmOH-MeOH.

This step may be performed proactively, as samples finish.

13.D.10 Using the same pipette, elute the SPE cartridge using this sample bottle rinse making sure to rinse the sides of the sample reservoir as much as possible.

13.D.11 Pull all of the eluent through the cartridge into the collection tube.

While the samples are still under vacuum, it is ill advised to remove the SPE cartridges while the stopcock is open. Doing so will pull sufficient air to force the sample extract from the centrifuge tube.

13.D.12 Shut off the vacuum and cap the collection tubes.

13.D.13 The extracts are concentrated to <2mL with a gentle stream of nitrogen and a dry bath at 65°C.

13.D.14 2ng of IIS are added and the extract is brought to 2mL final volume

13.D.15 10mg ENVICARB is added, the samples are immediately and briefly shaken, centrifuged, and syringe filtered into final injection vials.

See 6.F in Interferences and Potential Problems

13.D.16 The extracts are now ready for analysis according to SOP HPL537.

13.E Table B-15 Solid Sample Extraction

Table 5 QSM Table B-15 Solid Sample- QC Preparation Summary

	Each extraction batch contains	EIS Spike mix(s)	PFAS 200ng/mLSpike
Samples	≤20	0.02mL each	
BLK	1	0.02mL each	
LCS	1	0.02mL each	0.1mL
LCSD	If unable to perform MS/MSD	0.02mL each	0.1mL
MS	1	0.02mL each	0.1mL
DUP	None	0.02mL each	
MSD	1	0.02mL each	0.1mL

13.E.1 Weigh approximately 0.5 g of sample into a 15 mL centrifuge tube.
Homogenize the sample thoroughly before weighing, by using a spatula to mix the sample in the jar.

13.E.2 Fortify the sample with 0.02 mL of 100ng/mL EIS mix A and 0.02mL 1000ng/mL EIS mix B Add 3 mL of MeOH.

13.E.3 Vortex the sample thoroughly

13.E.4 Centrifuge

13.E.5 Decant the sample through an envicarb cartridge.
Alternatively, transfer using a disposable polyethylene transfer pipette.

13.E.6 Add 3 mL MeOH

13.E.7 Vortex the sample thoroughly
The sample may be difficult to break up after centrifugation. It is strictly necessary to break up the cake that forms at the bottom of the centrifuge tube. This may happen through vortexing alone but if not the sample should be shaken until it does break up. A disposable polyethylene transfer pipette may be employed to break up the cake.

13.E.8 Centrifuge

13.E.9 Decant the sample through the envicarb cartridge in step 14.F.6, collecting the extracts together.

13.E.10 Add 3 mL MeOH

13.E.11 Vortex the sample thoroughly

13.E.12 Centrifuge

13.E.13 Decant the sample through the envicarb cartridge in step 14.F.6, collecting the extracts together.

13.E.14 The extracts are concentrated to 2mL with a gentle stream of nitrogen and a dry bath at 65°C.

13.E.15 The extracts are brought up to 2mL final volume at 80%methanol, 20%water.

13.E.16 The extracts are now ready for analysis according to SOP HPL537.
See 6.F in Interferences and Potential Problems

13.F Table B-15 Solid Biota Sample Extraction

Table 6 QSM Table B-15 Biota Sample - QC Preparation Summary

	Each extraction batch contains	EIS Spike mix(s)	PFAS 200ng/mLSpike
Samples	≤20	0.05mL each	
BLK	1	0.05mL each	
LCS	1	0.05mL each	0.1mL
LCSD	If unable to perform MS/MSD	0.05mL each	0.1mL
MS	1	0.05mL each	0.1mL
DUP	None	0.05mL each	
MSD	1	0.05mL each	0.1mL

13.F.1 Weigh approximately 0.5 g of sample into a 15 mL centrifuge tube.
Homogenize the sample thoroughly before weighing, by using a spatula to mix the sample in the jar.

13.F.2 Fortify the sample with 0.05 mL of 100ng/mL EIS mix A and 0.05mL 1000ng/mL EIS mix B, add 3 mL of MeOH.

13.F.3 Vortex the sample thoroughly

13.F.4 Centrifuge

13.F.5 Decant the sample through an envicarb cartridge.
Alternatively, transfer using a disposable polyethylene transfer pipette.

13.F.6 Add 3 mL AmOH-MeOH

13.F.7 Vortex the sample thoroughly
The sample may be difficult to break up after centrifugation. It is strictly necessary to break up the cake that forms at the bottom of the centrifuge tube. This may happen through vortexing alone but if not the sample should be shaken until it does break up. A disposable polyethylene transfer pipette may be employed to break up the cake.

13.F.8 Centrifuge

13.F.9 Decant the sample through the envicarb cartridge in step 14.F.6, collecting the extracts together.

13.F.10 Add 3 mL AmOH-MeOH

13.F.11 Vortex the sample thoroughly

13.F.12 Centrifuge

13.F.13 Decant the sample through the envicarb cartridge in step 14.F.6, collecting the extracts together.

13.F.14 The extracts are concentrated to 5mL with a gentle stream of nitrogen and a dry batg at 40°C.
See 6.F in Interferences and Potential Problems

13.F.15 The extracts are brought up to a 2mL final volume at 80%methanol, 20%water, and IIS solution.

13.F.16 The extracts are now ready for analysis according to SOP HPL537.

13.G AFFF formulation preparation for SPE extraction.

13.G.1 A 50mL centrifuge tube sample container is placed on a balance, torn, and 0.02mL of sample are pipetted into the container, the sample is diluted to 45mL with H_2O. The sample is allowed to sit for 3 hours, or overnight. The sample is now ready for processing by 13.D Aqueous sample extraction.

11.B AFFF formulation by serial dilution.

Sets of samples are prepared with QC; BLK, LCS, LCSD at clients request, and a PS (post spike). All samples must be performed in duplicate.

13.H.1 Take a tray of 1mL-polypropylene autosampler vials and label them accordingly.

13.H.2 To each, measure gravimetrically 0.2mL of sample, H_2O for QC samples.

13.H.3 Spike with 0.01mL of each EIS mix A and EIS mix B.

13.H.4 To the LCS, LCSD and PS, pipette 0.05mL 200ng/mL PFAS mix.

13.H.5 Then pipette 0.780mL (0.730 for LCS, LCSD and PS) of MeOH to achieve a final volume of 1mL.

13.G.2 The samples are now ready for analysis according to SOP HPL537. Further dilutions will be performed as in SOP HPL537.

13.H PFAS Solid Sample TCLP Extraction

For solid samples requiring TCLP analysis, follow EPA 1311 Semi-volatile guidelines and APPL SOP PRE1311 for the tumbling portion. A separate container of TCLP fluid will be tumbled along side the samples to be used for the method blank and LCS. Follow the non-potable water cartridge extraction procedure above to extract the TCLP fluid. TCLP extracts are then subsampled into 50mL polypropylene container and then extracted.

13.I EPA 1633

14 Data Analysis and Calculations - NA

15 Data Assessment and Acceptance Criteria for QC - NA

16 Corrective Actions and Contingencies for Out of Control Data or Unacceptable Data

In the event that an out of control situation occurs, the project manager will be notified immediately. The affect of the out of control situation will be assessed according to the project DQO. If sufficient sample remains, and the situation will significantly affect the quality of the results, the analysis will be repeated. If the situation does not significantly affect the quality of the data, the project manager will notify the client and instructions from the client will be followed. In the event no sample remains, the client will be notified immediately. All situations will be documented on the multi level sheet and initialed by the project manager. All out of control situations will be brought to the attention of the QAU in the form of a NWR. The QAU has the final authority to approve the actions taken.

17 Method deviations

17.A For drinking water analysis, the following method deviations are noted as compared to EPA method 537.1 and 533:

17.B Method Requirement: EPA 537.1 and 533. Samples are concentrated to dryness.

17.B.1 APPL Practice: Samples volume is reduced sufficiently without going to complete dryness.

17.B.2 This potentially leaves samples with higher than intended water concentration.

17.B.3 120mg 30um Oasis weak anion exchange are used in place of Phenomenex brand cartridges.

17.C For non-potable water and solid sample analysis by EPA 537-mod, there are no method deviations, since this is a proprietary procedure developed by the laboratory. EPA 537.1, 533, ISO 25101:2009E and ISO 21675:2019 were used for guidance purposes.

17.C.1 Alternate spike concentrations are used to reflect instrument sensitivity and calibration range.

17.C.2 Samples are extracted within 28 days of collection and analyzed within 28 days of extraction rather than the whole procedure being performed in 14 days. Deviation and Justification: The holding time established by APPL is consistent with EPA 533.

17.C.3 Supelco® Supelclean™ ENVI-Carb™ Cartridges are used in addition to the SPE cartridges in order to meet DOD requirements.

18 Pollution Prevention -All hazardous materials that are generated during the testing of samples must be properly collected and stored. Drums are available in the storage room for the following types of wastes- acidic, basic and solvents

19 Waste Management

19.A Spent sample extracts and standards / surrogates / spikes are disposed in the LC Waste Drum (Profile ID 312936 Acetonitrile/water/methanol).

19.B Spent autosampler vials are disposed of as non-hazardous waste.

19.C It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and lands disposal restrictions. The laboratory has the responsibility to protect the environment by minimizing and controlling all releases from fume hoods and bench operations.

20 Method Performance - MDL studies on file electronically on HPLC department server. LOD/LOQ checks on file electronically with QAU.

21 Equipment/Instrument Maintenance and Troubleshooting - NA

22 Computer hardware and software - NA

23 References

23.A ISO/IEC 17025:2017

23.B ISO 21675:2019

23.C DoD/DOE QSM, Version 5.1, December 2017

23.D DoD/DOE QSM, Version 5.2, December 2018

23.E DoD/DOE QSM, Version 5.3, May 2019

23.F Method 537 Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry, Version 1.1, 2009.

23.G Method 537.1 Determination of Selected Per- and Polyflourinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry. Version 2.0 March 2020.

23.H 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

23.I EPA 1633 Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS

23.J Water quality – Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry, ISO25101, 1st Edition, 2009.

23.K EPA 1311 Toxicity Characteristic Leaching Procedure, Revision 0 July 1992.

23.L Any tables, diagrams, flowcharts, validation data EPA 537.1 Drinking Water Field Instructions for Preparing FRB.

24 Tables and Diagrams

24.A Table 1: Definitions Crosswalk
24.B Table 2: EPA 537.1 QC Preparation Summary
24.C Table 3: Table 3 EPA 533 QC Preparation Summary
24.D Table 4: QSM Table B-15 Aqueous Sample- QC Preparation Summary
24.E Table 5: Solid Sample- QC Preparation Summary
24.F Table 6: QSM Table B-15 Biota (Tissue) Sample- QC Preparation Summary
24.G Table 7: Extraction Methods Summary
24.H Appendix A: EPA 537 PFAS Drinking Water Field Instructions
24.I Appendix B: EPA 533 PFAS Drinking Water Field Instructions
24.J Appendix C: Filtration of Samples
24.K Appendix D: California Wastewater PFAS Sampling Guide

Section Manager Name: Hart Hedgpeth

Section Manager Signature: Hart Hedgpeth Date: 02/10/22

QAU Director Name: Paula McCartney

Paula McCartney

QAU Director Signature: Date: 02/10/22

Table 6:
SPE Methods Summary

	EPA 537.1	EPA 533	Table B-15 Table B-20? EPA 1633
Sample Prep	Preserved with Tris Buffer Spiked with isotopologues (surrogates)	Preserved with Ammonium acetate buffer Spiked with isotopologues	Spiked with Isotopologues Neutralized with AmAc, GAA, AmOH pH 6-7 Appendix C, when necessary
SPE Cartridge	500mg 100um PolymericReversePhase	200mg 33um Strata-X-AW	200mg 33um Strata-X-AW
Wash	3 x 5mL MeOH	2 x 5mL MeOH	10mL 0.1% AmOH-MeOH 10mL MeOH
Condition	4 x 5mL H_2O	3 x 5mL PO_4-buffer	10mL PO_4-buffer
Load	10-15 mL/min 3-6 drops/second	3mL/min 1-2 drops/second	3mL/min 1-2 drops/second
Wash	4 x 5mL H_2O	10mL AmAc-H_2O-d	10mL H2O 5mL FA MeOH:H2O
Dry	High vacuum 5 minutes	1mL MeOH, High Vacuum ~ 5 minutes	High Vacuum 2min
Elute	Dropwise 2 x 5mL MeOH	Dropwise 5mL AmOH-0.5-MeOH 5mL AmOH-0.5-MeOH	Dropwise 5mL AmOH-MeOH
Concentration	65°C dry bath w/ N2(g) To/near dryness.	65°C dy bath w/ N2(g) To/near dryness.	65°C dry bath w/ N2(g) Stop < 2mL
Final Volume MeOH:H2O IIS	2mL 96:4 0.050mL 537 IIS	2mL 80:20 0.05mL 533 IIS	2mL 80:20 0.05mL B-15 IIS

Appendix A

EPA 537 PFAS Drinking Water Field Instructions*

SAMPLE COLLECTION

1. Wash hands before sampling, and wear nitrile gloves during sampling.
2. Turn on tap water and flush the system for 3-5 min.
3. Fill sample bottles with gentle stream of water, taking care not to overfill causing spillage.
4. Cap the bottle and agitate by hand until preservative is dissolved.

FIELD REAGENT BLANKS (FRB)

1. A FRB must be prepared at each sampling site and at the same time as sample collection.
2. Unpack the contents of the ziploc bag containing the FRB Kit:
    - Lab Water (Trizma® preserved 250mL poly containing lab reagent water)
    - FRB (Un-preserved 250mL poly bottle- Empty)
3. Verify the Trizma® lot # for FRB is the same as Trizma® lot # for the sample containers. (Contact the lab if the lot numbers do not match before collecting samples)
4. Pour the lab water into the empty sample bottle, seal and label this bottle as the FRB.
5. Note the date and time on the FRB label, using an indelible pen.
6. Ship the FRB overnight to the lab, along with the samples packed in a cooler with ice.

*EPA 537 Rev 1.1 PFAS in Drinking Water, Version1.1, September 2009, sect 8.2 to 8.3.
EPA 537.1 PFAS in Drinking Water, Version 1.0, November 2018, sect 8.2 to 8.3.

Appendix B

EPA 533 PFAS Drinking Water Field Instructions*

SAMPLE COLLECTION

1. Wash hands before sampling, and wear nitrile gloves during sampling.
2. Turn on tap water and flush the system for 3-5 min.
3. Fill sample bottles with gentle stream of water, taking care not to overfill causing spillage.
4. Cap the bottle and agitate by hand until preservative is dissolved.

FIELD REAGENT BLANKS (FRB)

1. A FRB must be prepared at each sampling site and at the same time as sample collection.
7. Unpack the contents of the ziploc bag containing the FRB Kit:
   2. Reagent Water (250mL HDPE bottle containing 250mL reagent water)
   3. FRB (250mL HDPE bottle containing 0.5mL 0.5g/mL ammonium acetate)
4. Verify the ammonium acetate prep date for FRB is the same as ammonium acetate prep date for the sample containers. (Contact the lab if the lot numbers do not match before collecting samples)
5. Pour the reagent water into the preserved sample bottle, seal and label this bottle as the FRB.
6. Note the date and time on the FRB label, using an indelible pen.
7. Ship the FRB overnight to the lab, along with the samples packed in a cooler with ice.

*EPA 533 PFAS in Drinking Water, sect 8.4.2

Appendix C

Filtration of samples

Samples may be filtered, after acquiring client permission, and after spiking, by the following procedure.

1. using glass fiber or nylon filter material filter the sample from one sample container into a sufficiently large HDPE container (the sample amount, plus at most 10% more volume).
2. Rinse the sample bottle and filter with water until all solids are on the filter.
3. Move the filter into a 50mL centrifuge tube.
4. Rinse the original bottle and the filtration device with 5mL methanol, into the centrifuge containing the filter.
5. Vortex the filter.
6. Centrifuge the filter (if necessary)
7. Decant the centrifuge tube with methanol into the sample.
8. Repeat steps 4-7 twice more.
9. Discard the filter and solids.
10. The sample is now ready for extraction by SPE.
    A. The sample should not be less than 90% water before proceeding. If necessary the sample may be diluted in its current HDPE container, or on line.

Appendix D

California Wastewater PFAS Sampling guide

Influent and effluent sample

Due to high potential for suspended Solids we request you let your 24 hour composite sample settle first before sampling. Once the sample contents have settled please collect the following:

Per Influent or Effluent sample:

1. Fill and leave some headspace 2x 50 ml polypropylene centrifuge tubes
2. Total of 2 bottles per sample

Monitoring wells

1. Fill 2x 50 ml polypropylene centrifuge tubes

Biosolid

1. Fill 1x 50 ml polypropylene centrifuge tube

Field Blank (FB) and Equipment Blank (EB)

For each FB and EB, APPL will provide 1x 50ml polypropylene centrifuge tube with PFAS free water along with one empty 50ml polypropylene centrifuge tube.

1. For FB, Take lid off of full bottle during sampling. When finished pour PFAS free water into empty bottle and submit as your field blank.
2. For EB, Take lid off of full bottle and use it to rinse equipment. Catch the rinse water in the empty bottle and submit as your equipment blank.

Tips -

1. Fill COC out with ballpoint pen.
2. If you use a Sharpie pen try to avoid using it while containers are open.
3. Please identify the test on the Chain of Custody as PFAS CA 32
4. In the COC Comments, please indicate if you require reporting below the RL (with J-flags to the MDL) or at the RL (with no J-flags to the MDL).
5. Please put COC in ziplock bag in the cooler.
6. Please return samples with frozen-water ice. Please double bag ice in ziplock bags.
7. You will need to obtain the Global ID, Log ID, and Field Point Names for your GeoTracker EDF. For help with obtaining this information, please contact the GeoTracker Help Desk at (886)-480-1028 or Geotracker@waterboards.ca.gov

If you have questions please contact your Project Manager

ATTACHMENT B

PROJECT STANDARD OPERATING PROCEDURES

SOP No. 1 Water Level Measurement (PFAS Specific)
SOP No. 2 Groundwater Sampling (PFAS Specific)
SOP No. 3 Sample Handling and Management (PFAS Specific)
SOP No. 4 Sampling Equipment Decontamination (PFAS Specific)
SOP No. 5 Soil Sampling for Chemical Analysis (PFAS Specific)
SOP No. 6 Investigation Derived Waste (IDW) Management (PFAS Specific)

Field Instrument Manuals

- Horiba U-50 Multi-Parameter Water Quality Checker
- Eurotech TN-100/T-100 Portable Turbidity Meter
- Solinst Water Level Meter
- Honeywell PID
- Redi-Flo 2 Submersible Pump with Geotech VFD
- Extech 407750 Digital Sound Level Meter
- Trimble GeoExplorer 6000 Series

Standard Operating Procedure No. 1

Water Level Measurement

(PFAS Specific)

Revision:_____0.0_____

Reviewed:___11/01/21___

Reviewer:_____

David Conner, PG

SOP No. 1 – Water Level Measurement (PFAS Specific)

1.0 OBJECTIVE

The purpose of this document is to define the standard operating procedure (SOP) for measuring water elevations in monitoring wells included in environmental monitoring programs. This procedure describes equipment and field procedures necessary to collect water elevation measurements. The well locations and frequency of measurement are specified in project-specific work plans and Quality Assurance Project Plans (QAPPs).

2.0 EQUIPMENT AND MATERIALS

All equipment and materials must be Per- and Polyfluoroalkyl Substances- (PFAS) free and have not come into contact with a material containing PFAS. The equipment and materials necessary that may be used to measure water levels include:

- Electronic water level indicator capable of producing measurements to a precision of 0.01 feet
- 5-gallon buckets or equivalent for decontamination
- Polyethylene or polyvinyl chloride (PVC) brushes for decontamination
- Water Level Measurement Form or Groundwater Sampling Form (non-waterproof, non-recycled loose paper)
- Non-waterproof ball point pens
- Metal clipboard (plastic clipboards are not allowed)
- Non-waterproof field notebook (Rite in the Rain® notebooks are acceptable)
- Chemical-free paper towels or Kimwipes™
- Alconox®, Liquinox®, or Citranox® soap or detergent
- Laboratory supplied PFAS-free deionized water (preferred)
- Garden-type spray bottle made of PFAS-free High-Density Polyethylene (HDPE), glass, or polypropylene with Teflon®-free caps filled deionized or distilled water
- Appropriate health and safety equipment

3.0 WATER ELEVATION MEASUREMENT PROCEDURE

3.1 Discussion

Generally, water elevation measurements are used to construct potentiometric surface maps. Therefore, water level measurements at a given site should be collected within a 24-hour period. The device used to measure water levels should be adequate to attain an accuracy of 0.01 feet. Water levels should be allowed to stabilize for a minimum of 24 hours after well construction and development before measurements are taken.

3.2 Measurement Procedure

This section gives the steps to follow when measuring water levels. Note that appropriate health and safety steps should be implemented, and health and safety equipment should be worn during well opening, well measurement, and decontamination.

