Naval Facilities Engineering Command

Final

Investigation of Perfluorinated Compounds in Drinking Water
Sampling and Analysis Plan

Naval Air Station Whidbey Island
Oak Harbor, Washington

January 2017

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SAP Worksheet #1—Title and Approval Page

Final

Investigation of Perfluorinated Compounds in Drinking Water Sampling and Analysis Plan

Naval Air Station Whidbey Island
Oak Harbor, Washington

Contract Task Order 08

January 2017

Prepared for:

Department of the Navy
Naval Facilities Engineering Command
Northwest

Under the:

NAVFAC CLEAN 9000 Program
Contract N62470-16-D-9000

Prepared by:

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Virginia Beach, Virginia

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Approval Signatures:

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H.A.1461885000

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[redacted].1461885000
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI,
ou=USN, cn=[redacted]A.1461885000
Date: 2017.01.05 14:09:48 -05'00'

NAVFAC Atlantic – Chemist/Quality Assurance Officer Date

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Executive Summary

This Uniform Federal Policy (UFP) Sampling and Analysis Plan (SAP) outlines the sampling activities in support of an investigation of perfluorinated compounds (PFCs) in drinking water near Naval Air Station (NAS) Whidbey Island, Oak Harbor, Washington. Based on a desktop review of available data, groundwater is used as a source of drinking water in the vicinity of NAS Whidbey Island. Additionally, potable wells have been identified within 1 mile downgradient of the boundary of the on-Base sites where aqueous film-forming foam (AFFF) containing PFCs may have been used. Consequently, a release of PFCs to drinking water in the vicinity of NAS Whidbey Island is possible (CH2M, 2016). CH2M prepared this document under the U.S. Department of the Navy (Navy), Naval Facilities Engineering Command (NAVFAC), Comprehensive Long-term Environmental Action—Navy (CLEAN) 9000 Contract N62470-16-D-9000, Contract Task Order (CTO) 08, in accordance with the Navy's UFP-SAP policy guidance to help ensure that environmental data collected are scientifically sound, of known and documented quality, and suitable for intended uses.

NAS Whidbey Island was commissioned on September 21, 1942, and currently supports several types of aircraft, 7,600 military personnel, and 1,300 civilian personnel. The station's mission is to maintain and operate naval aircraft and aviation facilities and provide associated support.

Two sites, Area 16 (Ault Field Runway Ditches) and Area 31 (Former Runway Fire Training School), were identified at NAS Whidbey Island as locations where AFFF may have been used, resulting in a potential release of PFCs to groundwater. A third site, the current firefighting school, has a confirmed use of AFFF. AFFF use at these sites may have impacted off-Base drinking water sources located within 1 mile downgradient of the site boundaries.

The objective of this investigation is to determine whether PFCs are present above the project action limits (PALs) in the off-Base drinking water supply. Drinking water samples will be collected and analyzed to determine whether PFCs are present in drinking water above the PALs. If PFCs are present in drinking water above the PALs, an alternate drinking water source will be provided.

This SAP consists of 37 worksheets specific to the scope of work for the investigation of PFCs in off-Base drinking water at NAS Whidbey Island, Oak Harbor, Washington. All tables are embedded within the worksheets. All figures are included at the end of the document. Field standard operation procedures (SOPs) are included in **Appendix A**. Laboratory Department of Defense Environmental Laboratory Accreditation Program Accreditation letters are included in **Appendix B**. Meeting minutes from the initial project scoping sessions with NAVFAC Atlantic are included in **Appendix C**. The laboratory SOP is included in **Appendix D**.

The laboratory information cited in this SAP is specific to Test America–West Sacramento, Sacramento, California. If additional laboratory services are requested requiring modification to the existing SAP, revised SAP worksheets will be submitted to the Navy for approval.

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SAP Worksheets

Appendices

Tables

Figures

Acronyms and Abbreviations

±	plus or minus
%R	percent recovery
>	more than
≤	less than or equal to
°C	degree Celsius
µg/L	microgram per liter
AFFF	aqueous film-forming foam
AHA	activity hazard analysis
amu	atomic mass unit
bgs	below ground surface
CA	corrective action
CCV	continuing calibration verification
CH2M	CH2M HILL, Inc.
CLEAN	Comprehensive Long-term Environmental Action—Navy
CSM	conceptual site model
CTO	Contract Task Order
DL	detection limit
DoD	Department of Defense
DQI	data quality indicator
DV	data validation
EDD	electronic data deliverable
EDS	Environmental Data Services
ELAP	Environmental Laboratory Accreditation Program
FTL	Field Team Leader
H&S	health and safety
HA	Health Advisory
HQ	hazard quotient
HSM	Health and Safety Manager
HSP	Health and Safety Plan
ICAL	initial calibration
ID	identification
IS	internal standards
LCS	laboratory control sample
LCL	lower confidence limit
LOD	limit of detection
LOQ	limit of quantitation
mL	milliliter(s)
MPC	measurement performance criteria
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable

NAS	Naval Air Station
NAVFAC	Naval Facilities Engineering Command
Navy	U.S. Department of the Navy
OWS	oil/water separator
PAL	project action limit
PC	Project Chemist
PFC	perfluorinated compound
PFOA	perfluorooctanoic acid
PFOS	perfluorooctane sulfonate
PFBS	perfluorobutane sulfonate
PM	Project Manager
POC	point of contact
PQL	project quantitation limit
PQO	project quality objective
QA	quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	quality control
QM	Quality Manager
QSM	Quality Systems Manual
RL	reporting limit
RPD	relative percent difference
RPM	Remedial Project Manager
RT	retention time
SAP	Sampling and Analysis Plan
SBO	safe behavior observation
SOP	standard operating procedure
SSC	Site Safety Coordinator
STC	Senior Technical Consultant
TBD	to be determined
TestAmerica	TestAmerica Laboratories, Inc.
UCL	upper confidence limit
UFP	Uniform Federal Policy
USEPA	U.S. Environmental Protection Agency

SAP Worksheet #2—SAP Identifying Information

Site Name/Number:	Area 16 (Ault Field Runway Ditches) and Area 31 (Former Runway Fire Training School), Naval Air Station (NAS) Whidbey Island, Oak Harbor, Washington
Operable Unit/ Solid Waste Management Unit:	Not Applicable (N/A)
Contractor Name:	CH2M HILL, Inc. (CH2M)
Contract Number:	N62470-16-D-9000
Contract Title:	Comprehensive Long-term Environmental Action—Navy (CLEAN) 9000 Program
Work Assignment Number (optional):	Contract Task Order (CTO) 08

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the following:
   - *Guidance for Quality Assurance Project Plans (QAPP) (USEPA, 2002)*
   - *Uniform Federal Policy for Quality Assurance Project Plans (UFP)* (USEPA, 2005)
   - *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006)
2. Identify regulatory program: N/A
3. This document is a project-specific SAP.
4. List dates of scoping sessions that were held:

Scoping Session	Date
Project Scoping Session with Naval Facilities Engineering Command (NAVFAC) Atlantic[1]	August 2, 2016
Project Scoping Session NAVFAC Atlantic	August 31, 2016
Project Scoping Session with NAVFAC Atlantic and NAS Whidbey Island Remedial Project Manager (RPM)	October 5, 2016

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
Final Project-Specific Sampling and Analysis Plan, Hangar 5 and Rothbeck Ravine, NAS Whidbey Island	June 2015

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[1] Prior to the investigation outlined in this SAP, CH2M and NAVFAC Atlantic conducted two project scoping sessions, the outcomes of which were used to develop the overarching sampling plan, laboratory requirements, and other project-wide requirements. A copy of the meeting minutes from these project chartering meetings can be found in Appendix C.

SAP Worksheet #2—SAP Identifying Information (continued)

6. List organizational partners (stakeholders) and connection with lead organization:
   - NAS Whidbey Island – Base stakeholder
   - NAVFAC Atlantic – Technical Representative
7. Lead organization:
   - Department of the Navy (Navy)
8. If any required SAP elements or required information are N/A to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion as follows:
   - Crosswalk table is excluded because all required information is provided in this SAP.

SAP Worksheet #3—Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number	Email Address or Mailing Address
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

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SAP Worksheet #4—Project Personnel Sign-off Sheet

Name	Organization/Title/Role	Telephone Number	Signature/ E-mail receipt	SAP Section Reviewed	Date SAP Read
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]			

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SAP Worksheet #5—Project Organizational Chart

[redacted]

- - - - - - - - - - Lines of Communication
———————— Lines of Authority
QAO = Quality Assurance Officer

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SAP Worksheet #6—Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure
Communication with Navy (lead agency)	Technical Representative	[redacted]	[redacted] [redacted]	Primary point of contact (POC) for Navy; can delegate communication to other internal or external POCs.
Communication with Base	Base RPM	[redacted]	[redacted] [redacted]	Primary POC for facility; can delegate communication to other internal or external POCs. NAS Whidbey Island will notify RPM by email or telephone call within 24 hours for field changes affecting the scope or implementation of the design. NAS Whidbey Island will have 14 days for work plan review.
Communication regarding overall project status and implementation and primary POC with RPMs and project team	CH2M PM	[redacted]	[redacted] [redacted]	Oversees project and will be informed of project status by the Deputy PM and Task Manager. If field changes occur, PM will work with the RPM to communicate in-field changes to the team by email within 24 hours. All data results will be communicated to the project team following data receipt and review. All information and materials about the project will be forwarded to the Navy, as necessary. POC for FTL, deputy PM, and STC.
Quality issues during and technical communications for project implementation and data interpretation	CH2M QM and STC	[redacted]	[redacted] [redacted]	[redacted] the QM regarding quality issues during project implementation. The QM will report to the PM and the RPM. Contact STC regarding questions/issues encountered in the field, input on data interpretation, as needed. STC will have 24 hours to respond to technical field questions as necessary. Additionally, STC will review the data as necessary prior to Base and Navy discussions and reporting review.

SAP Worksheet #6—Communication Pathways (continued)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or Email	Procedure, Pathway, and so forth.
Health and safety (H&S)	CH2M HSM	[redacted]	[redacted]	Responsible for generation of the Health and Safety Plan (HSP) and approval of the activity hazard analyses (AHAs) prior to the start of fieldwork. The PM will contact the HSM as needed regarding questions/issues encountered in the field.
H&S	CH2M SSC	TBD	TBD	Responsible for the adherence of team members to the site safety requirements described in the HSP. Will report H&S incidents and near-misses to the PM as soon as possible.
Stop Work Order	CH2M PM	[redacted]	[redacted]	Any field member can immediately stop work if an unsafe condition that is immediately threatening to human health is observed. The field staff, FTL, or SSC should notify RPM the CH2M PM immediately. Ultimately, the FTL and PM can stop work for a period of time. NAVFAC Atlantic can stop work at any time.
	CH2M FTL Field Team Members	TBD TBD	TBD TBD	
Work plan changes in field	FTL	TBD	TBD	Documentation of deviations from the work plan will be made in the field logbook, and the PM will be notified immediately. Deviations will be made only with approval from the PM. The PM will communicate changes to the RPM.
Field changes/field progress reports	FTL	TBD	TBD	Documentation of field activities and work plan deviations (made with the approval of STC and/or QAO) in field logbooks; provide daily progress reports to PM.
Reporting laboratory data quality issues	Test America—West Sacramento	[redacted]	[redacted]	All quality assurance (QA)/quality control (QC) issues with project field samples will be reported within 2 days to the PC by the laboratory.
Analytical corrective actions (CAs)	PC	[redacted]	[redacted]	Any CAs for analytical issues will be determined by the FTL and/or the PC and reported to the PM within 4 hours. The PM will ensure SAP requirements are met by field staff for the duration of the project.