- Before any measurement is taken, the water level indicator shall be decontaminated. Decontamination procedures are discussed in SOP – Sampling Equipment Decontamination (PFAS Specific).
- Confirm that the monitoring well is labeled, and the location ID is visible on the protective casing and that the ID coincides with the expected location.
- After opening the well cover, measure the depth of the static water level and the total depth of the well using an electronic water level indicator. The measuring point for all the wells shall be the top of PVC or steel well casing. The measuring point will be marked by a notch or other mark in the PVC or steel casing. If no mark is present, measure from the top of the north side of the casing.
- The static water level and the depth of the well shall be measured with the indicator, logged on the field data sheet or field notebook as feet (ft) below top of casing (btoc), and verified before the indicator is removed from the well. Note any significant changes in water level, by comparing the most recent measurement with past measurements, if appropriate.
- The water level depth below the measuring point (i.e., ft btoc) will be subtracted from the measuring point elevation (i.e., ft above mean sea level [amsl]) to determine the elevation of the static water level. If measuring point elevations are available at the time of water level measurement, the calculated water elevation (ft amsl) should be checked in the field to see that it is reasonable, and the subtraction was performed correctly. If there is a significant discrepancy in the measured water level or calculated water elevation, the well should be measured again.
- All columns of field data sheets shall be completed, including time of measurement. If items on the sheet do not apply to a specific location, the item will be labeled as not applicable (NA).

3.3 Decontamination

The water level indicator must be decontaminated before use, between wells, and at the conclusion of measurements. The probe will be decontaminated according to the procedure for decontamination of sampling equipment described in SOP – Sampling Equipment Decontamination (PFAS Specific).

4.0 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on the activities concluded and provide a permanent record of field activities. The observations and data will be recorded with non-waterproof ink, such as a ballpoint pen (Sharpies® and permanent markers are not allowed), in a permanently bound non-waterproof field logbook with consecutively numbered pages, and on field data sheets on non-waterproof and non-recycled loose paper.

4.1 Field Data Sheet for Water Level Measurements

A field sampling data sheet for groundwater samples will be completed at each sampling location. If items on the sheet do not apply to a specific location, the item will be labeled as not applicable (NA). The information on the data sheet includes the following:

- Well number
- Field book reference number
- Field personnel
- Well I.D.
- Date and time of measurements
- Sample identification number
- Water level (ft btoc)
- Static Water Elevation Data (ft amsl)

The measurement point elevation (ft amsl) should be filled in if it has been determined at the time of measurement. If it is not known the form will be completed at a later time when further information is available. Any irregularities or problems that may have a bearing on sampling quality should be noted in the field.

Standard Operating Procedure No. 2

Groundwater Sampling

(PFAS Specific)

Revision:_____0.0_____

Reviewed:___11/03/21___

Reviewer:_____

David Conner, PG

SOP No. 2 – Groundwater Sampling (PFAS Specific)

MONITORING WELL SAMPLING

1.0 OBJECTIVE

The purpose of this document is to define the standard operating procedure (SOP) for collecting groundwater samples during environmental monitoring programs. This procedure describes equipment and field procedures necessary to collect groundwater samples using several methods. Procedures for groundwater sampling from a monitoring well are designed to obtain a sample for chemical analysis that is representative of natural aquifer conditions. Samples will be collected using disposable bailers or low-flow electric or pneumatic pumps. Sampling of Westbay® multiport (or multilevel) groundwater monitoring wells are presented in a separate SOP.

2.0 METHODOLOGY

2.1 Low-Flow Sampling Using a Submersible Pump

Low-flow purging is the preferred method because it results in (1) minimal loss of Volatile Organic Compounds (VOCs), (2) minimal mixing of chemically distinct zones, (3) minimal production of artificial turbidity and oxidation, and (4) minimal production of potentially contaminated purge water. The micropurging procedures described in this section are adapted from those procedures recommended by the U.S. Environmental Protection Agency (EPA) in Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures (EPA 1996).

Low-flow purging will be conducted using an electric submersible pump, or bladder pump, equipped with a positive-foot check valve to prevent purge water from flowing back into the well. High-density polyethylene (HDPE) tubing will be used for each monitor well. The water quality parameters listed in Table 1 will be regularly measured during purging. All water quality monitoring instruments will be calibrated, and measurements collected in accordance with the procedures specified in SOP – Instrument Calibration. All equipment and materials must be Per- and Polyfluoroalkyl Substances (PFAS)-free and have not come into contact with a material containing PFAS.

The following procedure will be used when purging wells using the low-flow method.

2.1.1 Decontaminate any equipment (i.e., water level indicators, pumps, and down-well probes) before placing in the well in accordance with the procedures specified in SOP – Sampling Equipment Decontamination (PFAS Specific).

2.1.2 Measure the water level in the well in accordance with the procedure described in SOP – Water Level Measurement (PFAS Specific), before beginning purging. Record the water level on the Monitor Well Sampling Form on loose non-waterproof and non-recycled paper. Do not measure the total well depth before low-flow sampling so that any sediment in the bottom of the well, if present, will not be disturbed and entrained in the water column.

2.1.3 Lower the pump into the well in a controlled manner until the pump intake is situated at the approximate midpoint of the saturated screened interval, as determined from well completion logs, or midpoint of saturated screen interval (information concerning total depth of well and screened interval can be obtained from the Monitoring Well Construction Form for each well). Do not allow the pump to touch or settle on the well bottom to prevent excess agitation and entrainment of sediment that may have accumulated in the bottom of the well.

2.1.4 Start the pump and determine the pumping rate by measuring the time required to fill a calibrated container. Adjust the pumping rate so that is does not exceed 0.5 liter (0.13 gallon) per minute in order to minimize the potential for stagnant water to be drawn into the pump intake.

2.1.5 Verify that the pumping rate is low enough by measuring the water level in the well every 3 to 5 minutes during purging. Adjust the pumping rate to maintain a drawdown of the water from the pre-sampling level at less than 0.1 meter (0.3 foot), if possible. When parameters are collected within in-line flow-through cells, the frequency of measurements shall be equal to the time required to provide complete exchange of the cell volume. If the drawdown cannot be maintained at less than 0.1-meter (0.3 foot) by reducing the pumping rate, note the condition on the Monitoring Well Sampling Form and continue to purge the well until (1) parameters and drawdown stabilize, (2) parameters stabilize and three casing volumes are removed, or (3) the well is purged dry.

2.1.6 Measure water quality parameters (as indicated in Table 1) at the start of purging and then every 3 to 5 minutes thereafter to determine when the well has been adequately purged, as discussed below.

2.1.7 Purge the well until either (1) the water quality parameters stabilize over three consecutive readings, (2) three casing volumes of water are removed, (3) the well is purged dry, (4) or the well has been pumped for at least 2 hours without drawdown. The stabilization criteria and proposed measurement methods for each of the parameters is presented in Table 1. If the well is purged dry, samples from the well can be collected when a sufficient volume of water has recharged to begin filling sample containers.

2.1.8 Collect samples as described in Sample Collection section below.

2.1.9 Sample containers in PFAS-free HDPE, glass, or polypropylene bottles will be labeled, and packed on ice or with reusable chemical ice packs if double bagged in LDPE resealable storage bags for shipment to the analytical laboratory under appropriate chain of custody protocol and SOP – Sample Handling and Management (PFAS Specific).

2.1.10 Contain and handle all purge water removed from well in accordance with the IDW management procedures specified in SOP – IDW Management (PFAS Specific).

2.1.11 Decontaminate all sampling and drilling equipment per SOP – Sampling Equipment Decontamination (PFAS Specific).

Table 1
WATER QUALITY PARAMETER STABILIZATION CRITERIA
AND PROPOSED MEASUREMENT METHODS

Parameter	Stabilization Criteria	Measurement Method[1]
Dissolved Oxygen	± 10 percent	Water quality meter
pH	± 0.2 units	Water quality meter
Specific Conductance	± 10 percent	Water quality meter
Temperature	± 10 percent	Water quality meter
Oxidation-reduction potential (ORP)	± 10 percent (or ± 10 units)	Water quality meter
Turbidity	50 NTU (or ± 10 percent)[2]	Water quality meter

Notes:

1 When possible, dissolved oxygen and ORP should be measured with a flow-through cell or down-well probe to avoid atmospheric influences. Other parameters may be measured using a flow-through cell or alternate method (i.e., Horiba U10 or equivalent).

2 For Turbidity, three consecutive readings within 10 percent is acceptable if the 5 NTU turbidity goal cannot be reasonably achieved. For ORP, ± 10 units is acceptable if ± 10% cannot be achieved (i.e., ORP is very close to zero and therefore ± 10% of the current reading is a very small number compared with the total ORP range).

NTU Nephelometric turbidity units

Blaine Tech Services, Inc.
Standard Operating Procedure

WESTBAY PRESSURE PROFILING AND SAMPLING
(5/13/2021)

- **Decon Procedure**
  - A series of three wash buckets and a final rinse will be setup to facilitate the decontamination procedure. The first bucket or wash will consist of a rinse with tap water and a laboratory-grade detergent (Alconox® or equivalent). The second wash will consist of a secondary rinse with tap water and no detergent. The third wash will consist of a rinse with commercially available deionized or distilled water. The fourth stage in the decontamination process will be a rinse with HPLC water using a squeeze bottle. Once this final stage in the process is completed, the sampling equipment will be allowed to air dry briefly prior to reuse. The first 3 stages fluids will be changed at minimum once per day and new HPLC water will be used each final rinse.

  - The sample intake valve and chamber on the Westbay® MP probe will be sprayed with deionized or HPLC water only, so as to not compromise the integrity of the inner-pressure transducer.

- **Pressure Profiling:**
  - The multiport wells have discrete/isolated sampling ports at various depths throughout their lengths. Pressure measurements from the measurement ports will be used to calculate the depth to water in the multiport wells using Westbay® Method A, as recommended by the manufacturer:

$$H = -D + (P2-P_{atm})*2.307 \text{ ft/psi}$$

Where:

H = depth to water

D = depth to measurement port

P_{atm} = atmospheric pressure

P2 = pressure measured in the measurement port

The groundwater elevation at each port will be calculated from the depth to water and the elevation of the measurement datum:

$$GW_{elev} = G_s – H$$

Where:

GW_{elev} = ground water elevation

G_s = ground surface elevation (measurement datum)

H = depth to water calculated by Method A (described above)

- Prior to collecting groundwater samples, barometric pressure, temperature and will be taken at the surface with the MODAX Sampler prior to collecting measurements in the well. Also, depth-to-water inside the Westbay casing will be measured with an electronic sounder.

- Surface checks will be completed to confirm that the probe activation motor and sampler valve motor are operating properly, and that there are no leaks through the sampler valve or the connectors between the sampler and the sample containers. The surface check procedure is repeated each time the sampler is to be lowered into the well.

- Using the casing log as a reference, the sample probe will be lowered to the lowest measurement port coupling to be monitored. Once the probe rests at the measurement port, the pressure and temperature inside the Westbay casing will be measured. After recording these readings, the shoe will be activated and the formation pressure measured by the probe. When readings have stabilized and been recorded, the shoe will be retracted and a second pressure reading taken inside the Westbay casing. This reading should be similar to first reading. The three pressure readings, temperature reading, and time constitute a complete set of readings at the measurement port coupling.

- The sampler will then move the sampler up the Westbay casing to obtain pressure data from other measurement ports.

- One last set of pressure and temperature readings will be taken at the surface after pressure profiling in the well has been completed. These readings should be similar to those readings taken prior to measuring pressures in the well.

- **Sampling Procedure:**
  - Prior to collecting groundwater samples, barometric pressure, temperature and will be taken at the surface with the MODAX Sampler prior to collecting measurements in the well. Also, depth-to-water inside the Westbay casing will be measured with an electronic sounder.

  - Surface checks will be completed to confirm that the probe activation motor and sampler valve motor are operating properly, and that there are no leaks through the sampler valve or the connectors between the sampler and the sample containers. The surface check procedure is repeated each time the sampler is to be lowered into the well. After the completion of the surface checks, a partial vacuum will be applied to the sample containers to assist in sample collection.

  - A sampling tool with up to four 250-mililiter (ml) stainless steel bottles will be used for sampling the Westbay® MP groundwater monitoring wells.

- Using the multiport casing log as a reference, the sample probe will be lowered to the lowest desired sampling port. The probe will be activated and the fluid pressure outside the measurement port will be recorded. When the sampler probe is properly activated and sealed in the port, the sampler valve will be opened, allowing fluid to flow from the monitoring zone into the sample containers. When the containers have filled and the measurement port closed, the fluid pressure will be recorded again to verify the water level inside the Westbay casing has not changed during sampling and the sample obtained is from outside the Westbay® casing.

- Once the tool has been retrieved at the surface, the sample will be immediately decanted to the appropriately cold laboratory prepared containers. The first portion of the sample drawn from each depth interval will be used to measure field parameters (temperature, pH, EC, DO, and turbidity). All measurements (including the location, time, and date of measurement) will be recorded on the field sampling forms.

- The remainder of the sample will be placed in laboratory provided containers in the following order: VOC containers will be filled first, followed by 1,4-dioxane (if required), NDMA, and one sample container for perchlorate, nitrate, and sulfate. VOCs containers should be filled allowing no headspace. After filling, the cap will be replaced and the container inverted and tapped sharply against the palm of the hand to check for bubbles. If bubbles are present, the sample will be discarded and a new VOC container will be filled. All samples will immediately be placed in an iced cooler.

- Once sampling has been completed, barometric pressure will be collected at the surface

BLAINE TECH SERVICES
METHODS AND PROCEDURES

PFAS SAMPLING

February 2021

APPLICABILITY

Blaine Tech Services (Blaine Tech) performs field sampling as per the current guidance document provided by the California State Water Quality Control Board:

> California State Water Quality Control Board. Division of Water Quality.
> **"Per- and Polyfluoroalkyl Substances (PFAS) Sampling Guidelines for Non-Drinking Water." September, 2020.**

The SWQCB document is the reference document for collection of PFAS samples.

This document, "Blaine Tech Services, Methods and Procedures, PFAS Sampling, February 2021" focuses on areas meriting special mention and Blaine Tech specific protocols only and as such, is intended to be used only as an addendum to the SWQCB reference document.

Any areas not covered below should be presumed addressed in the SWQCB guidance which is attached. Deference to more restrictive guidance documents will be made as required where client, site or other state specific documents exist.

GUIDANCE DOCUMENTS & PROTOCOLS REVIEW

Prior to field work, the technician(s) will:

- Review the applicable guidance documents such as the CA SWQCB or other state PFAS guidance documents and site-specific SAPs
- Review the *PFAS Sampling Checklist*
- Review and establish protocols for restrictions related to PFAS sampling including those pertaining to PPE, clothing, and eating
- Review sampling equipment options and selections for the site

SAMPLING VEHICLE PREPARATION

Sampling vehicle will be prepared by removing all known or suspected PFAS sources including Hi-Viz rain gear, Gore-Tex or Scotchgard treated clothing, coated Tyvek PPE, Teflon bailers, and/or connectors, Teflon tape, food, food packaging and non-allowable sun screen and insect repellant.

CLOTHING

Technicians must wear well washed 100% cotton clothing. Hi-Viz vests and raingear are not permitted.

GLOVES

New Nitrile gloves will be worn exclusively while handling sampling equipment and materials and during all gauging, sampling and sample collection activities.

Coated Kevlar gloves are not permitted to be worn during gauging or sampling related activities.

Kevlar gloves *may* be worn under new Nitrile gloves during well box and/or standpipe opening and closing activities. If worn, technicians will wash hands after removing Kevlar gloves and don new Nitrile gloves prior engaging in sampling related activities.

STEEL-TOED BOOTS

Due to health & safety concerns, steel-toed, leather boots are allowed in the Work Area.

> **Since it is highly likely that leather boots have water resistant treatment, no physical hand contact with the boots is permitted during gauging and sampling activities.**

If boots must be re-secured or handled between sampling activities, technicians will don new Nitrile gloves prior to handling and wash hands and don new Nitrile gloves after handling.

SUNSCREENS AND INSECT REPELLENT

PFAS may be present in many personal care products such as sunscreens and insect repellents. Only the following brands and products are acceptable for personal use*:

Sunscreens:

- Banana Boat® for Men Triple Defense Continuous Spray Sunscreen SPF 3
- Banana Boat® Sport Performance Coolzone Broad Spectrum SPF 30
- Banana Boat® Sport Performance Sunscreen Lotion Broad Spectrum SPF 30
- Banana Boat® Sport Performance Sunscreen Stick SPF 50
- Coppertone® Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50
- Coppertone® Sport High Performance AccuSpray Sunscreen SPF 30
- Coppertone® Sunscreen Stick Kids SPF 55
- L'Oréal® Silky Sheer Face Lotion 50
- Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 50
- Meijer® Sunscreen Continuous Spray Broad Spectrum SPF 30
- Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50
- Meijer® Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70

- Neutrogena® Beach Defense Water+Sun Barrier Lotion SPF 70
- Neutrogena® Beach Defense Water+Sun Barrier Spray Broad Spectrum SPF 30
- Neutrogena® Pure & Free Baby Sunscreen Broad Spectrum SPF 60+
- Neutrogena® UltraSheer Dry-Touch Sunscreen Broad Spectrum SPF 30

Insect Repellents:

- OFF® Deep Woods
- Sawyer® Permethrin

* Michigan Department of Environmental Quality. “MDEQ PFAS Sampling Quick Reference Field Guide”

WORK AREA CONTROL

A sterile, PFAS-free Work Area is a vital component of successful PFAS sampling. From the time the vehicle is staged at the well until PFAS sampling is completed at that well, the Work Area will be controlled, typically containing only the following items:

- Nitrile Gloves
- 100% Cotton Clothing
- HDPE Hardhat (if required)
- Metal Clipboard
- Ball Point Pen(s)
- PFAS Sample Container(s)
- Paper
- Sampling Equipment & Materials

Items typically found in the Work Area during non-PFAS sampling that are not permitted while performing PFAS sampling include:

- Raingear
- Hi-Viz Vest and/or Sun Scarf
- Water Resistant Pants
- Hi-tech Breathable Shirt
- Kevlar Gloves
- Sun Umbrella
- Sample Labels
- Baggies
- Sharpie Pens

The Work Area should be physically separated from the Support Areas, typically by closing all work/storage boxes and the truck cab doors as shown below:

Sterile Work Area with QED SamplePro Pump, HDPE tubing and YSI Pro Plus Multiparameter Meter & Flow Cell

DAILY CHECKLIST

Technicians performing PFAS sampling will complete the *PFAS Sampling Checklist* (attached) on a daily basis to verify adherence to and note any deviations from planned protocols so that a full and accurate accounting can be provided to our Clients.

SAMPLING EQUIPMENT & MATERIALS

Due to the known or suspected possibility that PFAS containing materials are present in certain types of portable pumps and equipment, allowable options for PFAS sampling are necessarily limited. Acceptable materials include HDPE, polypropylene, silicone, stainless steel nylon, PVC and cotton. Note that this list of acceptable materials applies to support equipment such as cable ties, extension rods, couplings and twine. Equipment options will be reviewed with Clients in light of the most current data to determine the best choices for their specific applications.

SAMPLE CONTAINERS

Only laboratory provided, PFAS specific containers will be used to collect PFAS samples. Typically these are HDPE containers fitted with Teflon-free caps.

SAMPLE COLLECTION

Sample containers for PFAS will be kept segregated and unlabeled until after they are filled and sealed to ensure no cross-contamination from labels, pens and baggies.

DECONTAMINATION

All re-usable equipment is brought to the site in clean and serviceable condition and is cleaned after use in each well and before subsequent use in any other well. Default decontamination consists of:

- Rinse with deionized water
- Wash with Liquinox + deionized water
- Triple rinse with laboratory supplied PFAS-free water

DEDICATED PFAS SAMPLERS

All attempts will be made to limit the number of technicians accessing and sampling any subset of PFAS sampled wells during field activities where 1) project size dictates the need for multiple sampling technicians onsite and 2) only a subset of the sampled wells require PFAS analysis. This decreases variability and ensures that results are as comparable as possible.

PFAS SAMPLES FIRST

When PFAS sampling is being “piggy-backed” onto an otherwise routine sampling event involving collection of other analytes, technicians will ensure that PFAS sampling is completed at an individual well with the PFAS bottle set labeled, bagged and placed on ice prior to collecting samples for other constituents at the well.

2020

Per- and Polyfluoroalkyl Substances (PFAS) Sampling Guidelines for Non-Drinking Water

CALIFORNIA STATE WATER QUALITY CONTROL BOARD

DIVISION OF WATER QUALITY

SWRCB PFAS Website: https://www.waterboards.ca.gov/pfas/
DDW PFAS Website: https://www.waterboards.ca.gov/drinking_water/certlic/drinkingwater/PFOA_PFOS.html

September 2020

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INTRODUCTION AND PURPOSE OF SAMPLING GUIDE

Per- and polyfluoroalkyl substances (PFAS) are a class of manufactured compounds that are extensively used to make everyday items more resistant to stains, grease, and water. These chemicals have been used in a variety of industrial, commercial, and consumer products and some of these products are present and/or used during routine sampling events. Use of these products could potentially contaminate samples during preparation of the sampling site, sample collection, decontamination of sample equipment, or shipment and storage of samples. With a relatively high probability of PFAS cross-contamination, care must be taken to design and implement effective PFAS sampling procedures. Therefore, this guidance document was developed to outline steps to take during PFAS sampling events to prevent or minimize sample contamination.

The Water Boards recognizes that there may be different sampling procedures that are recommended by a laboratory or in other guidance documents or standard operating procedures for PFAS sampling. However, this guidance document has been developed to assist samplers during PFAS sampling events to meet the quality objectives of the State Water Board and should be reviewed prior to collecting PFAS samples for State Water Board programs or projects. Users of this guide should make every effort to implement these sampling recommendations. There may be updates to this guidance document as more is learned about the sources of PFAS and effective measures or sampling procedures to prevent PFAS contamination during sampling events.

2.0 PROJECT PLANNING

The Water Boards recommend that a project-specific Quality Assurance Project Plan (QAPP) be developed and approved for PFAS sampling events prior to conducting the sampling. For compliance to the PFAS sampling requirements in the Recycled Water Policy, the development and approval of a QAPP is required. The QAPP should include, at minimum, the project objectives, a project organization with responsible individuals and respective duties identified, sampling design and procedures, analytical methods requirements, reporting requirements, and data assessment procedures. Additionally, the QAPP should clearly identify the PFAS-specific sampling procedures and necessary preventative measures required to prevent sample contamination from sources of PFAS, including sample equipment decontamination procedures and information on prohibited and acceptable sample containers, field clothing, personal protective equipment (PPE), personal products, food packaging, and sampling conditions.

Additional guidelines for the preparation of a QAPP can be found at:

- Water Board's QAPP Development Resources website, and
- U.S. Environmental Protection Agency's (EPA) *Guidance for Quality Assurance Project Plans*, EPA QA/G-5 (December 2002).

In the absence of a QAPP, it is important that a thorough, site-specific sampling plan is developed prior to sampling for PFAS. Since PFAS are prevalent in everyday products, the risk for cross contamination is elevated and preventing or minimizing contamination during sampling events will require extra steps to the sampling procedure. This sampling guide should be fully reviewed and can be used as a resource when preparing a sampling plan or QAPP. This will ensure that acceptable sampling supplies and sampling equipment are used and activities on site are performed in the correct order and location. The plan should include a setup of the staging area, including a checklist of sampling supplies and personal protective equipment, as well as the procedure for decontamination when moving in and out of the staging area. A site-specific sampling plan will help prevent errors and ensure a successful PFAS sampling event.

3.0 PRE-SAMPLING CONSIDERATIONS

Sampling materials and field supplies like plastic bags and sample containers, as well as, waterproof pens and paper, personal protective equipment, clothing, food packaging, and personal care products all have been known to contain PFAS compounds. Since PFAS are used in many traditional sampling equipment, materials, or products, and can be sources of contamination, this guidance document has divided commonly used sampling materials and field supplies into three categories:

1. ***Acceptable materials***: These materials are not known to be sources of PFAS cross contamination and can be used during all sampling stages and in the immediate sampling environment.
2. ***Staging area-only materials***: These materials may contain PFAS and should not come into direct contact with the sample but can be used in the staging area. However, these materials should be used away from sample bottles and equipment, and care should be taken to thoroughly wash hands and use new gloves after handling any of these materials.
3. ***Prohibited materials***: These materials are known to contain PFAS that may present a threat to the integrity of the sample and should not be used during any stage of the sampling event.

3.1 Sampling Equipment

Sampling equipment used for PFAS sampling must be made from acceptable materials, which include high-density polyethylene (HDPE), polypropylene, silicone, stainless steel, nylon, polyvinyl chloride (PVC), acetate, and cotton. Sampling equipment that contain PFAS-based (fluoropolymers) parts that would be in direct contact with the sample or sampling environment are prohibited. These fluoropolymers include, but are not limited to:

- Polytetrafluoroethylene (PTFE), including the trademark Teflon® and Hostaflon®, which can be in ball check-valves on certain bailers, lining of some hoses and tubing, wiring, certain kinds of gears, lubricant, and some objects that require the sliding action of parts.
- Polyvinylidene fluoride (PVDF), including the trademark Kynar®, which can be in tubing, films/coatings on aluminum, galvanized or aluminized steel, wire insulators, and lithium-ion batteries.
- Polychlorotrifluoroethylene (PCTFE), including the trademark Neoflon®, which can be in many valves, seals, gaskets, and food packaging.
- Ethylene-tetrafluoro-ethylene (ETFE), including the trademark Tefzel®, which can be in many wire and cable insulation and covers, films for roofing and siding, liners in pipes, and some cable tie wraps.
- Fluorinated ethylene propylene (FEP), including the trademarks Teflon® FEP and Hostaflon® FEP, and may also include Neoflon®, which can be in wire and cable insulation and covers, pipe linings, and some labware.

Equipment that contain PFAS-coated parts (e.g. Teflon-coated parts) can be used if the PFAS-coated part is internal to the equipment and is not in direct contact with the external environment or the sample. Sampling equipment that have parts made of low-density polyethylene (LDPE) should be avoided if the part comes in direct contact with the sample. However, if it is absolutely necessary, equipment that have parts made of LDPE may be used if an equipment blank has confirmed it to be PFAS-free.

Sampling equipment used for grab sampling, including cable ties, extension rods, and couplings, should be made of materials that are known to be PFAS-free. Recommended materials for this sampling equipment include:

- Cable ties made of natural rubber or nylon or uncoated metal springs.
- Extension rods made of materials that are known to be PFAS-free.

- Stainless-steel couplings

Automatic sampling has an increased potential for cross-contamination because the tubing, valves, strainers, suction lines, distribution nozzles, and other parts may be made from PFAS (fluoropolymers). Automatic sampling should only be used if a representative sample cannot otherwise be collected. If automatic sampling is used, then parts made from preferable materials including high-density, polyethylene (HDPE), polypropylene, silicone, stainless steel, nylon, PVC, and acetate should be used when possible. It is recommended that parts on the sampler be screened prior to sampling by reviewing the safety data sheets (if available) and collecting an equipment blank to verify that the parts are PFAS-free. Additionally, the strainer should be decontaminated or replaced between each sampling event.

Regardless of the sampling set-up or equipment selected, an equipment blank must be taken to verify that the equipment is not contaminating the sample.

3.2 Sample Containers

All sample containers used for PFAS sampling should come from the laboratory that is performing the PFAS analysis. High-density polyethylene (HDPE) or polypropylene sample bottles with Teflon®-free caps are the preferred sampling containers for PFAS sampling. PFAS may adsorb to glass containers and therefore should not be used for water, leachate, or other aqueous samples. Glass containers may be used for dry or solid samples, provided that adsorbed PFAS can be extracted by laboratory as part of the sample preparation procedure. Sample containers made from low-density polyethylene (LDPE) should not be used as PFAS are used in the manufacturing process of these containers. LDPE can be found in many sample containers including bottles and plastic bags.

3.3 Personal Protective Equipment and Field Clothing

PFAS are used to coat various clothing and personal protective equipment (PPE) to repel water, oil, and dirt. While preparing for sampling, pay attention to clothing or PPE that is advertised to have waterproof, water-repellant, or dirt and/or stain resistant characteristics because these types of clothing may have had PFAS used in their manufacturing and can be a source of contamination. However, personal safety is paramount and should not be compromised to prevent cross-contamination. Therefore, if the use of PPE is necessary to ensure the health and safety of sampling personnel and no PFAS-free alternative is available, then record the use of the PPE in the field notes and/or the chain of custody and discuss in the final analytical report, if necessary.

There are several industry standard PPE items that may be required during sampling events that have not been evaluated for PFAS, including hard hats, safety glasses, and uncoated Tyvek® products. If these items are used during the sampling event, then they should be screened by collecting an equipment blank prior to use. Additionally, if PPE is used and it is unknown if PFAS are used in its manufacturing, then collecting an equipment blank prior to use is recommended. For reference purposes, Table 1 below lists examples of clothing and personal protective equipment that should and should not be used during PFAS sampling events.

Table 1. Examples of Clothing and PPE Acceptable to Prohibited during PFAS Sampling

Acceptable materials	Staging area materials	Prohibited materials
• Synthetic or 100% cotton clothing that has been well-laundered (without use of fabric softener) • Waterproof clothing made with polyurethane, PVC, wax-coated fabrics, rubber, or neoprene • Boots made of polyurethane and/or PVC • Powderless nitrile gloves	• Non PFAS-free boots (e.g. steel-toed) • First-aid adhesive wrappers Note: Hands should be washed and gloves changed after handling these products.	• Water/stain/dirt-resistant treated clothes (including but not limited to Gore-Tex™, Scotchgard™, and RUCO®) • New unwashed clothing • Clothes recently washed with fabric softeners • Clothes chemically treated for insect resistance and ultraviolet protection • Coated Tyvek® • Latex gloves

3.4 Sun and Biological Protection

Biological hazards (UV from sun, mosquitos, ticks, etc.) may be encountered during sampling, so the elimination of specific clothing materials, sunscreens and insect repellants that are known to contain PFAS may not be possible because it could pose a health and safety hazard to field staff. While the potential for sunscreen, insect repellants, and personal products to contaminate PFAS samples is an active area of research, the personal safety of field staff is of top priority. Therefore, any deviation from this guidance document, including those necessary to ensure the health and safety of

field staff, should be recorded in field notes and/or the chain of custody and discussed in the final analytical report.

Sunscreens may be needed if field staff are subject to prolonged sun exposure. Sunscreens may have been manufactured using PFAS and could potential be a source of cross-contamination. Similarly, protection against insects may require the use of insect repellant, which also may have been manufactured with PFAS. Therefore, it is important to be aware of the sunscreen or insect repellant selected for use during a PFAS sampling events. The words "natural" and/or "organic" in a product name or used to describe the product does not mean that the product is PFAS-free. More information on sunscreens and insect repellants can be found in Michigan's PFAS Sampling Quick Reference Field Guide. Note that this is not a comprehensive list of sunscreens or insect repellants so other products not listed may meet the requirements for use. Listing or omission of any product does not imply endorsement or disapproval of the product. Also, there is no guarantee that these products will always remain PFAS-free.

If sunscreens or insect repellants are used during a PFAS sampling event, then the product should be applied in the staging area. Hands should be washed and new gloves used following application.

3.5 Personal Care Products

Many personal care products, including cosmetics, moisturizers, fragrances, and creams may contain PFAS or may become contaminated with PFAS from the containers they are supplied in. For this reason, the use of such products should be avoided or minimized on the day of sampling, and 24 hours prior to sampling. The words "natural" and/or "organic" in a product name or used to describe the product does not mean that the product is PFAS-free. More information on personal care products can be found in The Environmental Working Group's Skin Deep Guidance. Note that this is not a comprehensive listing of personal care products. Listing or omission of any product does not imply endorsement or disapproval of the product. Also, there is no guarantee that products will always remain PFAS-free, or maintain their status as possibly containing PFAS.

3.6 Food Packaging

PFAS are known to be prevalent in food packaging, including paper plates, aluminum foil, paper towels, food containers, bags, and wraps. Although long-chain PFAS have been banned for use in the manufacturing of contact food materials in the United States, short-chain PFAS have not been banned. Therefore, these products could be source of PFAS contamination. If food or beverages are to be consumed during the sampling event, then a dedicated eating area should be included in the sampling site plan (see Section 5.1 below).

4.0 DECONTAMINATION

Sampling equipment must be cleaned and decontaminated prior to use. Conventional procedures for cleaning and decontaminating sampling equipment can be used but must include a triple rinsing with PFAS-free water and adhere to the following decontamination guidance:

- Use of laboratory supplied PFAS-free deionized water is preferred for cleaning and decontamination.
- Commercially available deionized water may be used for cleaning and decontamination if the water is verified to be PFA-free.
- Municipal drinking water may be used for cleaning or decontamination if the water is known to be PFAS-free.
- Do not use Decon 90®
- Alconox®, Liquinox®, and Citranox® can be used for equipment cleaning and decontamination.
- Sampling equipment can be scrubbed using a polyethylene or Polyvinyl chloride (PVC) brush to remove particulates.

5.0 SAMPLING PROCEDURES

Conventional sampling procedures can be used to collect samples for PFAS analysis. However, there are PFAS-specific considerations and preventative measures that must be followed to prevent contamination of samples. It is recommended that supplies used for PFAS sampling that will come in direct contact with PFAS samples; including sample containers, pumps/tubing/collection equipment, and DI water be stored separately from other sampling supplies. These items should also be handled minimally, to reduce the risk of cross contamination from other field equipment. Hands must be washed and cleaned. Powderless nitrile gloves must be worn on hands before collecting samples, handling sample containers, or handling sampling equipment.

5.1 Site Set-Up

The sampling site should be evaluated prior to sampling to identify potential contamination risks and to select dedicated eating, staging, and sampling areas.

- ***Eating Area***: The eating area is separate from the sampling and staging areas, and the only place where food and drink should be stored and consumed. Food packaging must not be in the sampling and staging areas during sampling due to the potential for PFAS cross-contamination.
- ***Staging Area***: The staging area is where equipment is set-up and personal protective equipment is put on and taken off. PFAS-free over-boots and PPE should be put on in the staging area prior to sampling activities
- ***Sampling Area***: Sampling areas are the areas of the field where samples are collected. When staff requires a break to eat or drink, they should move to the staging area before removing gloves, coveralls, and any other appropriate PPE, if worn. Staff should move to the designated eating area for food and beverage consumption. When finished, staff should wash their hands and put on a fresh pair of powderless nitrile gloves and appropriate PPE at the staging area, before returning to the sampling area.

Before sampling begins, a sampling sequence should be established. To prevent cross-contamination, sampling should start in areas suspected to be least contaminated and continue to areas suspected to be most contaminated. If there is no existing sampling data from the area, potential PFAS sources and transport paths should be reviewed to help inform the best sampling sequence.

If multiple samples will be collected in an area where PFAS has been documented, SAMPLING SHOULD BEGIN IN AREAS THAT ARE KNOWN TO BE UPGRADIENT FROM THE SUSPECTED SOURCE, FOLLOWED BY THOSE THAT ARE FURTHEST DOWNGRADIENT. DOWNGRADIENT LOCATIONS SHOULD BE PROGRESSIVELY SAMPLED FROM THE FURTHEST DOWNGRADIENT TO THE CLOSEST SUSPECTED PFAS SOURCE.

5.2 Sample Collection

Sample collection procedures should include standard best practices for environmental sampling to prevent contamination and ensure a representative sample is collected. In addition, the following protocols should be followed when collecting PFAS samples:

- The sample container must be kept sealed and only opened during sample collection. The sampling container cap or lid should never be placed on the ground or on any other surface unless it is PFAS-free.
- When collecting and handling water samples, do not insert or let tubing or any materials inside the sample bottle. Dust and fibers must be kept out of sample bottles.

- If sample filtration is necessary, it should be performed by the laboratory. The laboratory may also be able to perform other methods, such as centrifuging, to reduce the need for filtration. Field filtration is not advised due to risks of contamination and sorption of PFAS to sampling media.

5.3 Sample Labeling and Field Documentation

Additional considerations should be taken when labelling samples and documenting field activities to prevent contamination including the following:

- Regular/thick size markers (Sharpie® or otherwise) should be avoided as they may contain PFAS.
- Fine and Ultra-Fine point Sharpie® markers are acceptable to label the empty sample bottle while in the staging area provided the lid is on the sample bottle and gloves are changed following sample bottle labeling.
- Ballpoint pens may be used when labeling sample containers. If ballpoint pens do not write on the sample container labels, preprinted labels from the laboratory may be used.
- Do not use sticky notes (e.g. Post-it Notes®), plastic clipboards, or waterproof paper and notebooks in the sampling area.
- Rite in the Rain® notebooks are acceptable to use in the staging area provided gloves are changed after note taking.

5.4 Sample Shipment and Storage

Samples must be chilled during storage and shipment. Temperatures must not exceed 50°F (10°C) during the first 48 hours after collection. Chemical or blue ice should not be used for storage or shipment of PFAS samples. When preparing samples for transportation or shipment, the samples and ice should be double bagged using bags made of materials that do not present a PFAS contamination risk, such as HDPE, if possible. LDPE bags may be used for bagging samples if special precautions are taken. LDPE bags should be kept separate from other sampling supplies in the staging area and should not come into direct contact with the sample media. Gloves should be changed after handling LDPE bags.

6.0 MATRIX-SPECIFIC SAMPLING CONSIDERATIONS

The following sections provide general guidelines to follow for matrix-specific sampling. The matrices included in this guide are not all inclusive but are those matrices currently being included per State Water Board's Investigative Orders (order).

6.1 General Guidelines

Although PFAS sampling guidance is provided in this document, field staff should refer to the project-specific QAPP and/or applicable analytical method for sample testing, collection, preservation, holding times, and storage requirements. If sampling is occurring under an order, refer to the order in addition to this document for additional requirements or guidance on sampling.

6.2 Wastewater

The point in the waste stream that should be sampled will be determined in the order or in a project-specific QAPP. The laboratory performing the PFAS analysis should be consulted before sampling to develop the sampling strategy. When selecting the best means for achieving a representative sample (automatic composite, grab-composite, single grab), consult with the laboratory performing the analysis. The laboratory may have to comply with specific requirements in the analytical method that determine the sample volume, number of samples, container types, and/or ability to dilute samples for analysis. Additionally, the number of samples sufficient to comprise a representative composite sample will vary depending on the project and should be determined through discussions with the regulator and laboratory.

PFAS are expected to accumulate at the air/water interface. Therefore, unless specifically required in the QAPP, it is not advisable to collect samples from the very top layer of any wastewater, as it is may not be representative of the bulk wastewater.

6.3 Surface water

Unless specifically required by the project objectives, surface water samples should not be taken at the top layer of the water body or of surface scums. PFAS are expected to accumulate at the surface water air interface or be present in the surface runoff, so samples taken at the surface are likely to result in high biased results that may not be representative of the bulk surface water.

6.4 Groundwater Wells

Prior to purging, the water level in the well and the total depth of the well should be measured to determine the volume of water in the well. Record the well purging method and field parameters (i.e. temperature, electrical conductivity, pH, and turbidity).

- ***Supply Wells*** - Activate the well and flush until the water temperature has stabilized, or until a minimum of one well casing volume has been flushed out. Wells should be allowed to flow for a minimum of 15 minutes before sampling to ensure that the sample reflects the water quality of the source. The sample tap should be flushed for a minimum of 5 minutes to ensure the impact of local sources of PFAS cross-contamination, such as tape and valve seats, are minimized.

- ***Monitoring Wells*** - Low-flow purge methods are preferred. For more information on groundwater sampling procedures, including suggestions for low-flow sampling methods, refer to the DTSC Guide for Representative Sampling of Groundwater for Hazardous Substances. (https://dtsc.ca.gov/wp-content/uploads/sites/31/2018/09/Representative_Sampling_of_GW_for_Haz_Subst.pdf)

  If low-flow sampling is not possible, then a submersible pump (dedicated or non-dedicated) or bailer can be used to sample the well. Ensure any purging components (pumps, bailers) or tubing are PFAS free. If it is uncertain whether the pump and/or tubing components are PFAS free, an equipment blank is recommended to determine the potential PFAS contribution by the equipment in contact with the sample. Nylon or cotton are recommended materials for the line attached to bailers for sample collection.

Purging should continue until the selected indicator parameters have stabilized. For suggested stabilization criteria, please refer to Table 2 below.

TABLE 2. Suggested Well Purge Stabilization Criteria for Water-Quality-Indicator Parameters (selected from DTSC's *Representative Sampling of Groundwater for Hazardous Substances,* 2008*)*

Parameter	Stabilization Criteria	Reference
Temperature	± 3% of reading (minimum of ± 0.2° C	SAM 2002
pH	+/- 0.1	Puls and Barcelona, 1996; USGS 2006
specific electrical conductance (SEC)	+/- 3%	Puls and Barcelona, 1996

6.5 Biosolids

If sampling is occurring under an order, always refer to the order for additional requirements or guidance on sampling, including the point in the waste stream that should be sampled.

Biosolids (and/or sewage sludge) typically contain both liquid and solid fractions. Therefore, as a general rule, samples should be collected with the highest solids content possible. When selecting the best means for achieving a representative biosolids sample (composite or single grab), consult with the laboratory performing the analysis prior to sampling. The laboratory may have to comply with specific requirements in the analytical method that determine the sample volume, number of samples, container types, and/or specific guidelines for biosolids samples, such as solids content percentage thresholds. Additionally, the number of samples sufficient to comprise a representative composite sample will vary depending on the project and should be determined through discussion with the regulator and laboratory.

Samples should be collected after treatment processes and prior to disposal (leaving the facility). If liquids are present, a representative whole sample aliquot that includes both liquid and solid fractions should be collected. However, samples with the least amount of liquid are preferred.

To ensure that biosolids samples are representative, consider whether it is appropriate to take a grab sample or a composite sample. A stream from a mechanical or treatment process where the biosolids and sludge are expected to be well mixed, is more likely to be representative of the composition of the stream. In this case, a single grab sample may sufficiently represent the composition of the biosolid stream passing through the sampling point. In cases where biosolids have undergone dewatering and have been stored in locations such as drying beds, various storage tanks, or compost piles; a composite sample may be more representative. If a composite sample is to be collected, consider requesting the laboratory to prepare the composite prior to analysis.

The following information should be collected and recorded during biosolids sampling, in addition to any other items required by the order, QAPP, laboratory, or project workplan.

- An estimate of the moisture content of each sample, reported as percentage by weight of solid for a given volume of sample.
- The sample location (if compositing used, a representative single location should be reported)
- A description of the sample location, biosolids classification, and step within the treatment plant's material processing sequence that the sample was taken.

- Moisture content should be tested by the laboratory on the biosolids sample
- All biosolids and sludge samples, including those with low solids content, should be analyzed as solids and reported in nanograms per kilogram (ng/kg) on a dry weight basis. This dry weight basis reporting requirement should be specified on the chain-of-custody sent to the laboratory.

6.6 Soil and Sediment

High-density polyethylene (HDPE) or polypropylene sample bottles with Teflon®-free caps are the preferred sampling containers for PFAS sampling. Glass containers may be used for dry or solid samples, provided that adsorbed PFAS can be extracted by laboratory as part of the sample preparation procedure. Bags used to store soil or sediment samples should be verified to be PFAS-free prior to using for storage.