SAP Worksheet #6—Communication Pathways (continued)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or Email	Procedure, Pathway, and so forth.
Data tracking from field collection to database upload Release of analytical data	PC	[redacted]	[redacted] [redacted]	Tracks data from sample collection through database upload daily. No analytical data can be released until validation of the data is completed and has been approved by the PC. The PC will review analytical results within 24 hours of receipt for release to the project team. The PC will inform the Navy CLEAN program chemist who will notify the Navy QAO of any laboratory issues that would prevent the project from meeting project quality objectives (PQOs) or would cause significant delay in project schedule.
Reporting data quality issues	Data validation (DV)	[redacted]	[redacted] [redacted]	The data validator reviews and qualifies analytical data as necessary. The data along with a validation narrative are returned to the PC within 7 calendar days.
Field CAs	FTL, PM, and Deputy PM	TBD [redacted] [redacted]	TBD [redacted] [redacted] [redacted] [redacted]	Field issues requiring CA will be determined by the FTL and/or PM on an as-needed basis; the PM will ensure SAP requirements are met by field staff for the duration of the project. The FTL will notify the PM via phone of any need for CA within 4 hours. The FTL will notify the PM and the PM may notify the RPM of any field issues that would negatively affect the schedule or the ability to meet project data quality objectives.

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SAP Worksheet #7—Personnel Responsibilities Table

Name	Title/Role	Organizational Affiliation	Responsibilities
[redacted]	Technical Representative	NAVFAC Atlantic	Oversees project.
[redacted]	RPM	Base RPM	Oversees project, provides base-specific information, and coordinates with Base Environmental Manager.
[redacted]	PM	CH2M	Oversees and manages project activities.
[redacted]	Deputy PM	CH2M	Oversees and manages project activities.
[redacted]	STC/Quality Assurance Manager	CH2M	Provides senior technical support for project approach and execution. Provides QA oversight.
[redacted]	HSM	CH2M	Prepares HSP and manages H&S for all field activities.
[redacted]	Navy Program Chemist	CH2M	Provides SAP project delivery support, reviews and approves SAP, and performs final data evaluation and QA oversight.
[redacted]	SAP Reviewer	CH2M	Reviews and approves changes or revisions to the UFP-SAP.
[redacted]	PC	CH2M	Data management: Performs data evaluation and QA oversight, is the POC with laboratory and validator for analytical issues.
[redacted]	Data Validator	EDS	Validates laboratory data from an analytical standpoint prior to data use.
TBD	FTL	CH2M	Coordinates all field activities and sampling.
TBD	Field Staff	CH2M	Conducts field activities.
[redacted]	Laboratory PM	Test America– West Sacramento	Manages samples tracking and maintains good communication with PC.
[redacted]	Laboratory QAO	Test America– West Sacramento	Responsible for audits, CA, and checks of QA performance within the laboratory.

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SAP Worksheet #8—Special Personnel Training Requirements Table

N/A. No specialized training beyond standard H&S training is required for this project.

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SAP Worksheet #9—Project Scoping Session Participants Sheet[2]

Project Name: Investigation of PFCs in Drinking Water **Projected Date(s) of Sampling:** TBD **PM:** [redacted]			**Site Name**: Areas 16 and 31 **Site Location:** NAS Whidbey Island, Oak Harbor, Washington	
Date of Session: October 5, 2016 **Scoping Session Purpose:** To obtain consensus on the proposed investigation approach and schedule.				
Name	**Title/Project Role**	**Affiliation**	**Phone #**	**Email Address**
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

Comments

The purpose of the scoping session was to obtain consensus on the proposed path forward for the investigation of perfluorinated compounds (PFCs) in drinking water off-Base. The rationale for including Areas 16 and 31 in this investigation was that site history indicated that aqueous film-forming foam (AFFF) may have been used and/or released at the site.

The team reviewed the conceptual site models (CSMs) for Areas 16 and 31. An investigation of PFCs in groundwater was conducted in 2014. PFCs were not detected in groundwater at Area 16 (the runway ditches). Area 31 is a former firefighting training area; results of the 2014 groundwater sampling indicated that PFCs exceeded the provisional health advisory levels. Additional investigation was recommended; however, an investigation is not currently planned. An evaluation of the downgradient areas was conducted, which determined that potable wells are present downgradient of Areas 16 and 31. A total of 126 parcels were identified; at least 44 private wells have been confirmed among the 126 parcels. Additional wells may be present and will be determined using information from the Island County Department of Health along with other sources (including parcel owners). Drinking water samples will be collected from the private wells identified downgradient (pending owner approval).

Field work is expected to begin in late November or early December 2016.

Consensus Decision

The Team agrees to the approach for the investigation for PFCs in drinking water at Areas 16 and 31.

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[2] Prior to the investigation outlined in this SAP, CH2M and NAVFAC Atlantic conducted two project scoping sessions, the outcomes of which were used to develop the overarching sampling plan, laboratory requirements, and other project-wide requirements. A copy of the meeting minutes from these project chartering meetings can be found in Appendix C.

SAP Worksheet #9—Project Scoping Session Participants Sheet (continued)

Action Items

- CH2M will complete preparation of SAP.
- [redacted] with an updated spreadsheet showing the downgradient area parcels and wells so that she can work with her contact at the County Department of Health to obtain additional well information.
- Outline the ditches for Area 16 on the figures that will be included in the SAP.
- Update the Area 31 boundary so that it does not extend off Base property, as currently shown.
- [redacted] will provide the Unregulated Contaminant Monitoring Rule 3 data for the onsite wells next week.

SAP Worksheet #10—Conceptual Site Model

NAS Whidbey Island is located in Oak Harbor, Washington (**Figure 1**). **Figure 2** presents the site locations. Site descriptions and a brief history of the sites included in the investigation are presented in **Table 10-1**.

Table 10-1. Areas 16 and 31, Description and Background
NAS Whidbey Island, Oak Harbor, Washington

Site Name	Area 16, Ault Field Runway Ditches Area 31, Former Runway Fire Training School Fire Fighting School
Site Location	Area 16 comprises the Ault Field Runway Ditches, including the flight line area and the onsite drainage areas through Clover Valley. The Ault Field Runway Ditches consist of approximately 9 miles of connected ditches and 1 mile of culverts that drain the runway area and receive discharge from many of the station's storm drains. Most of the ditches eventually connect with the Clover Valley Stream, which flows east toward the Clover Valley Lagoon and Dugualla Bay (Navy, 1994). Area 31 is located adjacent to Area 16, approximately 1,200 feet northeast of the intersection of Runways 14-32 and 7-25. It comprises nearly flat ground with a low area located in the middle. The original fire training area is located in the northern portion of Area 31. The entire training area encompasses 1 to 2 acres, sloping gently southwest. The Fire Fighting School is located in the southwestern portion of Ault Field, approximately 0.25 mile inland from the Strait of Juan de Fuca.
Site History	Area 16 historical information does not indicate the presence of sumps, pumps, storage tanks, or piping associated with AFFF (Navy, 2016). There is potential surface water or stormwater runoff from the airfield runways where AFFF that may have been discharged from fire training or emergency activities could have migrated to the drainage ditch network and impacted groundwater at Area 16. In September 2015, one well, located in the central drainage ditch portion of Area 16 and screened in the deeper portion of the shallow aquifer (60 feet below ground surface [bgs]), was sampled and analyzed for PFCs. Perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) were not detected; however, the data report (Navy, 2016) concluded that additional investigation at Area 16 is necessary to assess potential PFOA and PFOS contamination in shallow groundwater and other drainage ditch sections. Area 31 was used for firefighting training from 1967 to 1982. Waste fuels such as aviation gasoline and jet-propulsion fuel, Grade 5, waste oil, solvents, thinners, and other flammable materials were ignited and extinguished in a shallow concrete burn pad. Oily water from the burn pad was drained through underground piping to an oil/water separator (OWS) in the southwestern corner of the drill area, approximately 200 feet from the burn pad. Water from the OWS was discharged to a small earthen ditch that led to a depression in the southwestern portion of Area 31 and subsequently drained to the runway ditches (Navy, 2016). Remedial actions at Area 31 have included removal of the OWS, an underground storage tank, and 32 cubic yards of soil (1996); soil venting and product recovery (approximately 884 gallons, terminated in 2000); and groundwater monitoring (ongoing) (Navy, 2013). In September 2015, two monitoring wells (MW31-7A and MW31-9A, 28 and 26 feet bgs, respectively) were sampled and analyzed for PFCs. Concentrations of PFOA and PFOS exceeded USEPA provisional health advisory screening levels in samples collected from both wells. The data report (Navy, 2016) concluded that additional investigation of the nature and extent of PFCs at Area 31 is warranted on the basis of the sample results of this investigation. The Fire Fighting School has been in use since 2011. AFFF is used to extinguish fires during firefighting training.
Current Use	Area 16 comprises active runways and a perimeter gravel road. Area 16 is bounded to the southwest by hangars and building structures. Area 31 is no longer an active facility. Remaining structures include the former burn pad, two pump houses, several monitoring wells, and a Department of Ecology tagged well (unknown use). To the south of the former burn pad are two additional buildings, one marked electrical vault, and one unmarked vault connecting to a Dialog Master Meter water meter.

SAP Worksheet #10—Conceptual Site Model (continued)

Site Conditions	**Physical Characteristics**	Area 16 surface features include impervious surface runways, the gravel perimeter road, and runway ditches. Adjacent to the runways the ditches are gently sloping open ditches draining toward the east into a main ditch system. The main ditch system receives the surface water flow via culverts. The main ditch system is oriented parallel and south of Runway 7-25, and drains to the east. Adjacent to the runways is grass, which is maintained by mowing operations. The ditches contain tall grasses and aquatic plants. Trees are mostly found to the south, beyond the ditches but within the Area 16 boundary. Area 31 surface features include two east-west oriented ditches located on either side of the gravel road that borders the southern boundary of the burn pad area. Approximately 400 feet south of the burn pad area is a depression bounded by gravel roads to the north, west, and east. The southern end of the depression area drains to the southeast along Runway 7-25. Miscellaneous piles of debris (concrete rubble) are located near the depression area, which is otherwise covered in grass (maintained by mowing operations) and brush such as Scotch broom. Trees and an upward slope leading to an elevated area bound the northeastern edge of Area 31 where the base perimeter fence is located. The Fire Fighting School includes a building and a concrete pad surrounded by grass. Wooded areas are present to the east and west. The Strait of Juan de Fuca is approximately 0.25 mile to the west, beyond the wooded area. The area is relatively flat.
	Geology and Hydrogeology	The surface soil in the vicinity of Ault Field primarily consists of artificial fill, post-glacial deposits, glaciomarine drift, and glacial deposits. Artificial fill, consisting of coarse- or fine-grained material, underlies the runway areas. Post-glacial deposits, consisting of peaty sand and silt, are generally found in the low-lying marshy areas (Navy, 1994). The 1994 Remedial Investigation (RI) Report (Navy, 1994) identified a confined aquifer beneath Area 16 at a depth of approximately 20 to greater than 150 feet bgs and consisting of fine to medium sand with some silt. Clay and silt of the Everson glaciomarine drift forms the overlying confining layer. A single, unconfined aquifer was identified beneath Area 31, interpreted to be the same as that encountered in Area 16, but without the glaciomarine drift that confines the aquifer in Area 16 (presumed to pinch out). Ault Field is located in a valley, with elevated areas to the southwest, northeast, and southeast of the field. Because Area 31 lies at the base of the southwest side of Monkey Hill, groundwater flow mimics topography in that area, flowing to the southwest, away from the hill and toward the Strait of Juan de Fuca. This was confirmed by the RI Report (Navy, 1994). Across the remainder of the Base, east of the runway, groundwater generally flows to the east, northeast, and southeast toward Clover Valley Stream, Clover Valley Lagoon, and Dugualla Bay. West of the runway, there is likely a component of flow to the west toward the Strait of Juan de Fuca. However, groundwater flow information for Areas 16 and 31, and the Fire Fighting School is sparse and the impact of off-Base water supply wells (pumping conditions) on localized groundwater flow is unknown. Consequently, selection of the downgradient area for the drinking water evaluation was conservative.
Drinking Water Source Evaluation		Based on Island County real estate records, 177 parcels are located downgradient of the sites, of which 66 are documented as served by private wells (**Figure 3**). Wells are reportedly screened at depths between 100 and 200 feet. It is unknown whether the remaining parcels are served by private wells.
Source Area		Fire training areas or emergency activities near the runways are likely source areas for potentially impacted surface water or stormwater runoff migrating to the Area 16 drainage ditch network. The former firefighting training area at Area 31 is a potential source area for PFCs detected in groundwater in this area. AFFF is currently in use at the Fire Fighting School.
Contaminants of Potential Concern		PFCs: PFOA, PFOS, and Perfluorobutane Sulfonate (PFBS)

SAP Worksheet #10—Conceptual Site Model (continued)

Migration Pathways	• Direct release to surface and/or subsurface soil • Transport of PFCs currently and/or historically present from soil to groundwater via infiltrating precipitation • Transport via advection with groundwater flow • Transport via surface water flow within the ditch network
Potential Receptors/Exposure Routes	Current users of drinking water (ingestion)

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SAP Worksheet #12—Measurement Performance Criteria Table – Field QC Samples

Matrix: Drinking Water
Analytical Group: PFCs
Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Matrix Spike(MS)/Matrix Spike Duplicate (MSD)	PFCs (PFOA, PFOS, and PFBS)	One per 20 samples	Accuracy/Precision	See **Worksheet #28**.
Field Duplicate (FD)		One per 20 samples	Precision	Relative percent difference (RPD) < 30%
Field Reagent Blank		One per property, per well where drinking water sampled	Bias/Contamination	No analytes detected more than (>) ½ limit of quantitation (LOQ) or > 1/10 sample concentration, whichever is greater
Cooler Temperature Indicator		One per cooler PFCs (PFOA, PFOS, and PFBS)	Accuracy/Representativeness	Temperature less than or equal to (≤) 10 degrees Celsius (°C), not frozen

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SAP Worksheet #13—Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation/ collection dates)	How Data Will Be Used	Limitations on Data Use
Drinking water sources	*CH2M. 2016. Drinking Water Source Verification Technical Memorandum.*	Desktop data search performed by CH2M in September 2016 using available historical documents and public records to identify off-Base, potentially impacted, drinking water sources.	Identify drinking water sources	None
PFCs in groundwater	*MMEC Group. 2016. Summary Report. Groundwater Sampling for Perfluorinated Compounds, Hangar 5 and Areas 16 and 31, NAS Whidbey, Oak Harbor, Washington. April*	MMEC Group collected groundwater in 2014 for PFC analysis.	Determination of whether PFCs are present in groundwater	None

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SAP Worksheet #14—Summary of Project Tasks

Pre-sampling Tasks

- Subcontractor procurement
  - Analytical laboratory
  - Data Validator
- Fieldwork scheduling

Sampling Tasks

Applicable field book and forms should be filled out completely each day.

- Drinking Water Samples
  - Samples will be collected in accordance with **Worksheet #18** and with the standard operating procedures (SOPs) listed in **Worksheet #21** and provided in **Appendix A**.
  - Drinking water samples will be collected from properties following the sampling protocol as specified in **Worksheet #18**.
  - Drinking water samples will be collected, if possible, at a tap or spigot prior to treatment or filtering. Samples will be collected following 3 to 5 minutes of flushing.

Analyses and Testing Tasks

- The subcontracted analytical laboratory will process and prepare samples for analyses, and will analyze all samples for linear PFBS, and for branched and linear PFOA and PFOS in accordance with **Worksheets #18** and **#19**.

Quality Control Tasks

- Implement SOPs for field and laboratory activities being performed.
- QC samples are described on **Worksheets #14 and #20**.

Secondary Data

- See **Worksheet #13**.

Data Validation, Review, and Management Tasks

- See **Worksheets #34 through #36** for discussion of data management procedures.

Documentation and Reporting

- A summary of field activities as well as a data evaluation will be documented in a technical memorandum and submitted to NAS Whidbey Island Environmental, the Base RPM, and Navy Technical Representatives for review and approval.

Assessment/Audit Tasks

- See **Worksheets #31** and **#32**.

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SAP Worksheet #15-1—Reference Limits and Evaluation Table

Matrix: Drinking water

Analytical Group: PFCs

Analyte	Chemical Abstract Services (CAS) Number	PAL[a] (µg/L)	PAL Reference	PQL Goal[b] (µg/L)	Laboratory-specific Limits (µg/L)			LCS and MS/MSD Recovery Limits and RPD (percent)[c]		
					LOQs	LODs	DLs	LCL	UCL	RPD
Perfluorooctanesulfonic Acid (PFOS)	1763-23-1	0.07	USEPA Lifetime Health Advisory[a]	0.035	0.06	0.048	0.016	70	130	30
Perfluorooctanoic Acid (PFOA)	335-67-1	0.07	USEPA Lifetime Health Advisory[a]	0.035	0.03	0.024	0.0094	70	130	30
Perfluorobutanesulfonic Acid (PFBS)	375-73-5	380	RSLs Tapwater HQ = 1 (May 2016)	190	0.14	0.11	0.048	60	130	30

Notes:

The PFOS and PFOA U.S. Environmental Protection Agency (USEPA) Lifetime Health Advisories are from the USEPA Office of Water.

[b] The Project Quantitation Limit (PQL) Goal is half of the PAL.

[c] Accuracy and precision limits are in house laboratory limits.

DL = detection limit
HQ = hazard quotient
LCL = lower criteria limit
LCS = laboratory control sample
LOD = limit of detection
LOQ= limit of quantitation
MS = matrix spike
MSD = matrix spike duplicate
RPD = relative percent difference
RSL = regional screening level
UCL = upper confidence limit
µg/L = microgram per liter

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SAP Worksheet #16—Project Schedule/Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable
		Anticipated Date of Initiation	**Anticipated Date of Completion**	
SAP Schedule				
Draft SAP preparation	CH2M	09/30/16	10/14/16	Draft SAP
Navy SAP review	Navy, NAS Whidbey Island	10/14/16	10/19/16	Comments
Final SAP	CH2M	10/20/16	10/31/16	Final SAP
Sampling Schedule				
Off-Base Drinking Water Sampling	CH2M	11/28/16	12/16/16	N/A
Analytical Data	Subcontractor	7-day turnaround time		
Rapid Response – Drinking Water Supply	CH2M	Within 14 days of sample date, if warranted (See **Worksheet #11**). An alternative drinking water sources will be supplied within 24 hours of identification of results above the PALs.	Within 14 days of sample date, if warranted (See **Worksheet #11**). An alternative drinking water sources will be supplied within 24 hours of identification of results above the PALs.	N/A
Offsite Drinking Water Sampling Step-out 1	CH2M	TBD	TBD	N/A
Analytical Data	Subcontractor	7-day turnaround time		
Data management	CH2M	11/28/2016	TBD	N/A
Reporting	CH2M	December 2016	February 2016	Results Technical Memorandum

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SAP Worksheet #17—Sampling Design and Rationale

The objective of this investigation is to determine whether PFCs are present in the off-Base drinking water supply above the PALs near Areas 16 and 31 at NAS Whidbey Island. **Table 17-1** presents the sampling strategy and rationale.

Table 17-1. Areas 16 and 31, Sampling Strategy and Rationale
NAS Whidbey Island, Oak Harbor, Washington

Matrix	Depth of Samples	Analysis and Method	Number of Samples	Rationale
Drinking Water	N/A[a]	PFCs (PFOA, PFOS, and PFBS) USEPA Method 537	177[b]	Drinking water samples will be collected (**Figure 3**) and analyzed to determine whether PFOA, PFOS, and PFBS are present above the PALs (**Worksheet #15**)

Notes:
[a] Drinking water samples will be collected as described in **Worksheet #14**.
[b] Number of samples is estimated pending field verification; up to 177 samples identified within initial sampling area may be collected.

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SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
WI-AF-RW01	WI-AF-RW01-MMYY	DW[1]	N/A	PFCs	1	Refer to **Worksheet #21**
	WI-AF-RW01-MMYY-MS				1 (MS)	
	WI-AF-RW01-MMYY-SD				1 (SD)	
WI-AF-RW02	WI-AF-RW02-MMYY				1	
WI-AF-RW03	WI-AF-RW03-MMYY				1	
WI-AF-RW04	WI-AF-RW04-MMYY				1	
WI-AF-RW05	WI-AF-RW05-MMYY				1	
WI-AF-RW06	WI-AF-RW06-MMYY				1	
WI-AF-RW07	WI-AF-RW07-MMYY				1	
WI-AF-RW08	WI-AF-RW08-MMYY				1	
WI-AF-RW09	WI-AF-RW09-MMYY				1	
WI-AF-RW10	WI-AF-RW10-MMYY				1	
WI-AF-RW11	WI-AF-RW11-MMYY				1	
WI-AF-RW12	WI-AF-RW12-MMYY				1	
WI-AF-RW13	WI-AF-RW13-MMYY				1	
WI-AF-RW14	WI-AF-RW14-MMYY				1	
WI-AF-RW15	WI-AF-RW15-MMYY				1	
WI-AF-RW16	WI-AF-RW16-MMYY				1	Refer to **Worksheet #21**
WI-AF-RW17	WI-AF-RW17-MMYY				1	
WI-AF-RW18	WI-AF-RW18-MMYY				1	
WI-AF-RW19	WI-AF-RW19-MMYY				1	
WI-AF-RW20	WI-AF-RW20-MMYY				2 (FD)	
	WI-AF-RW20P-MMYY					
WI-AF-RW21	WI-AF-RW21-MMYY				1	
	WI-AF-RW21-MMYY-MS				1 (MS)	
	WI-AF-RW21-MMYY-SD				1 (SD)	
WI-AF-RW22	WI-AF-RW22-MMYY				1	
WI-AF-RW23	WI-AF-RW23-MMYY				1	
WI-AF-RW24	WI-AF-RW24-MMYY				1	
WI-AF-RW25	WI-AF-RW25-MMYY				1	
WI-AF-RW26	WI-AF-RW26-MMYY				1	
WI-AF-RW27	WI-AF-RW27-MMYY				1	
WI-AF-RW28	WI-AF-RW28-MMYY				1	
WI-AF-RW29	WI-AF-RW29-MMYY				1	
WI-AF-RW30	WI-AF-RW30-MMYY				1	
WI-AF-RW31	WI-AF-RW31-MMYY				1	
WI-AF-RW32	WI-AF-RW32-MMYY				1	
WI-AF-RW33	WI-AF-RW33-MMYY				1	
WI-AF-RW34	WI-AF-RW34-MMYY				1	
WI-AF-RW35	WI-AF-RW35-MMYY				1	
WI-AF-RW36	WI-AF-RW36-MMYY				1	
WI-AF-RW37	WI-AF-RW37-MMYY				1	