Sampling equipment used to collect soil or sediment samples for PFAS analysis must be made from acceptable materials. If collecting core samples, liners for core samplers should be made of acetate or other materials known to be PFAS-free.

7.0 FIELD QUALITY CONTROL SAMPLES

Due to the prevalence of PFAS in a wide range of materials, there may be a greater likelihood for cross-contamination during sampling, transport, and storage of samples. As such, it is recommended to collect field quality control samples to evaluate whether or not cross-contamination has occurred. The type and frequency of quality control samples should be identified in the project-specific QAPP. Additionally, analytical methods for PFAS analysis may provide instructions on the frequency and type of quality control samples required per sampling event.

7.1 Field Duplicate - Recommended

Field duplicates are replicate samples collected in the field and submitted to the laboratory as two distinct samples. Field duplicates are used to verify the precision of field and laboratory activities. The Field Duplicate (FD) is a sample collected from a sample location at the same time and under identical circumstances as the field sample and treated the same throughout field and laboratory procedures.

7.2 Field Blank - Required

A Field Blank (FB) is collected to verify that the sampling environment does not introduce PFAS and cross-contaminate samples during the sampling event. For the analysis of aqueous matrices, the field blank is collected by pouring PFAS-free reagent

water that is stored in an acceptable sample container for PFAS sampling into an empty, clean sample container at the sampling site. The sample containers and supplies to process a field blank should be prepared and provided by the laboratory prior to the sampling event. The field blank is treated the same throughout field and laboratory procedures.

7.3 Equipment Blank - Required

Equipment blank samples are collected by passing laboratory-verified PFAS-free water over or through decontaminated field sampling equipment before the collection of field samples to assess the adequacy of the decontamination process and/or to evaluate potential contamination from the equipment used during sampling.

7.4 Trip Blank – Not Required

Trip blanks are a bottle of PFAS-free water that is prepared in the laboratory, travels from the laboratory to the site, and then gets transported back to the laboratory without having been exposed to any sampling procedures. The trip blank sample is used to assess cross-contamination introduced from the laboratory and during shipping procedures.

8.0 APPENDICES

8.1 Useful Links

The following links are provided to be supplemental information to this guidance document and may be useful for PFAS sampling. Information from these resources must not be used as a substitute for the required or recommended practices outlined in this guidance document, nor the project-specific QAPP or analytical method for PFAS analysis.

- US EPA PFAS methods and guidance for sampling and analyzing water and other environmental media (Technical Brief)
- US EPA PFAS Website
- The State of Michigan Department of Environmental Quality (MDEQ) PFAS sampling guides
- MDEQ PFAS Sampling Quick Reference Field Guide
- Interstate Technology and Regulatory Council (ITRC) PFAS Technical and Regulatory Guidance Document website

8.2 Entering Data in Geotracker

For some PFAS sampling projects, the geographic locations of sample sites must be recorded in the field in the form of latitudes/longitudes and then uploaded into GeoTracker. These points can be non-surveyed field points, meaning that a licensed surveyor is not necessary to measure these points; a handheld GPS device or obtaining the coordinates using a readily available mapping program on the internet is sufficient. Step-by-step instructions for creating and uploading non-surveyed field points can be found in the document "How Do I Upload? Electronic Submittal of Information (ESI) Guide" under the "Getting Started" section. Templates and data formatting requirements can be found on the Electronic Submittal of Information (ESI) for GeoTracker home page (https://www.waterboards.ca.gov/ust/electronic_submittal/index.html).

When uploading the PFAS data into GeoTracker, the PARLABELs/CAF numbers for each unique PFAS is needed. The GeoTracker PARLABELs/CAF numbers are listed at the end of this guide but can also be found on the State Water Resources Control Board's Per- and Polyfluoroalkyl Substances (PFAS) webpage in the information and resources for Non-Drinking Water.

9.0 REFERENCES

Bartlett, Samuel A. and Davis, Katherine L. "Evaluating PFAS cross contamination issues." *Remediation*. 2018; 28:53-57.

Department of Defense Environmental Data Quality Workgroup. "*Bottle Selection and other Sampling Considerations When Sampling for Per and Poly-Fluoroalkyl Substances (PFAS)*." 2017. (https://www.denix.osd.mil/edqw/home/what-s-new/unassigned/edqw-pfas-sampling-factsheet-rev-1-2-july-2017/EDQW%20PFAS%20Sampling%20Factsheet%20Rev%201.2%20July%202017.pdf)

Department of Toxic Substances Control. "*Representative Sampling of Groundwater for Hazardous Substances*." Revised February 2008 (https://dtsc.ca.gov/wp-content/uploads/sites/31/2018/09/Representative_Sampling_of_GW_for_Haz_Subst.pdf)

Government of Western Australia, Department of Environment Regulation. "*Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS)*." January 2017. (https://www.der.wa.gov.au/images/documents/your-environment/contaminated-sites/guidelines/Guideline_on_Assessment_and_Management_of_PFAS_v2.1.pdf)

Heads of EPAs Australia and New Zealand. "PFAS National Environmental Management Plan." January 2018. (https://www.epa.vic.gov.au/-/media/epa/files/for-community/pfas-national-environmental-management-plan/final_pfas-nemp-20180110.pdf)

Interstate Regulatory Technology Council (ITRC). "Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS)." April 2020. (https://pfas-1.itrcweb.org/wp-content/uploads/2020/04/PFAS_Fact_Sheet_Site_Characterization_April2020.pdf)

Michigan Department of Environmental Quality. "General PFAS Sampling Guidance." 16 October 2018. (https://www.michigan.gov/documents/pfasresponse/General_PFAS_Sampling_Guidance_634597_7.pdf)

Michigan Department of Environmental Quality "Groundwater PFAS Sampling Guidance." October 2018. (https://www.michigan.gov/documents/pfasresponse/Groundwater_PFAS_Sampling_Guidance_637871_7.pdf)

Michigan Department of Environmental Quality "Biosolids and Sludge PFAS Sampling Guidance." November 2019. (https://www.michigan.gov/documents/pfasresponse/Biosolids_and_Sludge_PFAS_Sampling_Guidance_+_Quick_Reference_Field_Guide_679307_7.pdf)

Michigan Department of Environmental Quality "Soil PFAS Sampling Guidance." November 2018. (https://www.michigan.gov/documents/pfasresponse/Soil_PFAS_Sampling_Guidance_639407_7.pdf)

Michigan Department of Environmental Quality "Wastewater PFAS Sampling Guidance". October 2018. (https://www.michigan.gov/documents/pfasresponse/Wastewater_PFAS_Sampling_Guidance_636791_7.pdf)

Naval Facilities Engineering Command (NAVFAC). "Interim Per- and Polyfluoroalkyl Substances (PFAS) Site Guidance for NAVFAC Remedial Project Managers (RPMs)/September 2017 Update." September 2017.

Puls, R.W., and M.J. Barcelona. 1996. "Low-Flow (Minimal Drawdown) Groundwater Sampling Procedures." U.S. EPA Superfund Groundwater Issue, EPA/504/S-95/504. (https://www.csus.edu/indiv/h/hornert/Geol_210_Summer_2012/Week%202%20readings/Puls%20and%20Barcelona%201996%20Low%20flow%20sampling.pdf)

United States Geological Survey, "National Field Manual for the Collection of Water-Quality Data, Chapter A4 Collection of Water Samples". September 2006. https://pubs.usgs.gov/twri/twri9a4/twri9a4_Chap4_v2.pdf

Chemical Name/ Abbreviation	Geotracker PARLABEL	Chemical Abstracts Service (CAS) No.
Perfluorobutanoic acid (PFBA)	PFBTA	375-22-4
Perfluoropentanoiic acid (PFPeA)	PFPA	2706-90-3
Perfluorohexanoic acid (PFHxA)	PFHA	307-24-4
Perfluoroheptanoic acid (PFHpA)	PFHPA	375-85-9
Perfluorooctanoic acid (PFOA)	PFOA	335-67-1
Perfluorononanoic acid (PFNA)	PFNA	375-95-1
Perfluorodecanoic acid (PFDA)	PFNDCA	335-76-2
Perfluoroundecanoic acid (PFUnDA, PFUda, PFUnA)	PFUNDCA	2058-94-8
Perfluorododecanoic acid (PFDoDA, PFDoA)	PFDOA	307-55-1
Perfluorotridecanoic acid (PFTrDA)	PFTRIDA	72629-94-8
Perfluorotetradecanoic acid (PFTeDA, PFTA)	PFTEDA	376-06-7
Perfluorohexadecanoic acid (PFHxDA)	PFHXDA	67905-19-5
Perfluorooctadecanoic acid (PFODA)	PFODA	16517-11-6
Perfluorobutane sulfonic acid (PFBS)	PFBSA	375-73-5
Perfluoropentane sulfonoic acid (PFPeS)	PFPES	2706-91-4
Perfluorohexane sulfonic acid (PFHxS)	PFHXSA	355-46-4
Perfluoroheptane sulfonic acid (PFHpS)	PFHPSA	375-92-8
Perfluorooctane sulfonic acid (PFOS)	PFOS	1763-23-1

Chemical Name/ Abbreviation	Geotracker PARLABEL	Chemical Abstracts Service (CAS) No.
Perfluorononane sulfonic acid (PFNS)	PFNS	474511-07-4
Perfluorodecane sulfonic acid (PFDS)	PFDSA	335-77-3
Perfluorooctanesulfonamide (PFOSA, PFOSAm, FOSA)	PFOSA	754-91-6
N-Ethyl perfluorooctane sulfonamide ethanol (EtFOSE)	ETFOSE	1691-99-2*
N-Methyl perfluorooctane sulfonamide ethanol (MeFOSE)	MEFOSE	24448-09-7
N-Ethyl perfluorooctane sulfonamide (EtFOSA, EtFOSAm)	ETFOSA	4151-50-2
N-Methyl perfluorooctane sulfonamide (MeFOSA, MeFOSAm)	MEFOSA	31506-32-8
N-Methyl perfluorooctane sulfonamidoacetic acid (NMeFOSAA)	NMEFOSAA	2355-31-9
N-Ethyl perfluorooctane sulfonamidoacetic acid (NEtFOSAA)	NETFOSAA	2991-50-6
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	4:2FTS	757124-72-4
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	6:2FTS	27619-97-2
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	8:2FTS	39108-34-4
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	10:2FTS	120226-60-0

Chemical Name/ Abbreviation	Geotracker PARLABEL	Chemical Abstracts Service (CAS) No.
2H,2H,3H,3H-Perfluorohexanoic acid (3:3 FTCA)	3:3FTCA	356-02-5
2H,2H,3H,3H-Perfluorooctanoic acid (5:3 FTCA)	5:3FTCA	914637-49-3
2H,2H,3H,3H-Perfluorodecanoic acid (7:3 FTCA)	7:3FTCA	812-70-4
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	HFPA-DA	13252-13-6
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	ADONA	919005-14-4
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS)	9ClPF3ONS	756426-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS)	11ClPF3OUdS	763051-92-9
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	NFDHA	151772-58-6
Perfluoro(2-ethoxyethane) sulfonic acid (PFEESA)	PFEESA	113507-82-7
Perfluoro-3-methoxypropanoic acid (PFMPA)	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid (PFMBA)	PFMBA	863090-89-5

ng/L = nanogram per liter
µg/kg = microgram per kilogram

PFAS Sampling Checklist

Client/Site Name _____ Date _____

Techncian(s) Name(s) _____

	Yes	As noted below
Reviewed and understand requirements in PFAS sampling guidance documents (BLAINE PFAS Sampling SOP, CA SWQCB PFAS guidance, Site-Specific SAP)	☐	☐
Reviewed restrictions relating to PFAS sampling (PPE, clothing, eating).	☐	☐
Removed known/suspected PFAS sources from vehicle prior to mobilization.	☐	☐
Wearing well-washed, 100% cotton clothing.	☐	☐
Have not used cosmetics, moisturizers, hand creams or simliar products today.	☐	☐
Have not used unapproved sun screen or insect repellant today.	☐	☐
Understand restrictions concerning steel-toed, leather workboots.	☐	☐
Successfully implemented Work Area Control during all sampling related activities.	☐	☐
Only pre-approved sampling equipment and materials were used to complete work	☐	☐
Food and drink limited to cab of sampling vehicle. Hands washed after handling.	☐	☐

Equipment decontamination process consisted of (check steps): N/A

_____ Initial DI water rinse
_____ Liquinox + DI water wash
_____ Second DI water rinse
_____ Final triple-rinse with lab supplied PFAS-free water

Equipment Blank(s) was/were collected from the following equipment: N/A

_____

_____

Note any exceptions or deviations from planned protocols here:

_____

_____

_____

_____

Standard Operating Procedure No. 3
Sample Handling and Management
(PFAS Specific)

Revision:_____0.0_____

Reviewed:___11/01/21___

Reviewer:_____

David Conner, PG

SOP No. 3 – Sample Handling and Management (PFAS Specific)

1.0 PURPOSE AND SCOPE

The purpose of this document is to define the standard operating procedure (SOP) for sample management including sample handling, documentation, and analysis for environmental samples collected for chemical analyses including sediment, soil, surface water and groundwater. This procedure is intended to be used together with the other SOPs.

2.0 EQUIPMENT AND MATERIALS

All equipment and materials must be Per- and Polyfluoroalkyl Substances- (PFAS) free and have not come into contact with a material containing PFAS. The equipment and materials necessary that may be used for sampling management include:

- Shipping forms (non-waterproof, non-recycled loose paper)
- Sample containers (PFAS-free High-Density Polyethylene [HDPE], glass, or polypropylene bottles with non-Teflon® lined bottles or caps)
- Ziploc® bags (as long as the bags do not come into direct contact with the sample media and do not introduce cross-contamination)
- Ice (re-usable chemical ice packs are prohibited)
- Tape (clear and strapping)
- Scissors/knife
- Cooler/ice chest
- Custody seal
- Garbage bags
- Non-waterproof pens (acceptable pens include ballpoint pens); Sharpie® markers are not allowed
- Chain of Custody (COC) Forms (non-waterproof, non-recycled loose paper)
- Metal clipboard (plastic clipboards are not allowed)
- Sample Labels
- Non-waterproof logbook (Rite in the Rain® notebooks acceptable)
- Powderless nitrile gloves
- Preservative (if necessary)
- Packing material
- Trip blank (as necessary)
- Temperature blank

3.0 PROCEDURES FOR SAMPLE HANDLING, DOCUMENTATION, AND ANALYSIS

3.1 Sample Labeling

All sample labels should be filled out with non-waterproof ink. Soil and water sample labels may be supplied by the laboratory. For soil and sediment samples collected in jars and sample bottles for groundwater and surface water analyses, sample labels may be completed and attached prior to sample collection. Labels may be partially completed

prior to sample collection. The date and time should not be completed until the time of sample collection. At a minimum, each label shall contain the following information:

- Project/Facility Name
- Grab or composite sample
- Sampler's company affiliation
- Date and time of sample collection
- Analyses required
- Preservation used
- Sampler's initials
- Filtered (if applicable)
- Sample identification (see Section 5.2 below)

3.2 Sample Nomenclature Scheme

The sample identification (ID) varies significantly with each project. At minimum the sample ID should contain enough information to be correctly associated with a specific sampling location. The sample ID shall also be recorded on the sample form for the respective location. Additionally, Quality Control/Quality Assurance (QA/QC) samples should contain a sample ID such that the laboratory would not know it is a QA/QC sample.

3.3 Sample Handling

This section discusses proper sample containers, preservatives, and handling and shipping procedures. The QAPP also summarize the information contained in this section and also includes the sample holding times for each analysis.

3.3.1 Sample Containers

Certified, commercially clean sample containers shall be obtained from the contract analytical laboratory. If appropriate, the bottles shall be labeled by the laboratory to indicate the type of sample to be collected. Required preservatives shall be prepared and placed in the bottles for aqueous analyses at the laboratory prior to shipment to the site.

3.3.2 Sample Preservation

With the exception of samples that are to be hand-delivered to the laboratory during the day of sample collection, samples will be stored on ice to obtain a temperature of 4°C in an insulated cooler immediately following sample collection. Samples delivered to the laboratory during the day of sample collection are acceptable if they have been placed on ice in an insulated cooler but have not yet reached a temperature of 4°C. Soil and sediment samples do not require additional preservation. As noted above, sample containers for

aqueous samples will be obtained from the laboratory containing the appropriate preservatives.

3.4 Sample Shipping

Sample containers shall be wrapped in protective packing material (if appropriate). Samples will then be placed in a cooler with ice for shipment to the laboratory. The drain on the cooler shall be taped shut. Samples collected in glass containers will be packed in foam liners and/or bubble wrap to ensure that no breakage occurs during shipment. A temperature blank will be included in each cooler. Samples will be sent to the analytical laboratory via Federal Express or equivalent. Shipping receipts should be retained for documentation and sample tracking.

A completed chain-of-custody (COC) form for each cooler will be placed in a Ziploc® bag and taped to the inside of the cooler lid. Coolers will be wrapped with packing tape at two locations to secure lids. Signed and dated custody seals shall be placed on the outside of each cooler in two places in such a manner as to allow detection of tampering (i.e., the seals must be broken to open the cooler).

3.5 Holding Time Requirements

The holding time is specified as the maximum allowable time between sample collection and analysis and/or extraction, based on the analyte of interest, stability factors, and preservation methods. Allowable holding times for chemical analysis parameters are listed in the QAPP. Samples should be sent to the laboratory after collection in sufficient time to allow the laboratory to meet holding time requirements.

4.0 QUALITY CONTROL (QC) REQUIREMENTS

QC requirements relevant to analysis of environmental samples shall be followed during analytical activities to meet the quality objectives and criteria. The purpose of the QC program is to produce data of known and documented quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis.

4.1 QC Samples

A number of QC samples will be employed to assess various data quality parameters, such as representativeness of the environmental samples, the precision of sample collection and handling procedures, the thoroughness of the field equipment decontamination procedures, and the accuracy of laboratory analysis. Types of QC samples are discussed below.

4.1.1 Matrix Spike/Matrix Spike Duplicate

Matrix spike (MS) and matrix spike duplicate (MSD) samples are prepared by spiking additional aliquots of sample with known concentrations of all project target analytes.

The sample to be used for the MS/MSD analyses shall be designated on the chain of custody and additional sample volume shall be submitted, as necessary. The MS/MSD results are used to document the bias of a method due to sample matrix. Consequently, MSs and MSDs are not used to control the analytical process. Minimum numbers of MS and one MSD samples are indicated in the project specific QAPP, generally one for every 20 environmental samples of a given matrix. Alternately, a laboratory may prepare and analyze a MS sample and a laboratory duplicate sample as discussed below. Analysis of a MS/MSD or MS/LDS sample set to assess matrix effects on accuracy and precision is typically dependent on the analyte class (i.e., inorganic vs. organic) and the likelihood of detecting the target analyte.

4.1.2 Rinsate Blank

A rinsate blank is a sample of ASTM Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. ASTM Type II reagent grade water obtained from the laboratory is used to prepare the rinsate blank sample. Rinsate blanks are used to assess the effectiveness of equipment decontamination procedures used to prevent cross-contamination between sampling locations. The frequency of collection for rinsate blanks is indicated in the project specific QAPP, generally a minimum of 1 rinsate blank for every 20 environmental samples collected with a given type of sampling equipment, and only for sampling equipment which is decontaminated and reused to collect environmental samples. Rinsate blanks will be prepared in a manner identical to samples and shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site using the subject equipment and will use laboratory-grade, PFAS-free water for rinsate samples. Rinsate blanks are not necessary for disposable or dedicated sampling equipment.

4.1.3 Trip Blank

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

shall accompany each cooler containing samples for VOC analysis that is sent to the laboratory.

4.1.4 Field Duplicates

A field duplicate sample is a second, discrete sample volume collected at the same location as the original sample (homogenization is not performed between the original sample and the field duplicate). Aqueous field duplicate samples are collected from successive volumes from the same sample source and device (i.e., bailers). Sediment and soil field duplicates are collected in succession from the same sample source and device. Individual analytes for the primary and duplicate groundwater samples are to be collected in order (i.e., VOC primary then VOC duplicate, etc.) so that a long period of time does not pass between collection of each analyte. Field duplicate samples are collected using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned an identification number in the field such that they cannot be identified (blind duplicate) as field duplicate samples by laboratory personnel performing the analysis.

Field duplicate sample results are used to assess precision of the sample collection process and the heterogeneity of the medium sampled. The frequency of collection for field duplicates is indicated in the project specific QAPP, generally a minimum of one field duplicate sample from each group of 10 environmental samples of a given matrix. Specific locations for collection of field duplicate samples may be designated prior to the beginning of sample collection.

5.0 DOCUMENTATION AND TRACKING

5.1 Field Notes

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded with non-waterproof ink, such as ballpoint pens and Fine or Ultra-Fine Point Sharpie markers, in a permanently bound non-weatherproof field logbook with consecutively numbered pages and, if applicable, on field sampling data sheets.

The information in the field logbook will include the following as a minimum. Unless information is recorded on a field sample collection form and that form is cross referenced in the logbook entry. Additional information is included in the specific SOPs regarding the appropriate data sheets.

- Project name

- Location of sample
- Sampler's signature
- Date and time of sample collection
- Sample identification numbers and sample depth (if applicable)
- Description of samples (matrix sampled), composite or grab sample
- Description of QA/QC samples (if collected)
- Sample methods or reference to the appropriate SOP
- Field observations
- Decontamination information
- Calibration information
- Personnel present
- Method of shipment
- Any deviations from SOPs
- Any information pertaining to the sample that is not noted of the sample form.