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
WI-AF-RW38	WI-AF-RW38-MMYY	DW[1]	N/A	PFCs	1	Refer to **Worksheet #21**
WI-AF-RW39	WI-AF-RW39-MMYY				1	
WI-AF-RW40	WI-AF-RW40-MMYY				2 (FD)	
	WI-AF-RW40P-MMYY					
WI-AF-RW41	WI-AF-RW41-MMYY				1	
	WI-AF-RW41-MMYY-MS				1 (MS)	
	WI-AF-RW41-MMYY-SD				1 (SD)	
WI-AF-RW42	WI-AF-RW42-MMYY				1	
WI-AF-RW43	WI-AF-RW43-MMYY				1	
WI-AF-RW44	WI-AF-RW44-MMYY				1	
WI-AF-RW45	WI-AF-RW45-MMYY				1	
WI-AF-RW46	WI-AF-RW46-MMYY				1	
WI-AF-RW47	WI-AF-RW47-MMYY				1	
WI-AF-RW48	WI-AF-RW48-MMYY				1	
WI-AF-RW49	WI-AF-RW49-MMYY				1	
WI-AF-RW50	WI-AF-RW50-MMYY				1	
WI-AF-RW51	WI-AF-RW51-MMYY				1	
WI-AF-RW52	WI-AF-RW52-MMYY				1	
WI-AF-RW53	WI-AF-RW53-MMYY				1	
WI-AF-RW54	WI-AF-RW54-MMYY				1	
WI-AF-RW55	WI-AF-RW55-MMYY				1	
WI-AF-RW56	WI-AF-RW56-MMYY				1	
WI-AF-RW57	WI-AF-RW57-MMYY				1	
WI-AF-RW58	WI-AF-RW58-MMYY				1	
WI-AF-RW59	WI-AF-RW59-MMYY				1	
WI-AF-RW60	WI-AF-RW60-MMYY				2 (FD)	
	WI-AF-RW60P-MMYY					
WI-AF-RW61	WI-AF-RW61-MMYY				1	
	WI-AF-RW61-MMYY-MS				1 (MS)	
	WI-AF-RW61-MMYY-SD				1 (SD)	
WI-AF-RW62	WI-AF-RW62-MMYY				1	
WI-AF-RW63	WI-AF-RW63-MMYY				1	
WI-AF-RW64	WI-AF-RW64-MMYY				1	
WI-AF-RW65	WI-AF-RW65-MMYY				1	
WI-AF-RW66	WI-AF-RW66-MMYY				1	
WI-AF-RW67	WI-AF-RW67-MMYY				1	
WI-AF-RW68	WI-AF-RW68-MMYY				1	
WI-AF-RW69	WI-AF-RW69-MMYY				1	
WI-AF-RW70	WI-AF-RW70-MMYY				1	
WI-AF-RW71	WI-AF-RW71-MMYY				1	
WI-AF-RW72	WI-AF-RW72-MMYY				1	
WI-AF-RW73	WI-AF-RW73-MMYY				1	
WI-AF-RW74	WI-AF-RW74-MMYY				1	
WI-AF-RW75	WI-AF-RW75-MMYY				1	
WI-AF-RW76	WI-AF-RW76-MMYY				1	
WI-AF-RW77	WI-AF-RW77-MMYY				1	
WI-AF-RW78	WI-AF-RW78-MMYY				1	

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
WI-AF-RW79	WI-AF-RW79-MMYY	DW[1]	N/A	PFCs	1	Refer to **Worksheet #21**
WI-AF-RW80	WI-AF-RW80-MMYY				2 (FD)	
	WI-AF-RW80P-MMYY					
WI-AF-RW81	WI-AF-RW81-MMYY				1	
	WI-AF-RW81-MMYY-MS				1 (MS)	
	WI-AF-RW81-MMYY-SD				1 (SD)	
WI-AF-RW82	WI-AF-RW82-MMYY				1	
WI-AF-RW83	WI-AF-RW83-MMYY				1	
WI-AF-RW84	WI-AF-RW84-MMYY				1	
WI-AF-RW85	WI-AF-RW85-MMYY				1	
WI-AF-RW86	WI-AF-RW86-MMYY				1	
WI-AF-RW87	WI-AF-RW87-MMYY				1	
WI-AF-RW88	WI-AF-RW88-MMYY				1	
WI-AF-RW89	WI-AF-RW89-MMYY				1	
WI-AF-RW90	WI-AF-RW90-MMYY				1	
WI-AF-RW91	WI-AF-RW91-MMYY				1	
WI-AF-RW92	WI-AF-RW92-MMYY				1	
WI-AF-RW93	WI-AF-RW93-MMYY				1	
WI-AF-RW94	WI-AF-RW94-MMYY				1	
WI-AF-RW95	WI-AF-RW95-MMYY				1	
WI-AF-RW96	WI-AF-RW96-MMYY				1	
WI-AF-RW97	WI-AF-RW97-MMYY				1	
WI-AF-RW98	WI-AF-RW98-MMYY				1	
WI-AF-RW99	WI-AF-RW99-MMYY				1	
WI-AF-RW100	WI-AF-RW100-MMYY				2 (FD)	
	WI-AF-RW100P-MMYY					
WI-AF-RW101	WI-AF-RW101-MMYY				1	
	WI-AF-RW101-MMYY-MS				1 (MS)	
	WI-AF-RW101-MMYY-SD				1 (SD)	
WI-AF-RW102	WI-AF-RW102-MMYY				1	
WI-AF-RW103	WI-AF-RW103-MMYY				1	
WI-AF-RW104	WI-AF-RW104-MMYY				1	
WI-AF-RW105	WI-AF-RW105-MMYY				1	
WI-AF-RW106	WI-AF-RW106-MMYY				1	
WI-AF-RW107	WI-AF-RW107-MMYY				1	
WI-AF-RW108	WI-AF-RW108-MMYY				1	
WI-AF-RW109	WI-AF-RW109-MMYY				1	

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
WI-AF-RW110	WI-AF-RW110-MMYY	DW[1]	N/A	PFCs	1	Refer to **Worksheet #21**
WI-AF-RW111	WI-AF-RW111-MMYY				1	
WI-AF-RW112	WI-AF-RW112-MMYY				1	
WI-AF-RW113	WI-AF-RW113-MMYY				1	
WI-AF-RW114	WI-AF-RW114-MMYY				1	
WI-AF-RW115	WI-AF-RW115-MMYY				1	
WI-AF-RW116	WI-AF-RW116-MMYY				1	
WI-AF-RW117	WI-AF-RW117-MMYY				1	
WI-AF-RW118	WI-AF-RW118-MMYY				1	
WI-AF-RW119	WI-AF-RW119-MMYY				1	
WI-AF-RW120	WI-AF-RW120-MMYY				2 (FD)	
	WI-AF-RW120P-MMYY					
WI-AF-RW121	WI-AF-RW121-MMYY				1	
	WI-AF-RW121-MMYY-MS				1 (MS)	
	WI-AF-RW121-MMYY-SD				1 (SD)	
WI-AF-RW122	WI-AF-RW122-MMYY				1	
WI-AF-RW123	WI-AF-RW123-MMYY				1	
WI-AF-RW124	WI-AF-RW124-MMYY				1	
WI-AF-RW125	WI-AF-RW125-MMYY				1	
WI-AF-RW126	WI-AF-RW126-MMYY				1	
WI-AF-RW127	WI-AF-RW127-MMYY				1	
WI-AF-RW128	WI-AF-RW128-MMYY				1	
WI-AF-RW129	WI-AF-RW129-MMYY				1	
WI-AF-RW130	WI-AF-RW130-MMYY				1	
WI-AF-RW131	WI-AF-RW131-MMYY				1	
WI-AF-RW132	WI-AF-RW132-MMYY				1	
WI-AF-RW133	WI-AF-RW133-MMYY				1	
WI-AF-RW134	WI-AF-RW134-MMYY				1	
WI-AF-RW135	WI-AF-RW135-MMYY				1	
WI-AF-RW136	WI-AF-RW136-MMYY				1	
WI-AF-RW137	WI-AF-RW137-MMYY				1	
WI-AF-RW138	WI-AF-RW138-MMYY				1	
WI-AF-RW139	WI-AF-RW139-MMYY				1	
WI-AF-RW140	WI-AF-RW140-MMYY				2 (FD)	
	WI-AF-RW140P-MMYY					
WI-AF-RW141	WI-AF-RW141-MMYY				1	
WI-AF-RW142	WI-AF-RW142-MMYY				1	
WI-AF-RW143	WI-AF-RW143-MMYY				1	
WI-AF-RW144	WI-AF-RW144-MMYY				1	
WI-AF-RW145	WI-AF-RW145-MMYY				1	

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
WI-AF-RW146	WI-AF-RW146-MMYY	DW[1]	N/A	PFCs	3 (MS/MSD)	Refer to **Worksheet #21**
	WI-AF-RW146-MMYY-MS					
	WI-AF-RW146-MMYY-SD					
WI-AF-RW147	WI-AF-RW147-MMYY				1	
WI-AF-RW148	WI-AF-RW148-MMYY				1	
WI-AF-RW149	WI-AF-RW149-MMYY				1	
WI-AF-RW150	WI-AF-RW150-MMYY				1	
WI-AF-RW151	WI-AF-RW151-MMYY				1	
WI-AF-RW152	WI-AF-RW152-MMYY				1	
WI-AF-RW153	WI-AF-RW153-MMYY				1	
WI-AF-RW154	WI-AF-RW154-MMYY				1	
WI-AF-RW155	WI-AF-RW155-MMYY				1	
WI-AF-RW156	WI-AF-RW156-MMYY				1	
WI-AF-RW157	WI-AF-RW157-MMYY				1	
WI-AF-RW158	WI-AF-RW158-MMYY				1	
WI-AF-RW159	WI-AF-RW159-MMYY				1	
WI-AF-RW160	WI-AF-RW160-MMYY				2 (FD)	
	WI-AF-RW160P-MMYY					
WI-AF-RW161	WI-AF-RW161-MMYY				1	
WI-AF-RW162	WI-AF-RW162-MMYY				1	
WI-AF-RW163	WI-AF-RW163-MMYY				1	
WI-AF-RW164	WI-AF-RW164-MMYY				1	
WI-AF-RW165	WI-AF-RW165-MMYY				1	
WI-AF-RW166	WI-AF-RW166-MMYY				1	
WI-AF-RW167	WI-AF-RW167-MMYY				1	
WI-AF-RW168	WI-AF-RW168-MMYY				1	
WI-AF-RW169	WI-AF-RW169-MMYY				1	
WI-AF-RW170	WI-AF-RW170-MMYY				1	
WI-AF-RW171	WI-AF-RW171-MMYY				1	
WI-AF-RW172	WI-AF-RW172-MMYY				1	
WI-AF-RW173	WI-AF-RW173-MMYY				1	
WI-AF-RW174	WI-AF-RW174-MMYY				3 (MS/MSD)	
	WI-AF-RW174-MMYY-MS					
	WI-AF-RW174-MMYY-SD					
WI-AF-RW175	WI-AF-RW175-MMYY				1	
WI-AF-RW176	WI-AF-RW176-MMYY				1	
WI-AF-RW177	WI-AF-RW177-MMYY				2 (FD)	
	WI-AF-RW177P-MMYY					

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
WI-AF-QC	WI-AF-FB01-MMYY	QC	N/A N/A	PFCs	1 (FB)	Refer to **Worksheet #21**
	WI-AF-FB02-MMYY				1 (FB)	
	WI-AF-FB03-MMYY				1 (FB)	
	WI-AF-FB04-MMYY				1 (FB)	
	WI-AF-FB05-MMYY				1 (FB)	
	WI-AF-FB06-MMYY				1 (FB)	
	WI-AF-FB07-MMYY				1 (FB)	
	WI-AF-FB08-MMYY				1 (FB)	
	WI-AF-FB09-MMYY				1 (FB)	
	WI-AF-FB10-MMYY				1 (FB)	
	WI-AF-FB11-MMYY		N/A		1 (FB)	
	WI-AF-FB12-MMYY		N/A		1 (FB)	
	WI-AF-FB13-MMYY		N/A		1 (FB)	
	WI-AF-FB14-MMYY		N/A		1 (FB)	
	WI-AF-FB15-MMYY		N/A		1 (FB)	
	WI-AF-FB16-MMYY		N/A		1 (FB)	
	WI-AF-FB17-MMYY		N/A		1 (FB)	
	WI-AF-FB18-MMYY		N/A		1 (FB)	
	WI-AF-FB19-MMYY		N/A		1 (FB)	
	WI-AF-FB20-MMYY		N/A		1 (FB)	
	WI-AF-FB21-MMYY		N/A		1 (FB)	
	WI-AF-FB22-MMYY		N/A		1 (FB)	
	WI-AF-FB23-MMYY		N/A		1 (FB)	
	WI-AF-FB24-MMYY		N/A		1 (FB)	
WI-AF-QC	WI-AF-FB25-MMYY	QC	N/A	PFCs	1 (FB)	
	WI-AF-FB26-MMYY		N/A		1 (FB)	
	WI-AF-FB27-MMYY		N/A		1 (FB)	
	WI-AF-FB28-MMYY		N/A		1 (FB)	
	WI-AF-FB29-MMYY		N/A		1 (FB)	
	WI-AF-FB30-MMYY		N/A		1 (FB)	
	WI-AF-FB31-MMYY		N/A		1 (FB)	
	WI-AF-FB32-MMYY		N/A		1 (FB)	
	WI-AF-FB33-MMYY		N/A		1 (FB)	
	WI-AF-FB34-MMYY		N/A		1 (FB)	
	WI-AF-FB35-MMYY		N/A		1 (FB)	
	WI-AF-FB36-MMYY		N/A		1 (FB)	
	WI-AF-FB37-MMYY		N/A		1 (FB)	
	WI-AF-FB38-MMYY		N/A		1 (FB)	

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
	WI-AF-FB39-MMYY		N/A		1 (FB)	Refer to **Worksheet #21**
	WI-AF-FB40-MMYY		N/A		1 (FB)	
	WI-AF-FB41-MMYY		N/A		1 (FB)	
	WI-AF-FB42-MMYY		N/A		1 (FB)	
	WI-AF-FB43-MMYY		N/A		1 (FB)	
	WI-AF-FB44-MMYY		N/A		1 (FB)	
	WI-AF-FB45-MMYY		N/A		1 (FB)	
	WI-AF-FB46-MMYY		N/A		1 (FB)	
	WI-AF-FB47-MMYY		N/A		1 (FB)	
	WI-AF-FB48-MMYY		N/A		1 (FB)	
	WI-AF-FB49-MMYY		N/A		1 (FB)	
	WI-AF-FB50-MMYY		N/A		1 (FB)	
	WI-AF-FB51-MMYY		N/A		1 (FB)	
	WI-AF-FB52-MMYY		N/A		1 (FB)	
	WI-AF-FB53-MMYY		N/A		1 (FB)	
	WI-AF-FB54-MMYY		N/A		1 (FB)	
	WI-AF-FB55-MMYY		N/A		1 (FB)	
	WI-AF-FB56-MMYY		N/A		1 (FB)	
	WI-AF-FB57-MMYY		N/A		1 (FB)	
WI-AF-QC	WI-AF-FB58-MMYY	QC	N/A	PFCs	1 (FB)	
	WI-AF-FB59-MMYY		N/A		1 (FB)	
	WI-AF-FB60-MMYY		N/A		1 (FB)	
	WI-AF-FB61-MMYY		N/A		1 (FB)	
	WI-AF-FB62-MMYY		N/A		1 (FB)	
	WI-AF-FB63-MMYY		N/A		1 (FB)	
	WI-AF-FB64-MMYY		N/A		1 (FB)	
	WI-AF-FB65-MMYY		N/A		1 (FB)	
	WI-AF-FB66-MMYY		N/A		1 (FB)	
	WI-AF-FB67-MMYY		N/A		1 (FB)	
	WI-AF-FB68-MMYY		N/A		1 (FB)	
	WI-AF-FB69-MMYY		N/A		1 (FB)	
	WI-AF-FB70-MMYY		N/A		1 (FB)	
	WI-AF-FB71-MMYY		N/A		1 (FB)	
	WI-AF-FB72-MMYY		N/A		1 (FB)	
	WI-AF-FB73-MMYY		N/A		1 (FB)	
	WI-AF-FB74-MMYY		N/A		1 (FB)	
	WI-AF-FB75-MMYY		N/A		1 (FB)	
	WI-AF-FB76-MMYY		N/A		1 (FB)	

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
	WI-AF-FB77-MMYY		N/A		1 (FB)	Refer to **Worksheet #21**
	WI-AF-FB78-MMYY		N/A		1 (FB)	
	WI-AF-FB79-MMYY		N/A		1 (FB)	
	WI-AF-FB80-MMYY		N/A		1 (FB)	
	WI-AF-FB81-MMYY		N/A		1 (FB)	
	WI-AF-FB82-MMYY		N/A		1 (FB)	
	WI-AF-FB83-MMYY		N/A		1 (FB)	
	WI-AF-FB84-MMYY		N/A		1 (FB)	
	WI-AF-FB85-MMYY		N/A		1 (FB)	
	WI-AF-FB86-MMYY		N/A		1 (FB)	
	WI-AF-FB87-MMYY		N/A		1 (FB)	
	WI-AF-FB88-MMYY		N/A		1 (FB)	
	WI-AF-FB89-MMYY		N/A		1 (FB)	
	WI-AF-FB90-MMYY		N/A		1 (FB)	
WI-AF-QC	WI-AF-FB91-MMYY	QC	N/A	PFCs	1 (FB)	
	WI-AF-FB92-MMYY		N/A		1 (FB)	
	WI-AF-FB93-MMYY		N/A		1 (FB)	
	WI-AF-FB94-MMYY		N/A		1 (FB)	
	WI-AF-FB95-MMYY		N/A		1 (FB)	
	WI-AF-FB96-MMYY		N/A		1 (FB)	
	WI-AF-FB97-MMYY		N/A		1 (FB)	
	WI-AF-FB98-MMYY		N/A		1 (FB)	
	WI-AF-FB99-MMYY		N/A		1 (FB)	
	WI-AF-FB100-MMYY		N/A		1 (FB)	
	WI-AF-FB101-MMYY		N/A		1 (FB)	
	WI-AF-FB102-MMYY		N/A		1 (FB)	
	WI-AF-FB103-MMYY		N/A		1 (FB)	
	WI-AF-FB104-MMYY		N/A		1 (FB)	
	WI-AF-FB105-MMYY		N/A		1 (FB)	
	WI-AF-FB106-MMYY		N/A		1 (FB)	
	WI-AF-FB107-MMYY		N/A		1 (FB)	
	WI-AF-FB108-MMYY		N/A		1 (FB)	
	WI-AF-FB109-MMYY		N/A		1 (FB)	
	WI-AF-FB110-MMYY		N/A		1 (FB)	
	WI-AF-FB111-MMYY		N/A		1 (FB)	
	WI-AF-FB112-MMYY		N/A		1 (FB)	
	WI-AF-FB113-MMYY		N/A		1 (FB)	
	WI-AF-FB114-MMYY		N/A		1 (FB)	

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
	WI-AF-FB115-MMYY		N/A		1 (FB)	Refer to **Worksheet #21**
	WI-AF-FB116-MMYY		N/A		1 (FB)	
	WI-AF-FB117-MMYY		N/A		1 (FB)	
	WI-AF-FB118-MMYY		N/A		1 (FB)	
	WI-AF-FB119-MMYY		N/A		1 (FB)	
	WI-AF-FB120-MMYY		N/A		1 (FB)	
	WI-AF-FB121-MMYY		N/A		1 (FB)	
	WI-AF-FB122-MMYY		N/A		1 (FB)	
	WI-AF-FB123-MMYY		N/A		1 (FB)	
WI-AF-QC	WI-AF-FB124-MMYY	QC	N/A	PFCs	1 (FB)	
	WI-AF-FB125-MMYY		N/A		1 (FB)	
	WI-AF-FB126-MMYY		N/A		1 (FB)	
	WI-AF-FB127-MMYY		N/A		1 (FB)	
	WI-AF-FB128-MMYY		N/A		1 (FB)	
	WI-AF-FB129-MMYY		N/A		1 (FB)	
	WI-AF-FB130-MMYY		N/A		1 (FB)	
	WI-AF-FB131-MMYY		N/A		1 (FB)	
	WI-AF-FB132-MMYY		N/A		1 (FB)	
	WI-AF-FB133-MMYY		N/A		1 (FB)	
WI-AF-QC	WI-AF-FB134-MMYY	QC	N/A	PFCs	1 (FB)	Refer to **Worksheet #21**
	WI-AF-FB135-MMYY				1 (FB)	
	WI-AF-FB136-MMYY				1 (FB)	
	WI-AF-FB137-MMYY				1 (FB)	
	WI-AF-FB138-MMYY				1 (FB)	
	WI-AF-FB139-MMYY				1 (FB)	
	WI-AF-FB140-MMYY				1 (FB)	
	WI-AF-FB141-MMYY				1 (FB)	
	WI-AF-FB142-MMYY				1 (FB)	
	WI-AF-FB143-MMYY				1 (FB)	
	WI-AF-FB144-MMYY				1 (FB)	
	WI-AF-FB145-MMYY				1 (FB)	
	WI-AF-FB146-MMYY				1 (FB)	
	WI-AF-FB147-MMYY				1 (FB)	
	WI-AF-FB148-MMYY				1 (FB)	
	WI-AF-FB149-MMYY				1 (FB)	
	WI-AF-FB150-MMYY				1 (FB)	
	WI-AF-FB151-MMYY				1 (FB)	
	WI-AF-FB152-MMYY				1 (FB)	