If samples are held for an extended period of time (i.e., inadvertently missed Fed-Ex pick up), field personnel will document all sample handling and custody in the field logbook.

5.2 Chain-of-Custody Form

A record of each sample collected will be indicated on a COC form. Every sample in the coolers shall be covered by the COC form(s) accompanying the coolers. Coolers may contain a single COC covering only the samples in that cooler or may contain copies of the COC that covers all of the samples in all of the coolers. One cooler must contain the original COC. The COC form will provide an accurate written record which can be used to trace the custody of all samples from the time of collection through data analyses and reporting.

The following will be specified for each sample on the COC form as a minimum:

- Sample ID
- Sample date
- Sample time
- Requested analysis
- Number of containers
- Sampler's signature or initials
- Preservation technique
- Sample type (i.e., medium)

Also recorded on the COC is the signature of the person relinquishing custody, the date and time that custody was relinquished, the name and address of the laboratory, and the name and phone number of a contact person regarding the shipment.

A sample is considered in custody if it is:

1. In one's actual possession
2. In view, after being in physical possession
3. Locked so that no one can tamper with it, after having been in physical custody
4. In a secured area

The person responsible for custody of the sample prior to delivery of the samples to the laboratory will sign the COC form, retain the last copy of the three-part COC form, document the method of shipment, and send the original and the second copy of the COC form with the sample (taped in a Ziploc® bag to inner cooler lid). Upon receipt at the laboratory, the person receiving the samples will sign the COC form and return the second copy to the Project Manager or Quality Assurance Manager or specified designee. Copies of the COC forms and all custody documentation will be received and kept in the central files. The original COC forms will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory may dispose of the samples in an appropriate manner 60 to 90 days after data reporting. After sample disposal, a copy of the original COC will be sent to the Project Manager or Quality Assurance Manager or specified designee by the analytical laboratory to be incorporated into the central files.

Standard Operating Procedure No. 4
Sampling Equipment Decontamination
(PFAS Specific)

Revision:_____0.0_____

Reviewed:___11/01/21___

Reviewer:_____

David Conner, PG

SOP No. 4 – Sampling Equipment Decontamination (PFAS Specific)

1.0 OBJECTIVE

Decontamination is performed as a quality assurance measure and safety precaution. It helps prevent cross-contamination among samples and helps maintain a clean working environment for the safety of field personnel.

2.0 EQUIPMENT AND MATERIALS

All equipment and materials must be PFAS-free and have not come into contact with a material containing PFAS. The equipment and materials necessary that may be used to decontaminate sampling equipment include:

- Use of laboratory supplied PFAS-free deionized water is preferred for cleaning and decontamination
    - Commercially available deionized water may be used if water is verified to be PFAS-free
    - Municipal drinking water (i.e., potable water) may be used for cleaning or decontamination if the water is known to be PFAS-free
- Cleaning liquids such as soap or detergent solutions (Alconox®, Liquinox®, or Citranox®) and deionized water
- Polyethylene or polyvinyl chloride (PVC) cleaning brushes
- Cleaning containers, such as polypropylene buckets or tubs
- PFAS-free pump sprayers for dispensing rinse waters
- A high-pressure hot water sprayer for cleaning large equipment (i.e., drill rods)
- Polypropylene waste containers
- Health and safety equipment as outlined in the Site-Specific Health and Safety Plan

3.0 METHODOLOGY

Small, reusable equipment is decontaminated primarily by rinsing with PFAS-free liquids that include soap or detergent solutions, and deionized water. Steam cleaning may he used whenever visible contamination exists on large machinery or vehicles. Following decontamination, if the equipment is not to be reused immediately, it should be stored and protected from recontamination.

3.1 Pre-Sampling Decontamination Activities

1. Don the appropriate personal protective equipment, including powderless nitrile gloves, as specified in the Site-Specific Health and Safety Plan, and as required for the specific work area.
2. Assemble containers and equipment for decontamination.
3. Decontaminate new equipment or equipment not previously decontaminated before use. Disposable equipment, including polyethylene tubing and bailers, do not require decontamination prior to use.
4. Rinse equipment not previously decontaminated and appropriately protect from recontamination before the next use.

SOP No. 4 – Sampling Equipment Decontamination (PFAS Specific)

3.2 Decontaminating Sampling Equipment

1. Remove solid particles from the equipment or material by brushing and rinsing with laboratory supplied PFAS-free deionized water. This will remove gross contamination.
2. Wash equipment with a brush and a phosphate-free detergent solution (Alconox®, Liquinox®, or Citranox®).
3. Rinse equipment thoroughly with laboratory supplied PFAS-free deionized water.
4. Triple rinse the equipment with laboratory supplied PFAS-free deionized water.
5. Unless the equipment is going to be used immediately protect it from recontamination before the next use.

3.3 Decontaminating Large Equipment

Drilling equipment (i.e., rigs, drill rods, augers, rods, bits, casing, screen. etc.), downhole logging equipment, and other large pieces of field equipment may be high-pressure steam-cleaned before and after use. Steam cleaning will be performed at an appropriate decontamination area specified by the field supervisor. The decontamination area shall be capable of containing decontamination fluids and solids. The decontamination fluids shall be managed in accordance with SOP – IDW Management (PFAS Specific).

Additionally, the drilling subcontractor has the responsibility of making the drilling rig free of leaks (i.e., hydraulic fluid, oil, gas. etc.) that could contaminate the boreholes. PFAS-free anti-seize thread lubricant may be sparingly used on rod shoulders to ease rod breaking upon completion of a borehole. Rod joints should be wiped with a clean cotton cloth to minimize the amount of lubricant on the exterior of the rod.

4.0 Comments

Decontamination is critical for maintaining the integrity of the sampling program. Check equipment carefully prior to sampling, and if there is any doubt about the effectiveness of the decontamination, repeat the decontamination process as an extra precaution. Decontamination fluids will be containerized and disposed of following the procedures provided SOP – IDW Management (PFAS Specific). Decontamination procedures shall be documented in the field logbook.

Standard Operating Procedure No. 5

Soil Sampling for Chemical Analysis (PFAS Specific)

Revision:_____0.0_____

Reviewed:___11/01/21___

Reviewer:_____

David Conner, PG

SOP No. 5 – Soil Sampling for Chemical Analysis (PFAS Specific)

1.0 OBJECTIVE

The purpose of this document is to define the standard operating procedure (SOP) for collecting soil samples during Per- and Polyfluoroalkyl Substances (PFAS) investigations. This procedure describes equipment and field procedures necessary to collect soil samples using a hand auger. Soil samples will be collected for field screening and chemical analysis to help characterize the source areas and to determine the nature and extent of contamination in soil. Soil samples will be collected with a hand auger to depth specified in the Quality Assurance Project Plan (QAPP).

2.0 EQUIPMENT AND MATERIALS

All equipment and materials must be PFAS-free and have not come into contact with a material containing PFAS. The equipment and materials necessary that may be used for soil sampling with chemical analysis include:

- Hand auger with stainless-steel cutting head (i.e., bit) cylinder (i.e., bucket), and bail that attaches to extension rods or sections, and cross handle used to rotate the hand auguer.
- Appropriate number and types of sample containers (made of PFAS-free High-Density Polyethlyene (HDPE), glass, or polypropylene with Teflon®-free caps)
- Precleaned stainless steel sampling utensils (See SOP – Sampling Equipment Decontamination [PFAS Specific])
- Sample coolers and ice (chemical ice packs are prohibited).
- Appropriate field documentation forms (non-waterproof, non-recycled loose paper) and labels (non-waterproof) and a non-waterproof ink pen (i.e., ballpoint pen); Sharpie® markers are prohibited.
- Sampling equipment (i.e., hand auger).
- Decontamination equipment.
- Polypropylene waste containers.
- Health and safety equipment, as specified in the Health and Safety Plan.

3.0 METHODOLOGY

Soil samples collected by hand auger method will be collected as follows:

3.0.1 Decontaminate the hand auger prior to sampling activities in accordance with the procedures specified in SOP – Sampling Equipment Decontamination (PFAS Specific).

3.0.2 Hand auger holes are advanced one bucket at a time until the sample depth is achieved. Place the hand auger on top of the ground surface with bit facing downward and rotate

clockwise while applying downward pressure. Remove the hand auger once the bucket has been filled with soil. Empty the bucket, place the hand auger bucket into the borehole, and continue advancing the bucket to the desired sample depth.

3.0.3 The upper portion of soil in the sampler potentially represents material that has fallen from above or has been scraped from the sides of the auger hole; therefore, this portion is not representative of the sampling interval and should be discarded.

3.0.4 Samples should be minimally handled and placed directly into the sample container when possible. If compositing, mixing, or homogenization of the sample is desired, it should be preferably done at the laboratory so that a representative subsample will be analyzed.

3.0.5 Samples for PFAS will be collected first.

3.0.6 Samples for volatile organic compound (VOC) analysis will be collected second to minimize the potential for volatilization. A sufficient amount of soil will be collected and transferred directly to VOC sample containers using a stainless-steel utensil. The sample will be packed to completely fill the container and reduce the amount of headspace, which will minimize the loss of volatile compounds. Non-Teflon® lined septum lids will be immediately secured on the sample containers.

3.0.7 Next the remaining sample will be screen with a photoionization detector (PID) and the reading will be recorded on the field log.

3.0.8 Collect samples for additional analysis next.

3.0.9 Complete the sample labels in accordance with SOP – Sample Handling and Management (PFAS Specific) and affix them to the container.

3.0.10 Place protective packing on the sample container (non-Teflon® lined bottle or caps in PFAS-free High-Density Polyethylene HDPE), glass, or polypropylene container.

3.0.11 Decontaminate sampling equipment in accordance with SOP – Sampling Equipment Decontamination (PFAS Specific).

3.0.12 Contain and handle all soil removed from borehole in accordance with the Investigation Derived Waste (IDW) management procedures specified in SOP – IDW Management (PFAS Specific).

4.0 COMMENTS

Soil samples for PFAS analysis will be conducted first and not be stored in the same coolers as samples for VOC analysis. Samples for VOC analysis will be collected before any other logging or other sample handling is conducted. Due to the nature of VOCs, it is critical to collect these samples as quickly as possible and to immediately place them in a cooler with ice. VOC soil samples are collected via either TerraCore® or EnCore® sampling kits. Refer to the project specific work plan for the specific sampling protocol. The TerraCore® or EnCore® samplers for VOC soil samples should be prechilled in a cooler with ice prior to filling. This practice will further reduce the potential for volatilization during sample collection. It may be necessary to advance an additional boring for the purpose of

lithologic logging.

Standard Operating Procedure No. 6

IDW Management

(PFAS Specific)

Revision:_____1.0_____

Reviewed:___10/12/22___

Reviewer:_____

David Conner, PG

SOP No. 6 – IDW Management (PFAS Specific)

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) provides technical guidance and methods that will be used for the handling, management, and disposal of investigation derived waste (IDW) encountered or generated during environmental investigation activities. This SOP gives descriptions of equipment, field development procedures, field data collection, and personnel responsibilities.

2.0 EQUIPMENT AND MATERIALS

All equipment and materials must be PFAS-free and have not come into contact with a material containing PFAS. The equipment and materials necessary that may be used for IDW management:

- Personal protective equipment (PPE) as outlined in the Accident Prevention Plan (APP)/ Site Safety and Health Plan (SSHP)
- Decontamination equipment and supplies (i.e., wash/rinse tubs, polyethylene, or polyvinyl chloride (PVC) brushes, Alconox®, thin HDPE plastic sheeting, untreated paper towels, wipes, garden-type water sprayers, potable water, distilled water and/or deionized water)
- Department of Transportation (DOT)-rated 55-gallon drums or other approved containers for containing soil cuttings, decontamination water, and formation water
- Drum/bung wrench and drum funnel
- Heavy equipment forklift or vehicle with drum grappler (as necessary)
- Laboratory-supplied sample containers (PFAS-free High-Density Polyethylene (HDPE), glass, or polypropylene with Teflon®-free caps)
- Photoionization detector (PID)
- Wood pallets (as necessary)
- Stainless steel shovels
- Polyethylene tanks (as necessary)
- Non-waterproof field notebook and non-waterproof pens such as ballpoint pens

3.0 PROCEDURES

It is anticipated that both non-liquid and liquid IDW will be generated or encountered during field activities. IDW generated during the field investigation is expected to include:

- Soil cuttings and other soil wastes generated during sampling
- Well purge water

- Wash and rinse waste from decontamination activities
- Used PPE and other non-soil solid wastes

3.1 SOIL IDW

- Soil cuttings generated during soil sampling will be initially assessed in the field to determine if the potential exists for contamination with site related COCs. If the soils are generated outside of the original limits of the site (e.g., outside the fence line), they will be screened for volatile contaminants with a PID meter. If no readings are above background level, the soil will be spread on the ground at the point of generation. If PID readings indicate potential contamination, the soil will be placed into DOT-rated 55-gallon drums, or appropriately sized containers at the point of generation pending further analysis. Soil cuttings generated inside the original limits of the site will be containerized pending further analysis.
- Mixing of the cuttings from several borings or sampling locations is permissible in order to fill the drums.
- When drums or containers are full, or daily activities are completed, the drum lids and rings will be fastened. Full drums or containers will be transported to the designated IDW accumulation area on a regular basis to avoid accumulation of drums or containers at individual investigation sites for extended periods of time.
- Drums will be stored on pallets at the designated IDW accumulation area. Drums will be segregated to separate soil from liquid IDW.
- Drums will be sealed and labeled with permanent markings (using paint pens or drum labels) with the following information:
    - Source: the boring(s), well, or site identification number
    - Matrix (e.g., soil, water)
    - Sample interval
    - Fill date
    - Drum identification number
    - Contractor
    - Point of contact with phone number
    - Labeled "Contents Pending Analysis"
- If large volumes of soil IDW will be generated, soil IDW will be transferred from the drums into roll-off bins (lined and covered) located within the designated IDW accumulation area.

- If no associated investigation sample results exist, a composite soil sample will be collected from the soil IDW drums by collecting a drive or hand auger sample from each of the drums. The sample material from all of the drums will be composited into a single sample that will be used to characterize and dispose of the IDW.

3.2 LIQUID IDW

- Wastewater generated from groundwater sampling activities and equipment decontamination will be contained in the sampling vehicle's onboard 400-gallon purge/decontamination water tank.
- The wastewater will be transported to NASA's OU-1 source area treatment system.
- The wastewater will be transferred from the onboard tank to the treatment system for processing on a daily basis.
- No liquid IDW will be placed in drums or tanks for long-term storage.

3.3 PPE AND DISPOSABLE INVESTIGATION EQUIPMENT

- The plan for managing used PPE and other non-soil solid waste generated during field activities (e.g., sample handling) will be segregated separately and placed into dedicated heavy duty plastic bags or containers (e.g., drums)
- Potentially contaminated PPE or disposable investigation equipment will be decontaminated prior to placement in the plastic bags or containers, if warranted.
- Decontamination procedures consist of brushing off, or using small amounts of water to scrub off, gross potential contamination.

3.4 DISPOSAL OF IDW

IDW will be disposed of in accordance with federal, state, and local regulations.

For solid IDW, if PFAS are detected above the US EPA risk-based screening level (RSL) for soil of 0.126 parts per million (ppm), determined by calculation using the US EPA oral reference dose for PFOS/PFOA, then the waste must be disposed of using incineration or by solidification and landfill via U.S. Ecology. While treatment of liquid IDW may be commercially feasible and cheaper than incineration, on-site treatment options for soils and other solids are currently limited.

If other contaminants of concern (COCs) exceeding regulatory standards are identified in the waste, then the waste will be managed to address the regulated COC according to applicable legal requirements.

4.0 DOCUMENTATION

Project staff are responsible for thoroughly documenting IDW handling and disposal activities. Personnel will be responsible for documenting the collection, transportation,

labeling (if applicable), and staging or disposition of IDW. A Waste Inventory Tracking Form on loose non-waterproof and non-recycled paper is to be completed if necessary. Documentation should include the following:

- Project Name
- Names of personnel
- Site location
- Type of activities
- Date waste generated
- Boring, well, or site number(s)
- Matrix
- Type of container(s)
- Estimated volume
- Disposition of contents
- Comments (field evidence of contamination (e.g., PID reading, odors)
- Any variance to procedures described in this SOP

INSTRUCTION MANUAL

MULTI-PARAMETER WATER QUALITY CHECKER

U-50 series

CODE:GZ0000480566A

HORIBA
Process & Environmental

Preface

This manual describes the operation of the Multi-parameter Water Quality Checker, U-50 Series.
Be sure to read this manual before using the product to ensure proper and safe operation of the product. Also safely store the manual so it is readily available whenever necessary.

Product specifications and appearance, as well as the contents of this manual are subject to change without notice.

Warranty and responsibility

HORIBA Advanced Techno Co., Ltd. warrants that the Product shall be free from defects in material and workmanship and agrees to repair or replace free of charge, at option of HORIBA Advanced Techno Co., Ltd., any malfunctioned or damaged Product attributable to responsibility of HORIBA Advanced Techno Co., Ltd. for a period of one (1) year from the delivery unless otherwise agreed with a written agreement. In any one of the following cases, none of the warranties set forth herein shall be extended;

- Any malfunction or damage attributable to improper operation
- Any malfunction attributable to repair or modification by any person not authorized by HORIBA Advanced Techno Co., Ltd.
- Any malfunction or damage attributable to the use in an environment not specified in this manual
- Any malfunction or damage attributable to violation of the instructions in this manual or operations in the manner not specified in this manual
- Any malfunction or damage attributable to any cause or causes beyond the reasonable control of HORIBA Advanced Techno Co., Ltd. such as natural disasters
- Any deterioration in appearance attributable to corrosion, rust, and so on
- Replacement of consumables

HORIBA Advanced Techno Co., Ltd. SHALL NOT BE LIABLE FOR ANY DAMAGES RESULTING FROM ANY MALFUNCTIONS OF THE PRODUCT, ANY ERASURE OF DATA, OR ANY OTHER USES OF THE PRODUCT.

Trademarks

Company names and brand names are either registered trademarks or trademarks of the respective companies. (R), (TM) symbols may be omitted in this manual.

Regulations

EU regulations

Conformable standards

This equipment conforms to the following standards:

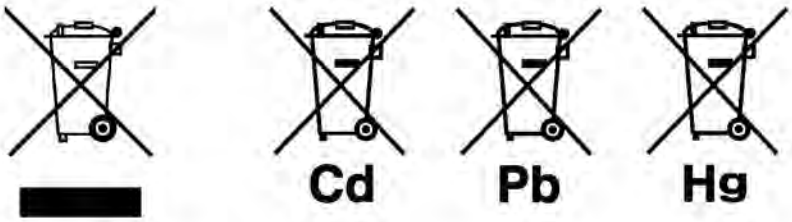
CE

EMC: EN61326-1
Class B, Portable test and measurement equipment
RoHS: EN50581
9. Industrial monitoring and control instruments

Warning: This product is not intended for use in industrial environments. In an industrial environment, electromagnetic environmental effects may cause the incorrect performance of the product in which case the user may be required to take adequate measures.

Information on disposal of electrical and electronic equipment and disposal of batteries and accumulators

The crossed out wheeled bin symbol with underbar shown on the product or accompanying documents indicates the product requires appropriate treatment, collection and recycle for waste electrical and electronic equipment (WEEE) under the Directive 2012/19/EU, and/or waste batteries and accumulators under the Directive 2006/66/EC in the European Union.
The symbol might be put with one of the chemical symbols below. In this case, it satisfies the requirements of the Directive 2006/66/EC for the object chemical.
This product should not be disposed of as unsorted household waste.
Your correct disposal of WEEE, waste batteries and accumulators will contribute to reducing wasteful consumption of natural resources, and protecting human health and the environment from potential negative effects caused by hazardous substance in products.
Contact your supplier for information on applicable disposal methods.



Authorised Representative in EU

HORIBA Europe GmbH
Hans-Mess-Str.6, D-61440 Oberursel, Germany

FCC rules

Any changes or modifications not expressly approved by the party responsible for compliance shall void the user's authority to operate the equipment.

■ Warning

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications.

Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Korea certification

■ B급 기기 (가정용 방송통신기자재)

이 기기는 가정용(B 급) 전자파적합기기로서 주로 가정에서 사용하는 것을 목적으로 하며, 모든 지역에서 사용할 수 있습니다.

Taiwan battery recycling mark

廢電池請回收

China regulation

本标记适用在中华人民共和国销售电器电子产品，标记中央的数字表示环境保护使用期限的年数。(不是表示产品质量保证期间。) 只要遵守这个产品有关的安全和使用注意事项，从制造日开始算起在这个年限内，不会给环境污染、人体和财产带来严重的影响。请不要随意废弃本电器电子产品。

This marking is applied to electric and electronic products sold in the People's Republic of China. The figure at the center of the marking indicates the environmental protection use period in years. (It does not indicate a product guarantee period.) It guarantees that the product will not cause environment pollution nor serious influence on human body and property within the period of the indicated years which is counted from the date of manufacture as far as the safety and usage precautions for the product are observed. Do not throw away this product without any good reason.

本マークは、中華人民共和国で販売される電気電子製品に適用され、マークの中央の数字は環境保護使用期限の年数を意味します(製品の品質保証期間を示すものではありません)。この製品に関する安全や使用上の注意をお守り頂く限り、製造日から起算するこの年限内では、環境汚染や人体や財産に深刻な影響を及ぼすことはありません。本製品をみだりに廃棄しないでください。

产品中有害物质的名称及含量

Name and amount of hazardous substance used in a product

部件名称 Unit name		有害物质 Hazardous substance					
		铅 Lead (Pb)	汞 Mercury (Hg)	镉 Cadmium (Cd)	六价铬 Hexavalent chromium (Cr (VI))	多溴联苯 Polybromo-biphenyl (PBB)	多溴二苯醚 Polybromo-diphenyl ether (PBDE)
控制器	Controller	×	○	○	○	○	○
探针	Probe	×	○	○	○	○	○
配件	Accessories	○	○	×	○	○	○
包装	Package	○	○	○	○	○	○

本表格依据 SJ/T 11364 的规定编制。

This form is prepared in accordance with SJ/T 11364.

○: 表示该有害物质在该部件所有均质材料中的含量均在 GB/T 26572 规定的限量要求以下。

Denotes that the amount of the hazardous substance contained in all of the homogeneous materials used in the component is below the limit on the acceptable amount stipulated in the GB/T 26572.

×: 表示该有害物质至少在该部件的某一均质材料中的含量超出 GB/T 26572 规定的限量要求。

Denotes that the amount of the hazardous substance contained in any of the homogeneous materials used in the component is above the limit on the acceptable amount stipulated in the GB/T 26572.

For Your Safety

Hazard classification and warning symbols

Warning messages are described in the following manner. Read the messages and follow the instructions carefully.

■ Hazard classification

DANGER	This indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury. This is to be limited to the most extreme situations.
WARNING	This indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.
CAUTION	This indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert against unsafe practices.

■ Warning symbols

Description of what should be done, or what should be followed

Description of what should never be done, or what is prohibited

Safety label list

The following table lists the labels attached on the product.
For more details, refer to the descriptions of the individual labels provided later in this manual.

Hazard level	Hazard type	Label ID number
WARNING	FALLING	3200647816

The following pages describe the safety information in German, French, Italian, Swedish, Spanish, Polish, Dutch, and Japanese (8 languages), and the safety labels affixed to the product (9 languages, including the above and English).

[DEU] Sicherheitsinformation

Lesen Sie vor der Verwendung des Produkts unbedingt diese Anleitung, um den ordnungsgemäßen und sicheren Betrieb des Produkts zu gewährleisten. Bewahren Sie die Anleitung sicher auf, damit sie bei Bedarf jederzeit zur Hand ist.

Die technischen Daten und das Erscheinungsbild des Produkts sowie der Inhalt dieser Anleitung können unangekündigt geändert werden.

Installationsumgebung

Dieses Produkt ist nicht zum Gebrauch in industriellen Umgebungen, wie in EN61326-1 definiert, vorgesehen.
In einer industriellen Umgebung können die elektromagnetischen Störungen eventuell zu Produktfehlfunktionen führen. Um dieses Produkt unter solchen Umständen verwenden zu können, muss der Benutzer ggf. angemessene Maßnahmen ergreifen.

Gefahrenklassifikation und Warnsymbole

Warnmeldungen werden wie folgt beschrieben. Lesen Sie die Meldungen und befolgen Sie die Anleitungen sorgfältig.

GEFAHR

Dies weist auf eine unmittelbar gefährliche Situation hin, die im Tod oder in schweren Verletzungen resultiert, falls sie nicht vermieden wird. Dies ist auf die extremsten Situationen zu begrenzen.

WARNUNG

Dies weist auf eine potentiell gefährliche Situation hin, die im Tod oder in schweren Verletzungen resultieren könnte, falls sie nicht vermieden wird.

VORSICHT

Dies weist auf eine potentiell gefährliche Situation hin, die in leichten oder mäßigen Verletzungen resultieren könnte, falls sie nicht vermieden wird. Sie kann auch zur Warnung vor unsicheren Praktiken verwendet werden.

Liste der Sicherheitsschilder

Die folgende Tabelle listet die am Produkt befestigten Schilder auf.
Weitere Details entnehmen Sie den Beschreibungen der individuellen Schilder weiter hinten in dieser Anleitung.

Gefahrenstufe	Gefahrentyp	Schild-ID-Nummer
WARNUNG	HINFALLEN	3200647816

[FRA] Informations de sécurité

Veillez à lire le présent manuel avant d'utiliser le produit de manière à garantir son utilisation correcte et sûre. De même, rangez le manuel dans un lieu sûr de manière à pouvoir vous y reporter lorsque cela est nécessaire.

Les spécifications et l'aspect du produit, ainsi que le contenu du présent manuel peuvent être modifiés sans notification préalable.

■ Environnement d'installation

Ce produit n'est pas destinés à une utilisation dans des environnements industriels, tels que définis dans la norme EN61326-1.
Dans un environnement industriel, les interférences électromagnétiques peuvent entraîner un dysfonctionnement du produit. Pour utiliser le produit dans ce type d'environnements, l'utilisateur peut avoir à prendre des mesures appropriées.

■ Classification des risques et symboles d'avertissement

Les messages d'avertissement sont décrits comme suit. Lisez les messages et suivez attentivement les instructions.

DANGER

Cela indique une situation dangereuse imminente qui, si elle n'est pas évitée, entraînera la mort ou des blessures graves. Cela fait uniquement référence aux situations les plus extrêmes.

AVERTISSEMENT

Cela indique une situation potentiellement dangereuse qui, si elle n'est pas évitée, peut entraîner la mort ou des blessures graves.

ATTENTION

Cela indique une situation potentiellement dangereuse qui, si elle n'est pas évitée, peut entraîner des blessures mineures ou modérées. Cela peut également être utilisé pour signaler des pratiques dangereuses.

■ Liste des étiquettes de sécurité

Le tableau suivant répertorie les étiquettes fixées sur le produit.
Pour plus de détails, reportez-vous aux descriptions des étiquettes individuelles fournies ultérieurement dans ce manuel.

Niveau de risque	Type de risque	Numéro d'identification de l'étiquette
AVERTISSEMENT	CHUTE	3200647816

[ITA] Informazioni sulla sicurezza

Leggere attentamente questo manuale prima di utilizzare il prodotto al fine di utilizzarlo in modo sicuro e adeguato. Inoltre, conservare in un luogo sicuro il manuale per poterlo consultare se necessario.

Le specifiche e l'aspetto del prodotto, nonché i contenuti di questo manuale, sono soggetti a modifica senza preavviso.

Ambiente di installazione

Questo prodotto non è stati progettati per essere utilizzati in ambienti industriali, secondo la norma EN61326-1.
In un ambiente industriale, le interferenze elettromagnetiche potrebbero causare un malfunzionamento del prodotto. Per utilizzare il prodotto in tali ambienti, all'utente potrebbe essere richiesto di adottare le contromisure necessarie.

Categoria di pericolo e simboli di avvertenza

I messaggi di avvertenza sono descritti come segue. Leggere i messaggi e seguire con attenzione le istruzioni.

PERICOLO — Indica un pericolo immediato che, se non evitato, può causare il decesso o lesioni gravi. Limitato alle situazioni più estreme.

AVVERTENZA — Indica una situazione potenzialmente pericolosa che, se non evitata, potrebbe causare il decesso o lesioni gravi.

ATTENZIONE — Indica una situazione potenzialmente pericolosa che, se non evitata, potrebbe causare lesioni di media e piccola entità. Potrebbe essere usato anche per informare circa pratiche non sicure.

Elenco etichette di sicurezza

La tabella seguente elenca le etichette applicate sul prodotto.
Per maggiori informazioni, consultare la descrizione di ciascuna etichetta più avanti nel presente manuale.

Livello di pericolo	Tipo di pericolo	Numero ID etichetta
AVVERTENZA	CADUTA	3200647816

[SWE] Säkerhetsinformation

Se till att du läser denna handbok innan du börjar använda produkten för en korrekt och säker användning av den. Spara sedan handboken på en säker och lättåtkomlig plats så att du kan konsultera den när så behövs.

Produktspecifikationerna och utseendet, samt även innehållet i denna handbok, kan komma att ändras utan föregående meddelande därom.

■ Installationsmiljö

Detta produkten är ej avsedda för användning i industriella miljöer enligt riktlinjerna i EN61326-1.
Om den används i industrimiljöer kan de elektromagnetiska störningarna orsaka tekniska fel hos produkten. Om produkten ska användas i sådana miljöer kan användaren behöva vidta lämpliga åtgärder för att lösa dessa problem.

■ Riskklassificering och varningssymboler

Varningsmeddelandena beskrivs på följande sätt. Läs meddelandena och följ anvisningarna noggrant.

FARA

Denna varnar för en omedelbart risksituation som kan orsaka allvarliga personskador eller dödsfall om den inte följs. Detta omfattar endast de mest extrema situationerna.

VARNING

Denna varnar för en potentiell risksituation som kan orsaka allvarliga personskador eller dödsfall om den inte följs.

OBSERVER

Denna varnar för en potentiell risksituation som kan orsaka mindre person- eller materialskador om den inte följs. Den kan även användas för att indikera olämplig användning.

■ Lista över säkerhetsetiketter

I följande tabell listas de etiketter som sitter fastsatta på produkten.
Läs beskrivningarna för varje enskild etikett som finns längre fram i handboken för mer information.

Risknivå	Risktyp	Etikett-ID-nummer
VARNING	FALLANDE	3200647816

[SPA] Información de seguridad

Asegúrese de leer este manual antes de utilizar el producto para garantizar un uso correcto y seguro del mismo. Asimismo, guarde de forma segura el manual para que esté disponible siempre que sea necesario.

El aspecto y las especificaciones del producto, así como el contenido de este manual, están sujetos a cambios sin previo aviso.

■ Entorno de instalación

Este producto está diseñado para su uso en entornos industriales, tal y como se define en EN61326-1.
En un entorno industrial, las interferencias electromagnéticas pueden provocar un funcionamiento incorrecto del producto. Para usar el producto en tales entornos, el usuario debe tomar las medidas adecuadas.

■ Clasificación de peligrosidad y símbolos de advertencia

Los mensajes de advertencia se describen de la siguiente manera. Lea los mensajes y siga las instrucciones atentamente.

⚠ PELIGRO

Esto indica una situación de peligro inminente que, si no se evita, tendrá como resultado la muerte o lesiones graves. Esto se debe limitar a las situaciones más extremas.

⚠ ADVERTENCIA

Esto indica una posible situación de peligro que, si no se evita, podría tener como resultado la muerte o lesiones graves.

⚠ ATENCIÓN

Esto indica una posible situación de peligro que, si no se evita, puede tener como resultado lesiones leves o moderadas. También se puede usar para alertar de prácticas no seguras.

■ Lista de etiquetas de seguridad

En la siguiente tabla se muestran las etiquetas adheridas al producto.
Para obtener más información, consulte las descripciones de las etiquetas individuales que se proporcionan más adelante en este manual.

Nivel de riesgo	Tipo de riesgo	Número de ID de etiqueta
ADVERTENCIA	CAÍDA	3200647816

[POL] Informacje dotyczące bezpieczeństwa

Przed przystąpieniem do użytkowania tego produktu należy dokładnie zapoznać się z niniejszą instrukcją, aby zapewniona była prawidłowa i bezpieczna eksploatacja produktu. Instrukcję przechowywać w bezpiecznym miejscu, aby w razie potrzeby była zawsze dostępna.

Specyfikacja i wygląd produktów oraz treść niniejszej instrukcji może ulec zmianie bez wcześniejszego powiadomienia.

■ Środowisko instalacji

Ten produkt nie są przeznaczone do użytkowania w środowisku przemysłowym, zgodnie z definicją określoną w normie EN61326-1.
W środowisku przemysłowym zakłócenia elektromagnetyczne mogą powodować nieprawidłowe działanie produktów. Możliwe, że aby użytkować produkt w takich środowiskach, użytkownik będzie musiał podjąć stosowne środki zaradcze.

■ Klasyfikacja zagrożeń i symbole ostrzegawcze

Ostrzeżenia są opisane w następujący sposób. Należy zapoznać się z ostrzeżeniami i ściśle przestrzegać instrukcji.

NEBEZPIECZEŃSTWO — Oznacza bezpośrednio niebezpieczną sytuację, która — jeśli do niej dojdzie — spowoduje zgon lub poważne obrażenia ciała. To ostrzeżenie dotyczy najbardziej skrajnych sytuacji.

OSTRZEŻENIE — Oznacza potencjalnie niebezpieczną sytuację, która — jeśli do niej dojdzie — może spowodować zgon lub poważne obrażenia ciała.

PRZESTROGA — Oznacza potencjalnie niebezpieczną sytuację, która — jeśli do niej dojdzie — może spowodować niewielkie lub umiarkowane obrażenia ciała. Ten rodzaj ostrzeżenia może także być używany do ostrzegania przed niebezpiecznymi sposobami postępowania.

■ Lista etykiet bezpieczeństwa

W poniższej tabeli wymieniono etykiety umieszczone na produkcie.
Bardziej szczegółowe informacje można znaleźć w opisach poszczególnych etykiet, które znajdują się w dalszej części niniejszej publikacji.

Poziom zagrożenia	Typ zagrożenia	Numer identyfikacyjny etykiety
OSTRZEŻENIE	UPADEK	3200647816

[NLD] Veiligheidsinformatie

Lees deze handleiding voordat u dit product gebruikt zodat u het op de juiste manier en veilig kunt gebruiken. Bewaar de handleiding goed zodat u hem wanneer nodig kunt raadplegen.

De specificaties en het uiterlijk van het product en de inhoud van deze handleiding kunnen zonder voorafgaande kennisgeving worden gewijzigd.

Installatieomgeving

Dit product is niet bedoeld voor gebruik in een industriële omgeving zoals gedefinieerd in EN 61326-1.
In een industriële omgeving kan de elektromagnetische interferentie de werking van dit product storen. Voor gebruik van het product in een dergelijke omgeving moet de gebruiker mogelijk maatregelen treffen om de storing te verhelpen.

Indeling naar gevarencategorie en waarschuwingssymbolen

De waarschuwingen hebben de volgende betekenis. Lees de uitleg en volg de instructies aandachtig.

GEVAAR

Dit wijst op een onmiddellijk gevaarlijke situatie die zal leiden tot dodelijk of ernstig letsel als die niet wordt vermeden. Dit wordt alleen in de meest extreme gevallen gebruikt.

WAARSCHUWING

Dit wijst op een mogelijk gevaarlijke situatie die kan leiden tot dodelijk of ernstig letsel als die niet wordt vermeden.

VOORZICHTIG

Dit wijst op een mogelijk gevaarlijke situatie die kan leiden tot klein of matig letsel als die niet wordt vermeden. Dit kan ook gebruikt worden als waarschuwing tegen onveilig gebruik.

Lijst van veiligheidslabels

In de volgende tabel worden de labels vermeld die op het product zijn aangebracht.
Raadpleeg voor meer details de beschrijving van de afzonderlijke labels verder in deze handleiding.

Risiconiveau	Risicotype	ID-nummer van label
WAARSCHUWING	VALLEN	3200647816

[JPN] 安全情報

ご使用になる前に、本書を必ずお読みください。お読みになった後は必要なときにすぐに取り出せるように大切に保管してください。
ご使用の際、安全に関してお気付きの点がありましたら、弊社にご連絡ください。
製品の仕様・外観は、改良のため予告なく変更することがあります。
また、本書に記載されている内容も予告なく変更される場合があります。あらかじめご了承ください。

■ 設置環境

本製品は、EN61326-1で定義される工業環境で使用することを想定した製品ではありません。
工業環境においては、電磁妨害の影響を受ける可能性があり、その場合には使用者が適切な対策を講ずることが必要となることがあります。

■ 警告の種類と表示方法

本書および製品では、以下のような警告表示をしています。内容をよく理解して、正しく安全にご使用ください。

危　険　取り扱いを誤った場合、使用者が死亡または重傷を負うことがあり、かつその切迫の度合いが高いもの

警　告　取り扱いを誤った場合、使用者が死亡または重傷を負う可能性が想定されるもの

注　意　取り扱いを誤った場合、使用者が傷害を負うことが想定されるか、または物的損害の発生が想定されるもの

■ 安全ラベル一覧

製品には以下の安全ラベルが貼り付けられています。
安全ラベルの内容については、後述の各ラベルの説明を参照してください。

危険レベル	危険の種類	ラベル識別番号
警告	落下	3200647816

3200647816

[ENG]	FALLING Do not attach the hook to the body. The probe may be caught by a strong current and you may fall into the water. In addition, if the distance to the water surface is large or the flow is fast, attach the hook to a place where it can be fastened firmly.
WARNING	
FALLING	
[DEU]	HINFALLEN Bringen Sie den Haken nicht am Körper an. Die Sonde kann von einem starken Strom erfasst werden und Sie können in das Wasser fallen. Wenn außerdem der Abstand zur Wasseroberfläche groß ist oder die Strömung schnell ist, bringen Sie den Haken an einem Ort an, wo er sicher befestigt werden kann.
WARNUNG	
HINFALLEN	
[FRA]	CHUTE Ne pas fixer le crochet au corps. La sonde peut être prise dans un fort courant et vous pouvez tomber à l'eau. En outre, si la distance jusqu'à la surface de l'eau est grande ou que le courant est rapide, fixer le crochet à un endroit où il peut être fixé fermement.
AVERTISSEMENT	
CHUTE	
[ITA]	CADUTA Non fissare il gancio al corpo. La sonda può essere catturata da una forte corrente e si può cadere in acqua. Inoltre, se la distanza dalla superficie dell'acqua è grande o il flusso è veloce, ancorare il gancio in un luogo in cui può essere fissata saldamente.
AVVERTENZA	
CADUTA	

FALLANDE Fäst inte kroken i kroppen. Sonden kan dras med av en stark ström och du kan falla i vattnet. Fäst dessutom kroken på en plats där den kan fästas ordentligt om avståndet till vattenytan är stort eller flödet är stritt.	[SWE] VARNING FALLANDE
CAÍDA No coloque el gancho en el cuerpo. La sonda podría quedar atrapada en una corriente fuerte y podría caerse al agua. Además, si la distancia a la superficie del agua es grande o si la corriente es rápida, coloque el gancho en un lugar donde quede sujeto firmemente.	[SPA] ADVERTENCIA CAÍDA
UPADEK Nie mocować haka do ciała. Sonda może zostać pochwycona przez silny prąd i spowodować upadek użytkownika do wody. Ponadto, jeśli odległość do powierzchni wody jest duża lub przepływ jest szybki, zaczepić hak w miejscu, gdzie może zostać solidnie zamocowany.	[POL] OSTRZEŻENIE UPADEK
VALLEN Verbind de haak niet met het lichaam. De sonde kan worden opgevangen door een sterke stroming en u kunt in het water vallen. Daarnaast, als de afstand tot het wateroppervlak groot is of de stroom snel, bevestig dan de haak aan een plaats waar deze stevig kan worden vastgezet.	[NLD] WAARSCHUWING VALLEN
落下注意 フックを人体に取り付けないでください。 流れに巻き込まれて落下するおそれがあります。 また、水面までの落差が大きい場合や流速が速い場合は、しっかり固定できる場所に取り付けてください。	[JPN] 警告 落下

Safety precautions

This section provides precautions for using the product safely and correctly and to prevent injury and damage. The terms of DANGER, WARNING, and CAUTION indicate the degree of imminency and hazardous situation. Read the precautions carefully as it contains important safety messages.

⚠ WARNING
Do not disassemble or modify the meter. May cause overheating or fire, resulting in accidents.

⚠ CAUTION
The pH and ORP sensors are made of glass. Handle them carefully to avoid breakage.
Do not ingest the DO, pH or ORP standard solutions. If it comes into contact with the eyes, rinse thoroughly with water. If swallowed, consult a physician.
Keep away from water when using USB communication. Improper use may result in fire or damage.

Product Handling Information

Operational precautions

Use of the product in a manner not specified by the manufacturer may impair the protection provided by the product. And it may also reduce product performance.
Exercise the following precautions:

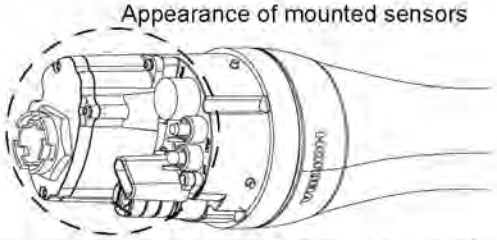
■ General

- Only use the product including accessories for their intended purpose.

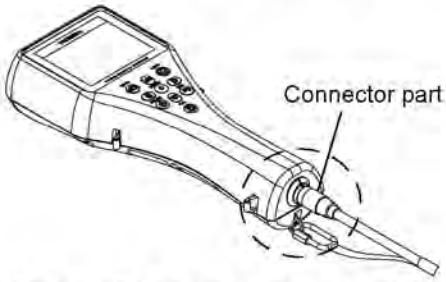
■ Sensor probe

- After seawater measurement, promptly wash the sensor probe thoroughly in water.
- Do not immerse the sensor probe in alcohol, organic solvent, strong acid, strong alkaline, and other similar solutions.
- Do not subject to strong shocks.
- Do not perform measurement in environments of magnetic fields. Measurement errors may result.
- The sensor probe is no longer waterproof when the sensors are not mounted.

Appearance of mounted sensors



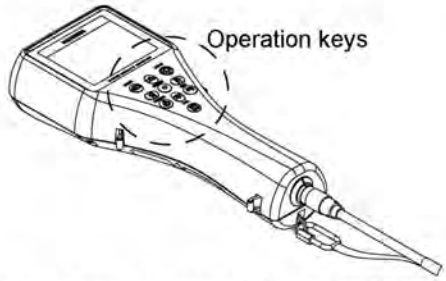
- Does not support measurement of samples containing fluorine.
- Do not mistake the combination of sensor probe and turbidity sensor.
  To disconnect the sensor cable or interface cable, pull them out with holding the connector part. Do not pull the cable part; it may cause breakage.