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Station Identification (ID)	Sample ID	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
	WI-AF-FB153-MMYY				1 (FB)	Refer to **Worksheet #21**
	WI-AF-FB154-MMYY				1 (FB)	
	WI-AF-FB155-MMYY				1 (FB)	
	WI-AF-FB156-MMYY				1 (FB)	
	WI-AF-FB157-MMYY				1 (FB)	
	WI-AF-FB158-MMYY				1 (FB)	
	WI-AF-FB159-MMYY				1 (FB)	
	WI-AF-FB160-MMYY				1 (FB)	
	WI-AF-FB161-MMYY				1 (FB)	
	WI-AF-FB162-MMYY				1 (FB)	
	WI-AF-FB163-MMYY				1 (FB)	
	WI-AF-FB164-MMYY				1 (FB)	
	WI-AF-FB165-MMYY				1 (FB)	
	WI-AF-FB166-MMYY				1 (FB)	
	WI-AF-FB167-MMYY				1 (FB)	
	WI-AF-FB168-MMYY				1 (FB)	
	WI-AF-FB169-MMYY				1 (FB)	
	WI-AF-FB170-MMYY				1 (FB)	
	WI-AF-FB171-MMYY				1 (FB)	
	WI-AF-FB172-MMYY				1 (FB)	
	WI-AF-FB173-MMYY				1 (FB)	
	WI-AF-FB174-MMYY				1 (FB)	
	WI-AF-FB175-MMYY				1 (FB)	
	WI-AF-FB176-MMYY				1 (FB)	
WI-AF-QC	WI-AF-FB177-MMYY	QC	N/A	PFCs	1 (FB)	

Notes:

[1] Drinking water samples will be collected as described in **Worksheet #14**.

SAP Worksheet #19—Analytical SOP Requirements Table

Matrix	**Analytical Group[a]**	**Analytical and Preparation Method/ SOP Reference**	**Containers**	**Sample Volume**	**Preservation Requirements**	**Maximum Holding Time[1] (preparation/ analysis)**
Drinking Water	PFCs	USEPA Method 537/ WS-DW-0004 Rev 1.4	2 × 250-milliliter (mL) high-density polyethylene bottles	250 mL	≤10°C but not frozen, Trizma (5.0 grams per liter)	14 days/ 28 days

Notes:

[a] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

SAP Worksheet #20—Field Quality Control Sample Summary Table

Samples will be collected as detailed in **Worksheets #14, #17,** and **#18** of this SAP. Field QA/QC samples will be collected as detailed in **Worksheet #12**.

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SAP Worksheet #21—Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP-001	Chain-of-Custody, rev. April 2015	CH2M	Chain-of-custody form	No	
SOP-002	Preparing Field Log Books, rev. April 2015	CH2M	PFC-free logbook and indelible pen	No	
SOP-003	Potable Water Supply Sampling rev. September 2016	CH2M	2 × 250-mL high-density polyethylene bottles	No	
SOP-004	Packaging and Shipping Procedures for Low-Concentration Samples, rev. April 2015	CH2M	Laboratory-supplied coolers	No	

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SAP Worksheet #22—Field Equipment Calibration, Maintenance, Testing, and Inspection Table

N/A

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SAP Worksheet #23—Analytical SOP References Table

Lab SOP Number	Title, Revision Date, and/or Number	Date Reviewed if not Revised	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance to QSM	Modified for Project Work (Y/N)
WS-DW-0004	*Determination of Selected Perfluorinated Alkyl Acids (PFAA) in Drinking Water by Solid Phase Extraction (SPE) and Analysis by Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS); Rev. 1.4; November 4, 2016*		Definitive	Drinking Water/ PFCs	LC/MS/MS	TestAmerica Laboratories, Inc. (TestAmerica)–West Sacramento	N	N
WS-EHS-0001	*Waste Disposal; Rev. 4.5; September 18, 2015*		N/A (Disposal)	Drinking Water/ PFCs	N/A (Disposal)	TestAmerica–West Sacramento	N	N
WS-QA-0003	*Sample Receipt and Procedures; Rev. 12.1; June 24, 2016*		N/A (Receiving)	Drinking Water/ PFCs	N/A (Receiving)	TestAmerica–West Sacramento	N	N

Notes:

[a] Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) certification is required for all definitive data. Test America–West Sacramento has DoD ELAP certification that is valid through January 31, 2017.

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SAP Worksheet #24—Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
LC/MS/MS	Tune Check	Prior to ICAL and after any mass calibration or maintenance is performed	Tuning standard must contain analytes of interest or appropriate substitute. Mass assignments of tuning standard within 0.5 amu of true value.	Retune instrument. If the tuning will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Laboratory Manager/ Analyst	WS-DW-0004
	Minimum five-point initial calibration for target analytes, lowest concentration standard at or below the RL	Initial calibration prior to sample analysis	Each calibration point for each analyte (natives and surrogates) must calculate to be within 70-130 percent, except the lowest calibration point, which must calculate to within 50 to 150 percent for natives.	Evaluate standards, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat initial calibration.		
	Peak Asymmetry Verification	With initial calibration	Calculated factor in the range of 0.8 to 1.5.	Change instrument conditions to correct, then repeat initial calibration.		
	Second-source calibration verification	Once per initial calibration, following initial calibration	All reported analytes and labeled compounds within ± 30 percent of true value.	Evaluate data. If problem (e.g., concentrated standard, plugged transfer line) found, correct, then repeat second source verification. If it still fails, then repeat initial calibration.		
	Daily calibration verification	Before sample analysis, after every 10 samples, and at the end of the sequence	If CCV ≤ LOQ, analytes must be within ± 50 percent of true value, surrogates within ± 30 percent of true value. For CCVs > LOQ, all analytes (including surrogates) must be within ± 30 percent of true value. For all CCVs, internal standards must be within ± 50 percent of true value and 70 to 140 percent of the most recent prior CCV.	Evaluate failure and impact on samples. If samples non-detect for analytes that have a high bias, report non-detect results with case narrative comment. For closing CCVs, if compounds are not identified as critical compounds of concern report results with qualifiers. For closing CCVs, if the compound is identified as a critical compound of concern, then recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take CA(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		

Notes:

amu = atomic mass unit

ICAL = initial calibration

RL = reporting limit

± = plus or minus

CCV = continuing calibration verification

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SAP Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference
LC/MS/MS	Replace columns as needed, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	TestAmerica Chemist	WS-DW-0004

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SAP Worksheet #26—Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Project Field Team, FTL/CH2M. Field SOPs are in Appendix A of this SAP.
Sample Packaging (Personnel/Organization): Project Field Team, FTL/CH2M. Field SOPs are in Appendix A of this SAP.
Coordination of Shipment (Personnel/Organization): FTL/CH2M.
Type of Shipment/Carrier: FedEx Priority Overnight
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Receiving – Test America–West Sacramento
Sample Custody and Storage (Personnel/Organization): Sample Receiving – Test America–West Sacramento
Sample Preparation (Personnel/Organization): Test America–West Sacramento
Sample Determinative Analysis (Personnel/Organization): Test America–West Sacramento
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 45 days
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 90 days
Biological Sample Storage (No. of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization): Sample Disposal – Test America–West Sacramento
Number of Days from Analysis: 45 days

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SAP Worksheet #27—Sample Custody Requirements Table

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

Samples will be collected by field team members under the supervision of the FTL. As samples are collected, they will be placed into containers and labeled. Labels will be taped to the jar to ensure they do not separate. Samples will be cushioned with packaging material and placed into coolers containing enough ice to keep the samples cooler than 10°C (but not frozen) until they are received by the laboratory.

The chain-of-custody form will be placed into the cooler in a resealable zip-top plastic bag. Coolers will be taped and shipped to the laboratories via FedEx overnight, with the air bill number indicated on the chain-of-custody form (to relinquish custody). Upon delivery, the laboratory will log each cooler and report the status of the samples to CH2M.

See **Worksheet #21** for SOPs containing sample custody guidance.

The CH2M field team will ship all environmental samples directly to the laboratory performing the analysis. This will require shipment to Test America in Sacramento, California.

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

Laboratory custody procedures can be found in the laboratory SOPs, which will be provided upon request.

Sample ID Procedures:

Sample labels will include, at a minimum, client name, site, sample ID, date/time collected, analysis group or method, preservation, and sampler's initials. The field logbook will identify the sample ID with the location and time collected and the parameters requested. The laboratory will assign each field sample a laboratory sample ID based on information in the chain-of-custody form. The laboratory will send sample log-in forms to the PC to check that sample IDs and parameters are correct.

Chain-of-Custody Procedures:

Chains of custody forms will include, at a minimum, laboratory contact information, client contact information, sample information, and relinquished by/received by information. Sample information will include sample ID. Date/time collected, number and type of containers, preservative information, analysis method, and comments. The chain-of-custody form will link location of the sample from the field logbook to the laboratory receipt of the sample. The laboratory will use the sample information to populate the Laboratory Information Management Systems database for each sample.