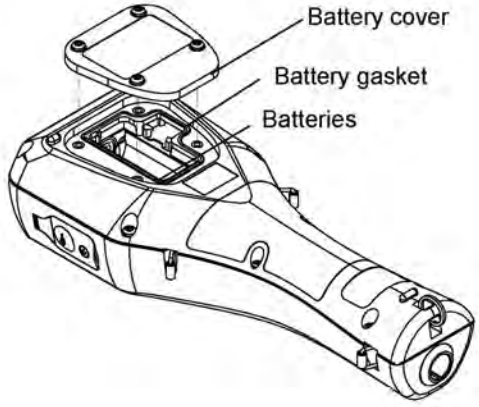
- Sensor probe of U-54 and U-54G cannot be used with the conventional control unit.
  Unusable control units are following version.
  P2000266001D, P2000266001C, P2000266001B, P2000266001A, P2000266001-
  To confirm the version, see "● Version" (page 37).

■ Control unit

- Do not subject to strong shocks.
- The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.



- The control unit is no longer waterproof when the USB cable is connected.
- When operating the control unit only, protect the connector with the connector cap provided.
- Remove the batteries when not using the control unit for an extended period of time. Battery fluid leakage may cause equipment failure.
- Do not wipe the control unit with organic solvents or powder polish. The surface may deteriorate or its printing may disappear. If the display becomes dirty, wipe the dirt off with a soft cloth soaked in neutral detergent.
- Do not turn the power OFF or disconnect the cable during calibration or setting. Memory data may be erased.
- To perform measurement, connect the sensor probe cable before turning the power ON.
- Do not remove the battery gasket or twist it.
- When opening the battery case, make sure that no foreign matter is attached to the battery gasket.
- Do not use any unspecified batteries; it may cause breakage.



Measurement

- Before lowering the sensor probe into the sample, do not connect the hook on the unit to a human body.
- The correct values are not displayed if the sensor is not mounted when the measurement display is activated.
- Perform DO measurement with no air bubbles in the internal solution.
- Do not reuse a membrane cap of DO sensor.
- Use the spanner for DO sensor provided to attach or remove the DO sensor.
- Avoid both U-53 and U-53G turbidity measurement in air, since the rubber wiper will quickly become damaged.
- Attach sensor guard to sensor probe in the measurement. When sensor probe is used in calibration cups (black and transparent) or flow cell, the sensor guard can be taken off.

Calibration

During atmosphere calibration for the DO electrode with DO salinity compensation set to automatic, values are compensated based on electrical conductivity, but calibration is performed normally.

Location of use and storage

- Storage temperature: –10°C to 60°C
- Relative humidity: Under 80% and free from condensation

Avoid using this product in the following sort of locations.

- Locations with a lot of dust or dirt
- Locations subject to strong vibrations
- Locations exposed to direct sunlight
- Locations where corrosive gas may be generated
- Close to air conditioning equipment
- Locations exposed directly to the wind

Disposal of the product

When disposing of the product, follow the related laws and/or regulations of your country.

Manual Information

Description in this manual

Note

This interprets the necessary points for correct operation and notifies the important points for handling the product.

Reference

This indicates the part where to refer for information.

Tip

This indicates reference information.

Original language

This is the English translation of an original Japanese document.

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1 About this Unit

The U-50 Series Multi Water Quality Checker features an integrated control unit and sensors. It is capable of making a maximum of eleven simultaneous measurements for various parameters, and is perfect for use in the field. The U-50 Series is designed with on-site ease-of-use in mind, provides a wide variety of functions, and can be used for water quality measurements and inspections of river water, groundwater, and waste water.

2 Device Information

2.1 Measurement parameters

Parameters	Model						
	U-51	U-52	U-52G	U-53	U-53G	U-54	U-54G
pH (pH)	✓	✓	✓	✓	✓	✓	✓
pH (mV)	✓	✓	✓	✓	✓	✓	✓
Oxidation reduction potential (ORP)	✓	✓	✓	✓	✓	✓	✓
Dissolved oxygen (DO)	✓	✓	✓	✓	✓	✓	✓
Electrical conductivity (COND)	✓	✓	✓	✓	✓	✓	✓
Salinity (SAL) [expressed as electrical conductivity]	✓	✓	✓	✓	✓	✓	✓
Total dissolved solids (TDS) [expressed as electrical conductivity]	✓	✓	✓	✓	✓	✓	✓
Seawater specific gravity (SG) [expressed as electrical conductivity]	✓	✓	✓	✓	✓	✓	✓
Water temperature (TEMP)	✓	✓	✓	✓	✓	✓	✓
Turbidity (TURB) [LED transmission/front 30° scattering method]	–	✓	✓	–	–	–	–
Turbidity (TURB) [tungsten lamp 90° transmission/scattering method] with wiper	–	–	–	✓	✓	–	–
Turbidity (TURB) [LED 90° transmission/scattering method]	–	–	–	–	–	✓	✓
Water depth (DEP)	–	–	✓	✓	✓	2 m: – 10 m, 30 m: ✓	2 m: – 10 m, 30 m: ✓
GPS	–	–	✓	–	✓	–	✓

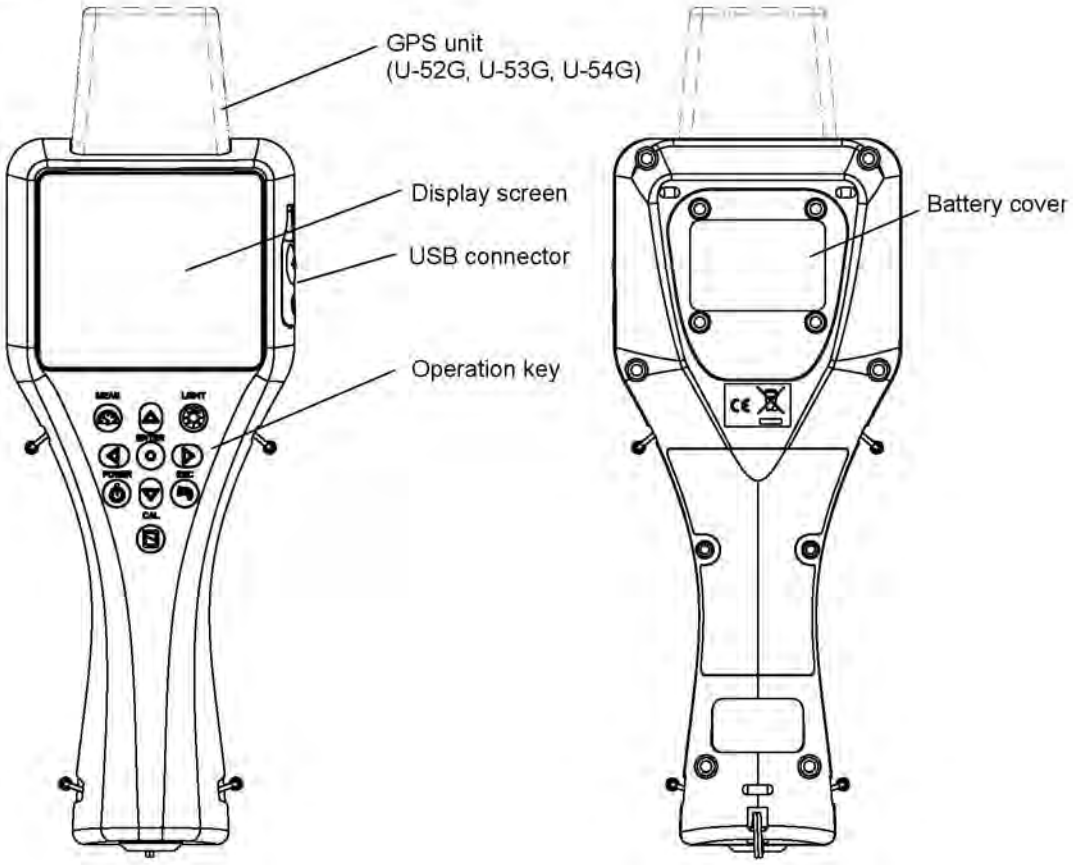
"✓" indicates a measurable parameter.

2.2 Packing list

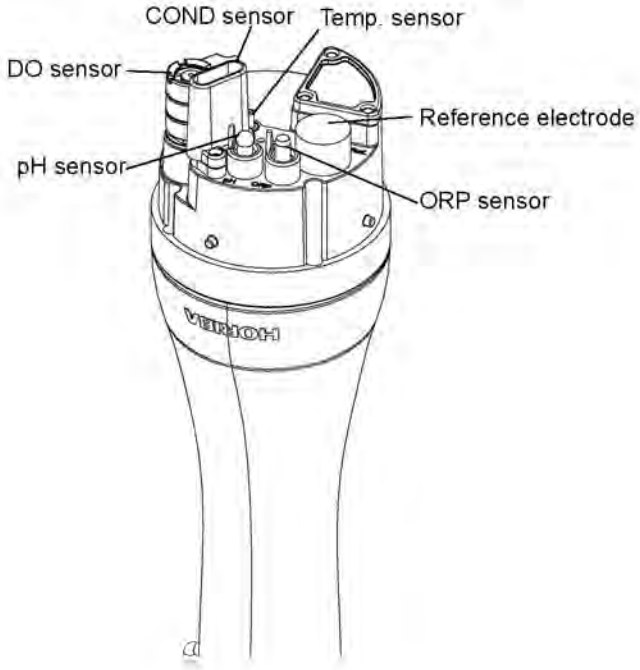
Parts Name	Quantity	Note
Control unit	1	
Sensor probe	1	
pH sensor (#7112)	1	
ORP sensor (#7313)	1	
Reference electrode (#7210)	1	
DO sensor (#7543)	1	
Turbidity sensor (#7800)	1	With U-52/U-52G only. Attached to the sensor probe.
Turbidity sensor (#7801)	1	With U-53/U-53G only. Attached to the sensor probe.
Turbidity sensor (#7802)	1	With U-54/U-54G only. Attached to the sensor probe.
pH 4 standard solution (#100-4)	1	500 mL
pH reference internal solution (#330)	1	250 mL
DO sensor internal solution set (#306)	1	Internal solution (50 mL), Sandpaper (#8000, #600), Syringe
DO Membrane spare parts set	1	
Spanner for DO sensor	1	
Cleaning brush	1	
calibration cup	1	transparent calibration cup, black calibration cup
Back pack	1	
Strap	1	
Alkaline batteries	4	LR14
Silicon grease	1	
Instruction manual	1	

2.3 Parts name and functions

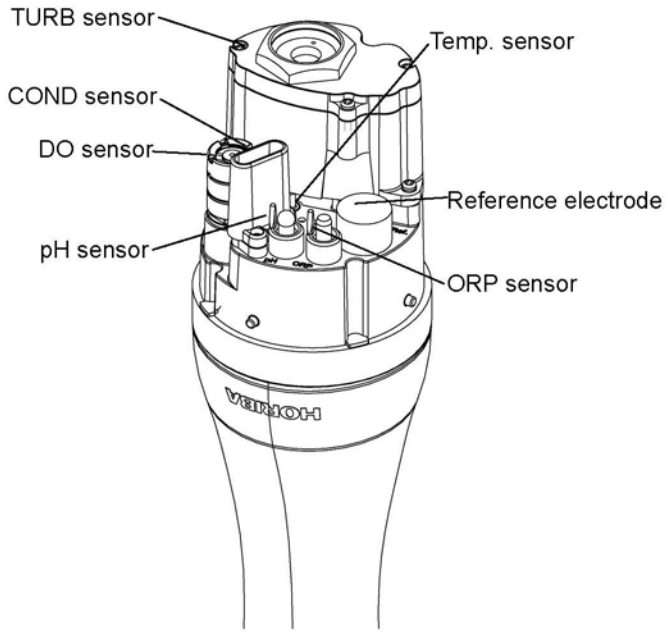
● Display



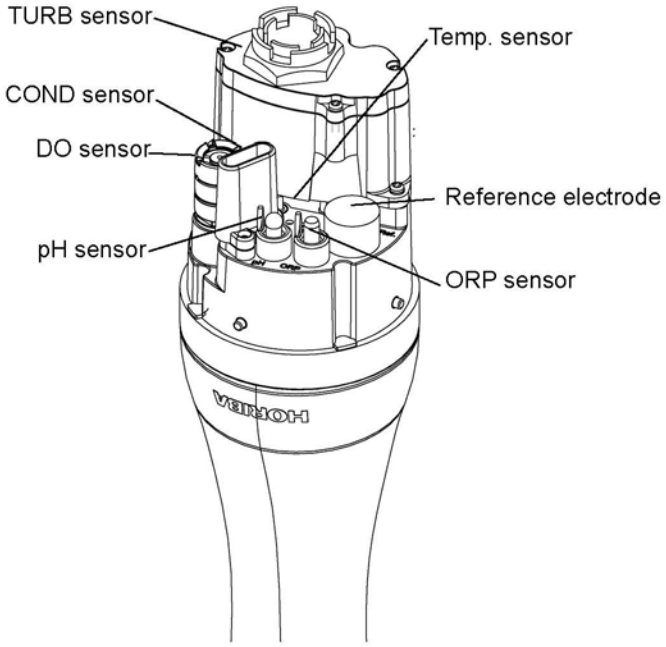
● Sensor probe (U-51)



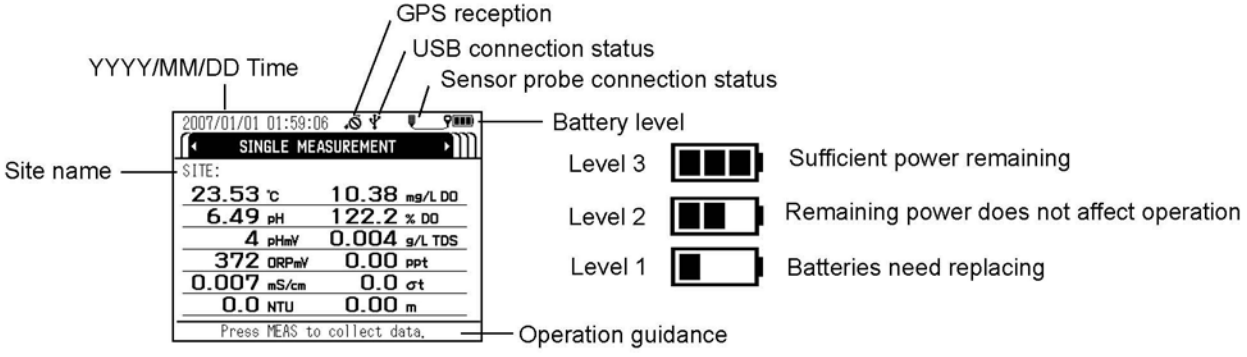
● Sensor probe (U-52)



● Sensor probe (U-53, U-54)



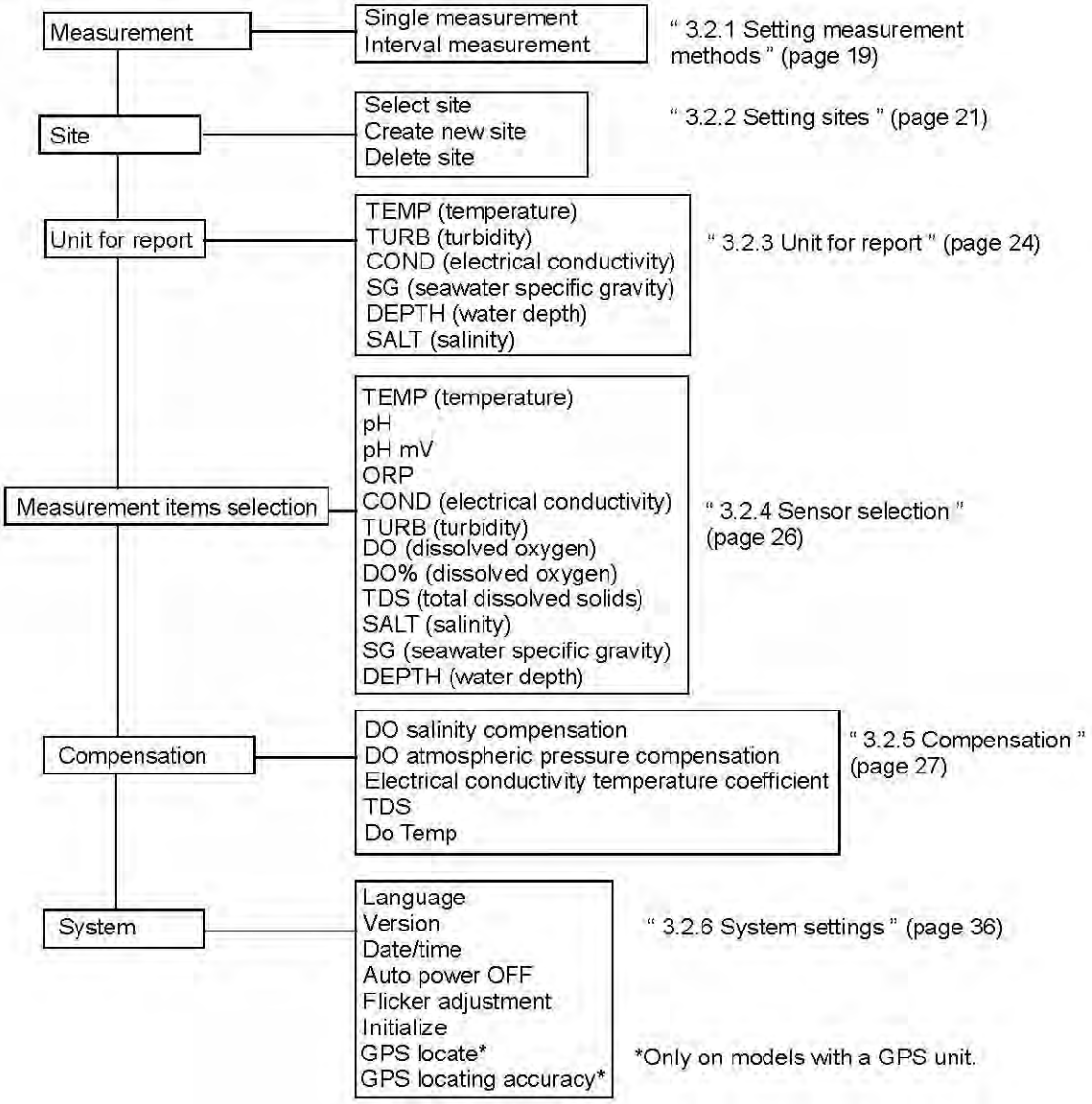
● Display screen



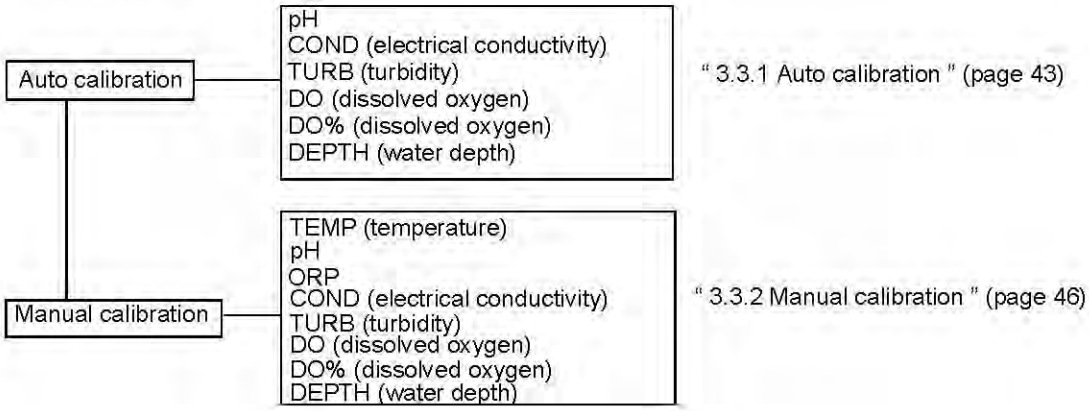
Operation key

	Key name	description
	POWER key	Turns the system's power ON/OFF. The initial screen appears immediately after turning the power ON. Press the POWER key for about 1 second to turn the power ON and OFF.
	MEAS key	When pressed in the measurement screen, used to set the measurement values of all the measurement parameters. Measurement values flash until the data stabilizes.
		When pressed in the setting, calibration or data operation screen, returns to the measurement screen.
	ENTER key	Used to execute functions, set entered values or store data in memory. Used to change the size of measurement value.
	CAL key	When pressed in the measurement screen, switches to the calibration screen.
	ESC key	Returns to the immediately preceding operation. When pressed during measurement, measurement is stopped.
	LIGHT key	Switches backlight between bright and dark. Setting backlight to bright shortens battery life.
	Left key	Moves the cursor to the left.
	Right key	Moves the cursor to the right.
	Up key	Moves the cursor up.
	Down key	Moves the cursor down.

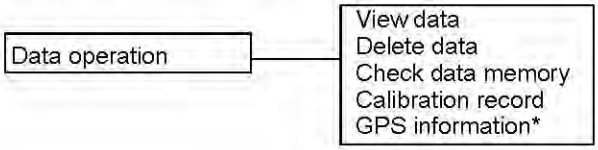
2.4 Setting menu items



2.5 Calibration menu items



2.6 Data operation menu items



" 3.5 Data operations " (page 68)

*Only on models with a GPS unit.