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SAP Worksheet #28—Laboratory QC Samples Table

Matrix: Drinking Water

Analytical Group: PFCs

Analytical Method/SOP Reference: USEPA Method 537/WS-DW-0004

QC Sample	Frequency/Number	Method/ SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria (MPC)
Method Blank	One per prep batch of 20 or fewer samples of similar matrix; or one per day, whichever comes first	No analytes detected > 1/2 LOQ or >1/10 sample concentration or >1/10 regulatory limit, whichever is greater. For common laboratory contaminants, no analytes detected >LOQ.	Verify instrument clean (evaluate calibration blank and samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Analyst/ Supervisor	Bias/ Contamination	Same as Method/ SOP QC Acceptance Limits
LCS	One per prep batch of 20 or fewer samples of similar matrix; or one per day, whichever comes first	See **Worksheet #15**	Reanalyze LCS once. If acceptable, report. Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical compounds of concern, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and narrated.		Accuracy/ Bias	
MS/MSD	One per prep batch of 20 or fewer samples of similar matrix; or one per day, whichever comes first	Method Limits of 70 to 130 percent for spikes > LOQ, and 50 to 150 percent for spikes at or below the LOQ.	Evaluate the data, and re-prepare /reanalyze the native sample and MS/MSD pair if laboratory error is indicated.		Precision/ Accuracy/ Bias	
Internal Standards (IS)	Every sample, spiked sample, standard, and method blank	Percent recovery (%R) for each IS within 50 to 150 percent of the average from the initial calibration, and within 70 to 140 percent of the most recent CCV standard.	Reanalyze affected samples. Assess matrix, dilute and/or re-extract as needed. If matrix affect demonstrated for a representative sample set, discuss with PC.		Accuracy	
Surrogates	Every samples, spiked sample, and method blank	%R within 70 to 130 percent.	Evaluate the data, and re-prepare/reanalyze if laboratory error is indicated. If matrix affect demonstrated for a representative sample set, discuss with PC.		Accuracy/ Bias	

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SAP Worksheet #29—Project Documents and Records Table

Document	Where Maintained
• Field Notebooks • Chain-of-Custody Records • Air Bills • Custody Seals • CA Forms • Electronic data deliverables (EDDs) • ID of QC Samples • Meteorological Data from Field • Sampling instrument calibration logs • Sampling Locations and Sampling Plan • Sampling Notes and Drilling Logs • Water quality parameter • Sample Receipt, Chain of Custody Forms, and Tracking Records • Standard Traceability Logs • Equipment Calibration Logs • Sample Preparation Logs • Run Logs • Equipment Maintenance, Testing, and Inspection Logs • CA Forms • Reported Field Sample Results • Reported Result for Standards, QC Checks, and QC Samples • Instrument printouts (raw data) for Field Samples, Standards, QC Checks, and QC Samples • Data Package Completeness Checklists • Sample disposal records • Extraction/Cleanup Records • Raw Data (archived per Navy CLEAN contract) • DV Reports • CA Forms • Laboratory QA Plan • Method Detection Limit Study Information	• Field data deliverables (e.g., logbooks entries, chains of custody, air bills, and EDDs) will be kept on CH2M's network server. • Field parameter data will be loaded with the analytical data into the Navy database • Analytical laboratory hard copy deliverables and DV reports will be saved on the network server and archived per the Navy CLEAN contract. • Electronic data from the laboratory will be loaded into Navy database • Following project completion, hard copy deliverables (e.g., logbooks, chain-of-custody forms) will be archived at Iron Mountain: Iron Mountain Headquarters 745 Atlantic Avenue Boston, Massachusetts 02111 (800) 899-IRON • Following project completion, hard copy deliverables including chains-of-custody and raw data will be archived at the Washington National Records Center: Washington National Records Center 4205 Suitland Road Suitland, Maryland 20746-8001 301-778-1550

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SAP Worksheet #30—Analytical Services Table

Matrix	**Analytical Group**	**Sample Locations/ID**	**Analytical Method**	**Data Package Turnaround Time**	**Laboratory/Organization**	**Backup Laboratory/ Organization [a]**
Drinking Water	PFCs	Refer to **Worksheets #18** and **#20**	USEPA Method 537	7 calendar days	Test America– West Sacramento	Vista Analytical Laboratory

Notes:

[a] Vista Analytical has been selected as the backup laboratory. It is currently in the process of getting Method 537 added to its DoD ELAP Accreditation letter.

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SAP Worksheet #31—Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing CA (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Performance Audit	One during first quarter sampling event	Internal	CH2M	PM CH2M	FTL CH2M	PM CH2M	PM CH2M
Safe Work Observation	One during each quarterly sampling event	Internal	CH2M	SSC CH2M	Field Team Member observed CH2M	HSM CH2M	SSC CH2M
Field Document Review	Daily during each quarterly sampling event	Internal	CH2M	PM or Task Manager CH2M	FTL CH2M	PM CH2M	PM CH2M

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SAP Worksheet #32—Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of CA Response Documentation	Individual(s) Receiving CA Response (name, title, organization)	Timeframe for Response
Field Performance Audit	Checklist and Written Audit Report	FTL CH2M	Within 1 day of audit	Verbal and Memorandum	FTL CH2M	Within 1 day of receipt of CA Form
Safe Behavior Observation (SBO)	SBO Form	HSM CH2M	Within 1 week of SBO	Memorandum	Field Team Member CH2M	Immediately
Field Document Review	Markup copy of field documentation	FTL CH2M	Within 1 day of review	Verbal and Memorandum	FTL CH2M	Within 1 day of receipt of markup

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SAP Worksheet #32-1—Laboratory Corrective Action Form

Person initiating CA: ____ Date: ____________________

Description of problem and when identified:

____
____
____
____
____

Cause of problem, if known or suspected:

____
____
____
____
____

Sequence of CA: (including date implemented, action planned and personnel/data affected)

____
____
____
____
____

CA implemented by: ______________________________ Date: ____________________

CA initially approved by: ______________________________ Date: ____________________

Follow-up date: ______________________________

Final CA approved by: ______________________________ Date: ____________________

Information copies to:

Anita Dodson, CH2M Navy CLEAN Program Chemist

____
____
____
____

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SAP Worksheet #32-2—Field Performance Audit Checklist

Project Responsibilities

Project No.: ____ Date: ____________________________________

Project Location: ____________________________________ Signature: ________________________________

Team Members

Yes No 1) Is the approved work plan being followed?

Comments ____

____

Yes No 2) Was a briefing held for project participants?

Comments ____

____

Yes No 3) Were additional instructions given to project participants?

Comments ____

____

Sample Collection

Yes No 1) Is there a written list of sampling locations and descriptions?

Comments ____

____

Yes No 2) Are samples collected as stated in the Master SOPs?

Comments ____

____

Yes No 3) Are samples collected in the type of containers specified in the work plan?

Comments ____

____

Yes No 4) Are samples preserved as specified in the work plan?

Comments ____

____

Yes No 5) Are the number, frequency, and type of samples collected as specified in the work plan?

Comments ____

____

Worksheet #32-2—Field Performance Audit Checklist (continued)

Yes No 6) Are QA checks performed as specified in the work plan?

Comments ____________________

____

Yes No 7) Are photographs taken and documented?

Comments ____________________

____

Document Control

Yes No 1) Have any accountable documents been lost?

Comments ____________________

____

Yes No 2) Have any accountable documents been voided?

Comments ____________________

____

Yes No 3) Have any accountable documents been disposed of?

Comments ____________________

____

Yes No 4) Are the samples identified with sample tags?

Comments ____________________

____

Yes No 5) Are blank and duplicate samples properly identified?

Comments ____________________

____

Yes No 6) Are samples listed on a chain-of-custody record?

Comments ____________________

____

Yes No 7) Is chain of custody documented and maintained?

Comments ____________________

____

SAP Worksheet #32-3—Safe Behavior Observation Form

☐ Federal or ☐ Commercial Sector (check one)		☐ Construction or ☐ Consulting (check one)	
Project Number:		Client/Program:	
Project Name:		Observer:	Date:
Position/Title of Worker Observed:		Background Information/ comments:	
Task/Observation Observed:			

- ❖ Identify and reinforce safe work practices/behaviors
- ❖ Identify and improve on at-risk practices/acts
- ❖ Identify and improve on practices, conditions, controls, and compliance that eliminate or reduce hazards
- ❖ Proactive PM support facilitates eliminating/reducing hazards (do you have what you need?)
- ❖ Positive, corrective, cooperative, collaborative feedback/recommendations

Actions & Behaviors	**Safe**	**At-Risk**	**Observations/Comments**
Current and accurate Pre-Task Planning/Briefing (for example, Project Safety Plan, Safety Training and Consulting, AHA, Pre-task Safety Plan, tailgate briefing, as needed)			**Positive Observations/Safe Work Practices:**
Properly trained/qualified/ experienced			
Tools/Equipment Available and Adequate			
Proper Use of Tools			**Questionable Activity/Unsafe Condition Observed:**
Barricades/Work Zone Control			
Housekeeping			
Communication			
Work Approach/Habits			
Attitude			
Focus/Attentiveness			**Observer’s CAs/Comments:**
Pace			
Uncomfortable/Unsafe Position			
Inconvenient/Unsafe Location			
Position/Line of Fire			
Apparel (hair, loose clothing, jewelry)			
Repetitive motion			**Observed Worker’s CAs/Comments:**
Other			

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SAP Worksheet #33—QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, and so forth)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Field Audit Report	One during sampling event	TBD	PM CH2M	Included in project files

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SAP Worksheet #34-36—Data Verification and Validation (Steps I and IIa/IIb) Process Table

Data Review Input	Description[3]	Responsible for Verification or Validation	Step I/IIa/IIb[1]	Internal/ External[2]
Field Notebooks	Field notebooks will be reviewed internally and placed into the project file for archival at project closeout.	FTL/CH2M	Step I	Internal
Chains of Custody and Shipping Forms	Chain of custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the chain of custody forms will be initialed by the reviewer, a copy of the chains of custody forms retained in the site file, and the original and remaining copies taped inside the cooler for shipment. Chain of custody forms will also be reviewed for adherence to the SAP by the PC.	FTL/CH2M PC/CH2M	Step I	Internal & External
Sample Condition upon Receipt	Any discrepancies, missing, or broken containers will be communicated to the PC in the form of laboratory logins.	PC/CH2M	Step I	External
Documentation of Laboratory Method Deviations	Laboratory Method Deviations will be discussed and approved by the PC. Documentation will be incorporated into the case narrative, which becomes part of the final hard copy data package.	PC/CH2M	Step I	External
EDDs	EDDs will be compared against hard copy laboratory results (10 percent check).	PC/CH2M	Step I	External
Case Narrative	Case narratives will be reviewed by the data validator during the DV process. This is verification that they were generated and applicable to the data packages.	Data Validator	Step I	External
Laboratory Data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	Laboratory QAO	Step I	Internal
Laboratory Data	The data will be verified for completeness by the PC. To ensure completeness, EDDs will be compared to the SAP. This is a verification that all samples were included in the laboratory data and that correct analyte lists were reported.	PC/CH2M	Step I	External
Audit Reports	Upon report completion, a copy of all audit reports will be placed in the site file. If CAs are required, a copy of the documented CA taken will be attached to the appropriate audit report in the QA site file. Periodically, and at the completion of site work, site file audit reports and CA forms will be reviewed internally to ensure that all appropriate CAs have been taken and that CA reports are attached. If CAs have not been taken, the site manager will be notified to ensure action is taken.	PM/CH2M PC/CH2M	Step I	Internal
CA Reports	CA reports will be reviewed by the PC or PM and placed into the project file for archival at project closeout.	PM/CH2M PC/CH2M	Step I	External
Laboratory Methods	During the pre-validation check, ensure that the laboratory analyzed samples using the correct methods specified in the SAP. If methods other than those specified in the SAP were used, the reason will be determined and documented.	PC/CH2M	Step IIa	External
Target Compound List and Target Analyte list	During the pre-validation check, ensure that the laboratory reported all analytes from each analysis group as per **Worksheet #15**. If the target compound list is not correct, then it must be corrected prior to sending the data for validation. Once the checks are complete, the project manager is notified via email	PC/CH2M	Step IIa	External
RLs	During the pre-validation check, the laboratory RLs will be compared to those listed in the project SAP. If RLs were not met, the laboratory will be contacted and asked to provide an explanation, which will then be discussed in the associated project report. Often times the cause for minor laboratory RL deviation from those presented in the SAP is due to the quarterly update of laboratory LOD.	PC/CH2M	Step IIb	External
Laboratory SOPs	Ensure that approved analytical laboratory SOPs were followed. Any such discrepancies will be discussed first in the data validation narrative and will be included in the associated project report.	Laboratory QAO	Step IIa	Internal
Sample Chronology	Holding times from collection to extraction or analysis and from extraction to analysis will be considered during the DV process.	Data Validator	Step IIa and IIb	External
Raw Data	Ten percent review of raw data to confirm laboratory calculations. For a recalculated result, the data validator attempts to re-create the reported numerical value. The laboratory is asked for clarification if a discrepancy is identified, which cannot reasonably be attributed to rounding. In general, this is outside 5 percent difference.	Data Validator	Step IIa	External