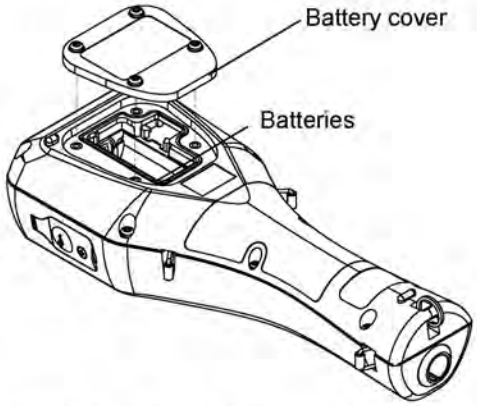
3 Basic Operation

3.1 System setup

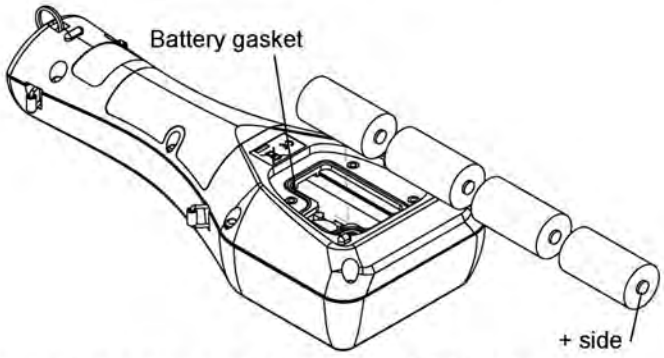
3.1.1 Inserting and replacing the batteries

The control unit is shipped without batteries. Follow the steps below to insert the batteries when using the system for the first time or replacing old batteries.

1. **Loosen the 4 screws on the battery cover by using No. 2 Phillips head screwdriver and remove the cover.**



2. **If replacing the batteries, discard the old batteries.**
3. **Insert new batteries in the control unit.**
   Check that the battery gasket is not dirty or twisted.



4. **Replace the battery cover and fasten it with the 4 screws.**
   Tighten the screws to less than 0.5 N·m.

Note

- Data and settings will not be lost when the batteries are replaced.
- If dirty or twisted, the battery gasket will fail to keep the batteries dry. Check its condition before closing the cover.
- To ensure long service life, replacing the battery gasket periodically (once a year) is recommended.

Precautions when using dry cell batteries

- Batteries to use: LR14 alkaline dry cell batteries (C-size dry cell batteries) or rechargeable nickel-metal hydride dry cell batteries (C-size)
  Do not use manganese batteries.
- Dry cell batteries used incorrectly may leak or burst. Always observe the following
  - Orient the batteries correctly (positive and negative ends in correct positions).
  - Do not combine new and used batteries, or batteries of different types.
  - Remove the batteries when not using the system for a prolonged period.
  - If batteries leak, have the system inspected at your nearest sales outlet.

● Battery life

- This battery life is an estimate for battery life when C-size alkaline dry cell batteries are used continuously.
- Using the backlight consumes a proportionate amount of battery power, shortening battery life.
- Searching position information using the GPS unit consumes a proportionate amount of battery power, shortening battery life.
- Nickel-metal hydride secondary batteries can be used, but the battery life is not guaranteed since it will vary according to usage (number of times data is saved, number of charges and charge state). In general, secondary batteries have one-half to two-thirds the life of C-size alkaline batteries.
- The 70-hour battery life figure applies to a control unit operating temperature of 20°C or more. The battery characteristics shorten the battery life at operating temperatures lower than 20°C, so check the remaining battery level, and replace the batteries before it reaches Level 1.
- The batteries packed with the system at the time of shipment are for checking operation. Their life is not guaranteed.
- The battery life is the amount of operating time the batteries can provide until the system stops operating. The system may fail to operate during measurement, so it is a good idea to check the remaining battery level and replace the batteries with new ones well before the batteries run out completely.

U-51/52/54

Battery life: 70 hours (for dark backlight)

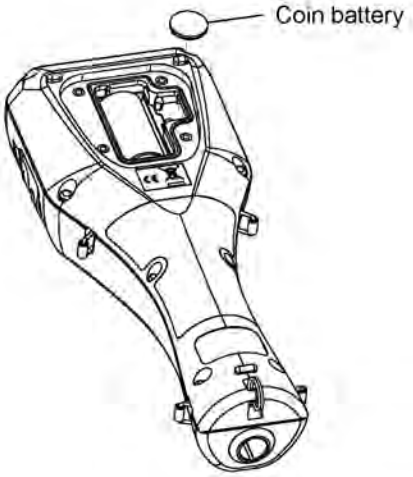
U-53

Battery life: 500 measurements (for dark backlight)

- Since U-53 is designed for turbidity measurement with wiper, its battery life is estimated in terms of the number of turbidity measurement sequences performed.
- Battery power is also consumed by measurement operations other than turbidity measurement.
- The battery life when turbidity measurement is not performed is about 70 hours.

3.1.2 Replacing the coin battery

- Coin battery to use: CR-2032
- The coin battery is only for the clock. It will provide problem-free operation for three years, but when using the clock continuously, it should be replaced once every two years as a precaution.
- When replacing the coin battery for the clock, leave the control unit ON. If the coin battery is replaced when the control unit is turned OFF, the clock will be reset to the default settings.



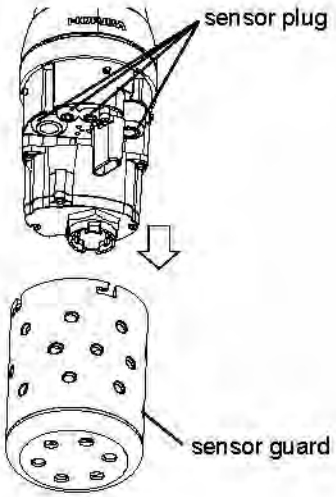
3.1.3 Attaching sensors

Note

- When attaching or replacing a sensor, wipe any moisture off the sensor probe and sensor.
- Be sure to keep water out of sensor connectors. If moisture comes in contact with a sensor connector, blow-dry it with dry air.
- The sensor probe is not waterproof when the sensor is not mounted.
- Take care not to tighten the sensor too much.

● Attaching the pH sensor

1. Remove the sensor guard.

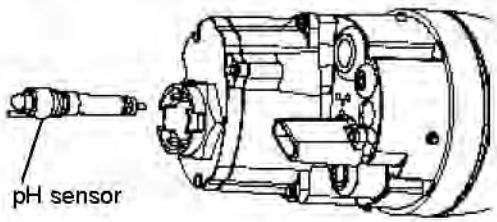


2. Remove the sensor plug.
3. Coat the pH sensor O-ring with a thin layer of silicon grease (part No. 3014017718).

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

4. Make sure there is no moisture on the sensor probe's sensor connector (marked "pH").
5. Fasten the pH sensor securely by hand.



6. Clean the sensor with an alcohol-soaked cloth.

Note

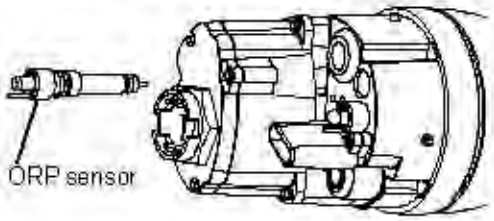
Do not throw away the black cap which has been put on the tip of the sensor. It will be used for the storage.

- **Attaching the ORP sensor**
  1. Remove the sensor guard.
  2. Remove the sensor plug.
  3. Coat the ORP sensor O-ring with a thin layer of grease (part No. 3014017718).

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

  4. Make sure there is no moisture on the sensor probe's sensor connector (marked "ORP").
  5. Fasten the ORP sensor securely by hand.



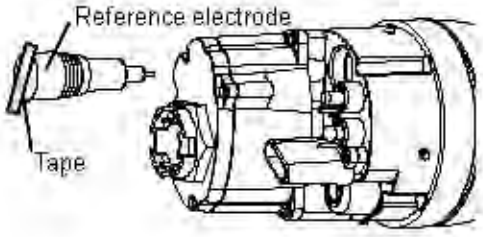
  6. Clean the sensor with an alcohol-soaked cloth.

- **Attaching the reference electrode**
  1. Remove the sensor guard.
  2. Remove the sensor plug.
  3. Coat the reference electrode O-ring with a thin layer of grease (part No. 3014017718).

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

  4. Make sure there is no moisture on the sensor probe's sensor connector (marked "REF").
  5. Fasten the reference electrode securely by hand.
  6. Remove the tape from the liquid junction part of the reference electrode.



Attaching the dissolved oxygen (DO) sensor

1. **Remove the membrane cap mounted on the DO sensor beforehand, and replace it with the new membrane cap provided. Replace the internal solution with fresh solution. The main component of the internal solution is potassium chloride (KCl), so the old solution can be disposed of down a sink or other drain.**

Reference

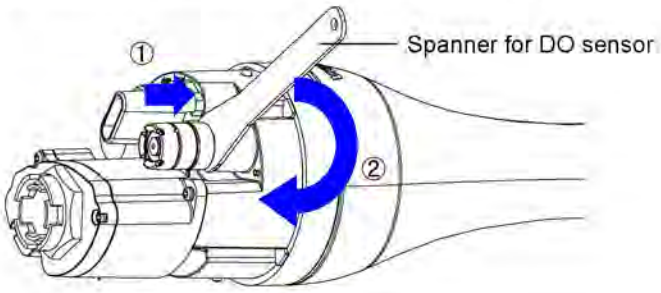
" 4.6 Replacing the membrane cap " (page 92)

2. **Screw in the DO sensor to attach it, allowing the internal solution to overflow slightly.**
3. **Use a soft cloth to wipe off the internal solution that overflowed onto the DO sensor.**
4. **Remove the sensor guard.**
5. **Remove the sensor plug.**
6. **Coat the DO sensor O-ring with a thin layer of grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

7. **Make sure there is no moisture on the sensor probe's sensor connector (marked "DO").**
8. **Fasten the DO sensor securely using the spanner for DO sensor.**
   - Hold the DO sensor with the provided spanner for DO sensor and push the sensor down. (Step 1 in figure below)
   - Screw the DO sensor in place. (Step 2 in figure below)



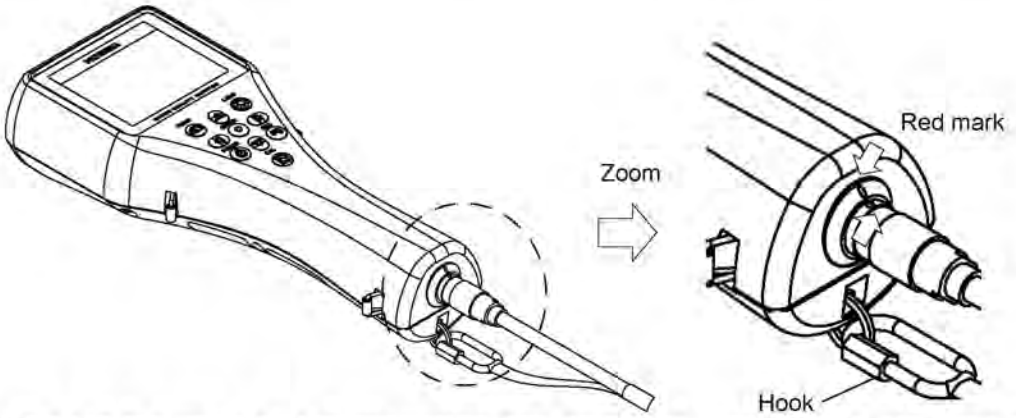
Note

Do not throw away the white cap which has been put on the tip of the sensor. It will be used for the storage.

3.1.4 Connecting the control unit and sensor probe

Note

Connect the control unit with its power OFF.

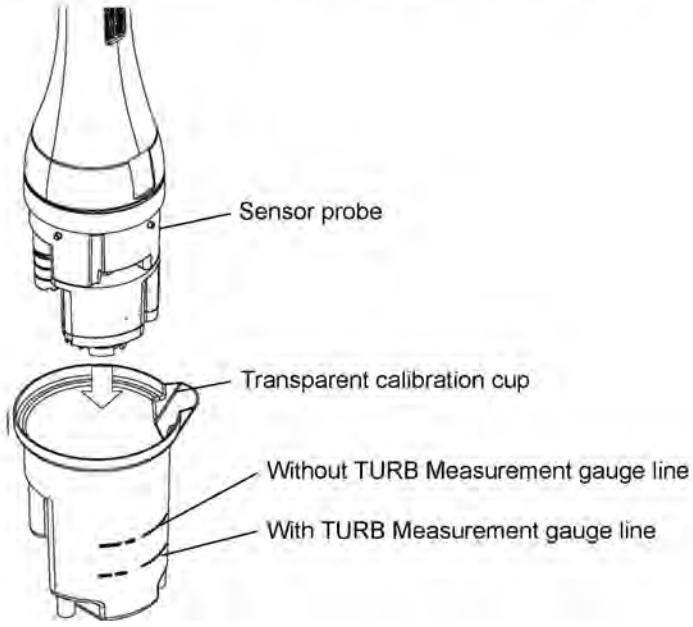


1. **Align the red mark on the connector, and press the connector in until you hear it click.**
2. **Connect the cable's hook to the display.**

3.1.5 Conditioning

Carry out the steps below when using the unit for the first time or when the system has not been used for 3 months or longer.

1. **Fill the transparent calibration cup to the line with pH 4 standard solution.**
   The transparent calibration cup has With TURB Measurement and Without TURB Measurement gauge lines.
2. **Insert the sensor probe in the transparent calibration cup.**



Note

Check that all sensors are attached.

3. **Press the control unit's POWER key for about 1 second to turn the power ON. Leave the unit for at least 30 minutes to condition the sensors.**

Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

Tip

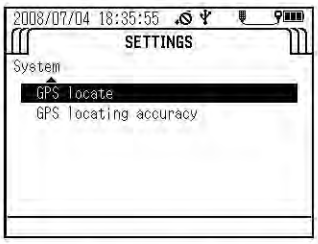
- The procedure for immersing the sensor probe in the pH standard solution is the same as that described in " 3.3.1 Auto calibration " (page 43).
  Auto calibration can be performed using the same pH 4 standard solution that was used in the conditioning procedure.
- Immersing the sensor in the standard solution is generally required for sensor conditioning, but a voltage supply is required for DO sensor conditioning. Turning ON the power of the control unit is necessary during sensor conditioning.
- DO value will be higher than usual for a while after turning power ON.
  See " 6.4.2 Conditioning of DO sensor " (page 107).

3.1.6 GPS (U-52G, U-53G, U-54G)

The GPS position measurement precision is proportional to the GPS position measurement time. When the position measurement precision increases, the position measurement time also increases. See " ● GPS locating accuracy" (page 18) for how to set the position measurement precision. See " ● GPS locate" (page 16) below for how to check acquired GPS data.

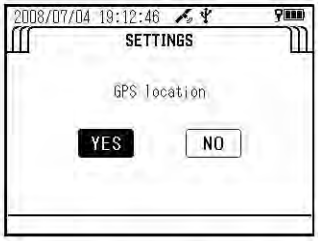
● **GPS locate**

1. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
2. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**
3. **Press the down (▽) key to move the cursor to "GPS locate", then press the ENTER key.**



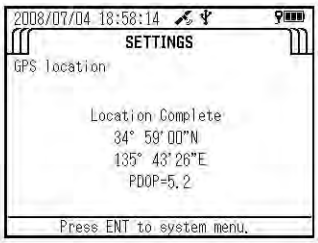
4. **The message "Press ENT key to start position measurement." appears. Press the ENTER key.**

5. The message "Execute GPS position measurement?" appears. Move the cursor to "YES", then press the ENTER key.



6. The message "Warming up. Please wait." appears. Wait until the system has finished warming up (about 10 seconds).

- Position measurement starts automatically when warmup has finished. Position measurement is performed up to 40 times.
- The GPS location complete screen appears after successful position measurement.

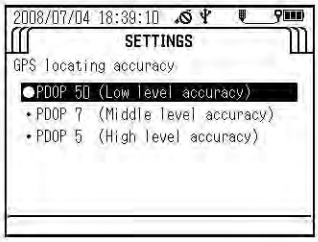


- The GPS location failure screen appears after position measurement has failed. Redo the measurement in a location free from obstacles, or wait for the meteorological conditions to improve before redoing the measurement.

● GPS locating accuracy

1. Press the right (▷) key to switch the display to the "SETTINGS" screen.
2. Press the down (▽) key to move the cursor to "System", then press the ENTER key.
3. Press the down (▽) key to move the cursor to "GPS locating accuracy", then press the ENTER key.

4. The screen below appears. Move the cursor to the locating accuracy, then press the ENTER key. The black circle (●) indicates the currently set precision.



3.2 Settings

3.2.1 Setting measurement methods

This section describes how to set the measurement method.

● Measurement methods

● U-51/U-52/U-54

Single measurement	Pressing the MEAS key acquires the 5-second average for the selected measurement parameter.
Interval measurement	Pressing the MEAS key acquires and saves the 5-second average for the selected measurement parameter in the set interval. The measurement interval can be set to any value between 10 seconds and 24 hours.

● U-53

The U-53 turbidity sensor uses a tungsten lamp. The lamp lights for about 10 seconds, and the average measurement value acquired during this interval is displayed.

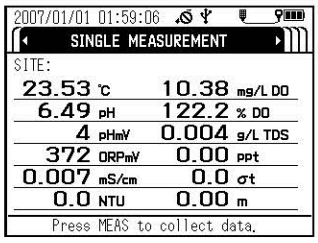
Single measurement	Pressing the MEAS key acquires the 5-second average for the selected measurement parameter after wiper operation. The 10-second average is acquired when measuring turbidity.
Interval measurement	Pressing the MEAS key acquires and saves the 5-second average for the selected measurement parameter in the set interval. The 10-second average is acquired when measuring turbidity. However, turbidity is an average value acquired over a period of approximately 10 seconds. The measurement interval can be set to any value between 30 seconds and 24 hours.

Reference

" 3.4 Measurement " (page 65)

● Operation method

1. **Press the control unit's POWER key for about 1 second to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

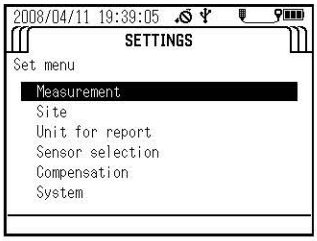


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

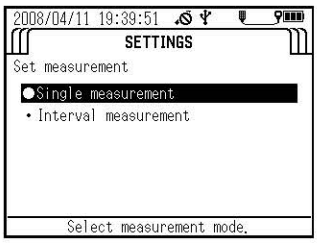
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Measurement", then press the ENTER key.**



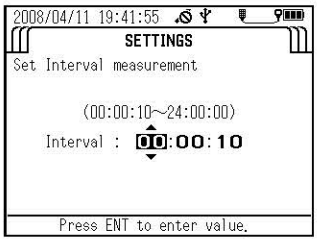
4. **Press the down (▽) key to move the cursor to the desired measurement mode. Press the ENTER key to save the setting.**

The black circle (●) indicates the currently selected measurement mode.



5. **If you selected "Interval measurement", the display switches to the screen used to set the measurement interval. Press the up (△) and down (▽) keys to set the measurement interval.**

The measurement interval can be set to any value between 10 seconds and 24 hours in the case of the U-51, U-52 and U-54, or between 30 seconds and 24 hours in the case of the U-53.



3.2.2 Setting sites

The site function allows position data to be connected to corresponding measurement data. Sites have the following specifications and features:

Site names: Text data consisting of up to 20 one-byte alphanumeric characters, spaces, etc.
Site names can be used for control unit searches and as labels for computer processing.
Site names allow measurement data to be saved with a name corresponding to the actual location where it was measured.

You can use site information as a search key when viewing data uploaded by a PC or data saved in the control unit (see " 3.5 Data operations " (page 68)).

- **Selecting sites**

  You can select previously created sites. The black circle (●) indicates the name of the currently selected site. No sites are created at new purchasing or after initialization. Select a site after first creating one from the "Create new site" menu.

- **Creating new sites**

  You can create and save new sites. Up to 20 site names can be registered.

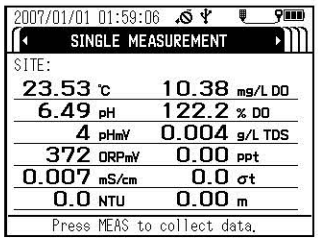
- **Deleting sites**

  You can select a previously created site and delete it.

● Operation methods

- **Selecting a site**

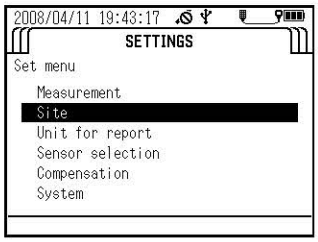
  1. **Press the control unit's POWER key for about 1 second to turn the power ON.**
     The "MEASUREMENT" screen appears after about 10 seconds.



Note

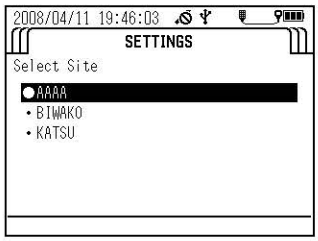
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

  2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
  3. **Press the down (▽) key to move the cursor to "Site", then press the ENTER key.**



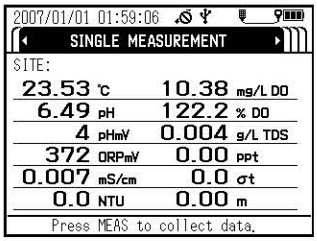
4. **Press the down (▽) key to move the cursor to "Select Site", then press the ENTER key to display the names of the currently saved sites.**

The black circle (●) indicates the currently selected site.



● **Creating a new site**

1. **Press the control unit's POWER key for about 1 second to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "Site", then press the ENTER key.**