SAP Worksheet #34-36—Data Verification and Validation (Steps I and IIa/IIb) Process Table (continued)

Data Review Input	Description[c]	Responsible for Verification or Validation	Step I/IIa/IIb[a]	Internal/ External[b]
Onsite Screening	All non-analytical field data will be reviewed against SAP requirements for completeness and accuracy based on the field calibration records.	FTL/CH2M	Step IIb	Internal
Documentation of Method QC Results	Establish that all required QC samples were run and met limits.	Data Validator	Step IIa	External
Documentation of Field QC Sample Results	Establish that all required QC samples were run and met limits, and will be discussed in the associated project report.	PC/CH2M	Step IIa	Internal
DoD ELAP Evaluation	Ensure that each laboratory is DoD ELAP certified for the analyses they are to perform. Ensure evaluation timeframe does not expire.	PC/CH2M	Step I	External
Analytical data for PFCs analyzed for in groundwater and drinking water	Analytical methods and laboratory SOPs as presented in this SAP will be used to evaluate compliance against QA/QC criteria. Should adherence to QA/QC criteria yield deficiencies, data may be qualified. The data qualifiers used are those presented in National Functional Guidelines for Superfund Organic Data Review (USEPA, 2014) may be used for DV, and the specific qualifiers listed therein may be applied to data should non-conformances against the QA/QC criteria as presented in this SAP be identified.	Data Validator	Step IIa and IIb	External

Notes:

[a] Verification (Step I) is a completeness check that is performed before the data review process continues to determine whether the required information (complete data package) is available for further review. Validation (Step IIa) is a review that the data generated is in compliance with analytical methods, procedures, and contracts. Validation (Step IIb) is a comparison of generated data against measurement performance criteria in the SAP (both sampling and analytical).

[b] Internal or external is in relation to the data generator.

[c] Should CH2M find discrepancies during the verification or validation procedures above, an email documenting the issue will be circulated to the internal project team, and a Corrections to File Memo will be prepared identifying the issues and the corrective action needed. This memo will be sent to the laboratory, or applicable party, and maintained in the project file.

SAP Worksheet #37—Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

- Non-detected site contaminants will be evaluated to ensure that project required quantitation limits in **Worksheet #15** were achieved. If PQLs were achieved and the verification and validation steps yielded acceptable data, then the data are considered usable.
- During verification and validation steps, data may be qualified as estimated with the following qualifiers: J, J+, J-, or UJ. The qualifiers represent minor QC deficiencies, which will not affect the usability of the data. When major QC deficiencies are encountered, data will be qualified with an R and in most cases is not considered usable for project decisions.
  - J = Analyte present. Reported value may or may not be accurate or precise.
  - J+ = Analyte present. Reported value is estimated and may be biased high.
  - J- = Analyte present. Reported value is estimated and may be biased low.
  - UJ = Analyte not detected. Associated non-detect value may be inaccurate or imprecise.
  - R = Rejected result. Result not reliable.
- The following additional qualifiers may be given by the validator:
  - N = Tentative ID. Consider Present. Special methods may be needed to confirm its presence or absence in future sampling efforts.
  - NJ = Qualitative ID questionable due to poor resolution. Presumptively present at approximate quantity.
  - U = Not Detected.
- Analytical data will be checked to ensure the values and any qualifiers are appropriately transferred to the electronic database. The checks include comparison of hard copy data and qualifiers to the EDD. Once the data have been uploaded into the electronic database, another check will be performed to ensure all results were loaded accurately.
- Field and laboratory precision will be compared as RPD between the two results.
- Deviations from the SAP will be reviewed to assess whether CA is warranted and to assess impacts to achievement of project objectives.

Describe the evaluative procedures used to assess overall measurement error associated with the project.

- To assess whether a sufficient quantity of acceptable data is available for decision making, the data will be compared to the 95 percent completeness goal and reconciled with MPC following validation and review of DQI.
- If significant biases are detected with laboratory QA/QC samples, they will be evaluated to assess impact on decision making. Low biases will be described in greater detail as they represent a possible inability to detect compounds that may be present at the site.
- If significant deviations are noted between laboratory and field precision, the cause will be further evaluated to assess impact on decision making.

SAP Worksheet #37—Usability Assessment (continued)

Describe the documentation that will be generated during the usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The following will be prepared by CH2M and presented to and submitted to the Navy and Base for review and decisions on the path forward for the site:

- Data tables will be produced to reflect detected and non-detected site analytes. Data qualifiers will be reflected in the tables and discussed in the data quality evaluation, and will be provided in a technical memorandum.

Identify the personnel responsible for performing the usability assessment.

The CH2M Team, including the PM and PC, will review the data and present to the Navy and Base for review and approval of usability.

References

CH2M HILL, Inc. (CH2M). 2016. *Technical Memorandum, Results of Desktop Evaluation to Verify Off-Base Drinking Water Sources, Naval Facilities Engineering Command, Northwest*. September.

U.S. Environmental Protection Agency (USEPA). 2002. *Guidance for Quality Assurance Project Plans, USEPA QA/G-5.* EPA/240/R-02/009. December.

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U.S. Department of the Navy (Navy). 1994. *Final Remedial Investigation Report for Operable Unit 3, Naval Air Station Whidbey Island. Prepared for Engineering Field Activity Northwest, Naval Facilities Engineering Command by URS Consultants Under Contract No. N62474-89-D-9295, CTO 0074*. January.

U.S. Department of the Navy (Navy). 2013. *Annual 2012-2-13 Groundwater Long-Term Monitoring Report for Operable Unit 1 Area 6, and Operable Unit 5 Area 31, NAS Whidbey Island, Whidbey Island, Washington*. Prepared for Naval Facilities Engineering Command Northwest by Sealaska Environmental Services under Contract No. N44255-09-D-4005, CTO 56. August 1.

U.S. Department of the Navy (Navy). 2016. *Summary Report, Groundwater Sampling for Perfluorinated Compounds, Hangar 5 and Areas 16 and 31, NAS Whidbey, Oak Harbor, Washington*. Prepared for Naval Facilities Engineering Command Northwest by MMEC Group under Contract. No. N62473-12-D-2012, CTP JP02. April 14.

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Figures

Figure 1
Base Location Map
Naval Air Station Whidbey Island
Oak Harbor, WA

For Official Use Only

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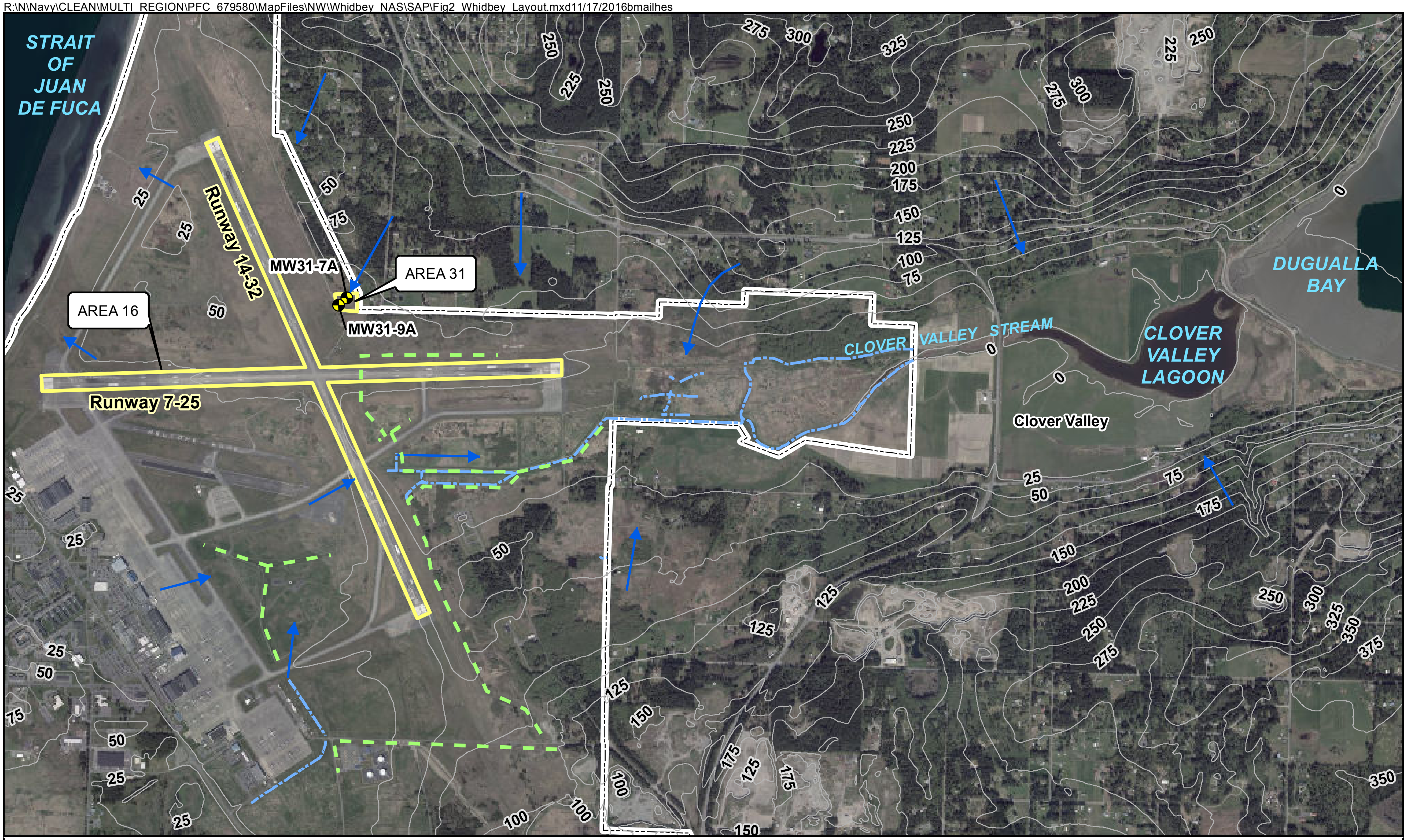


Legend

- ● Wells Sampled
- ▭ Site Boundary (suspected source)
- ⬚ Base Boundary
- → Estimated Groundwater Flow Direction
- - · Surface Water
- - - Drainage Ditch (Part of Area 16)
- — Elevation Contour - 25 ft Interval

N

0 0.2 0.4 Miles

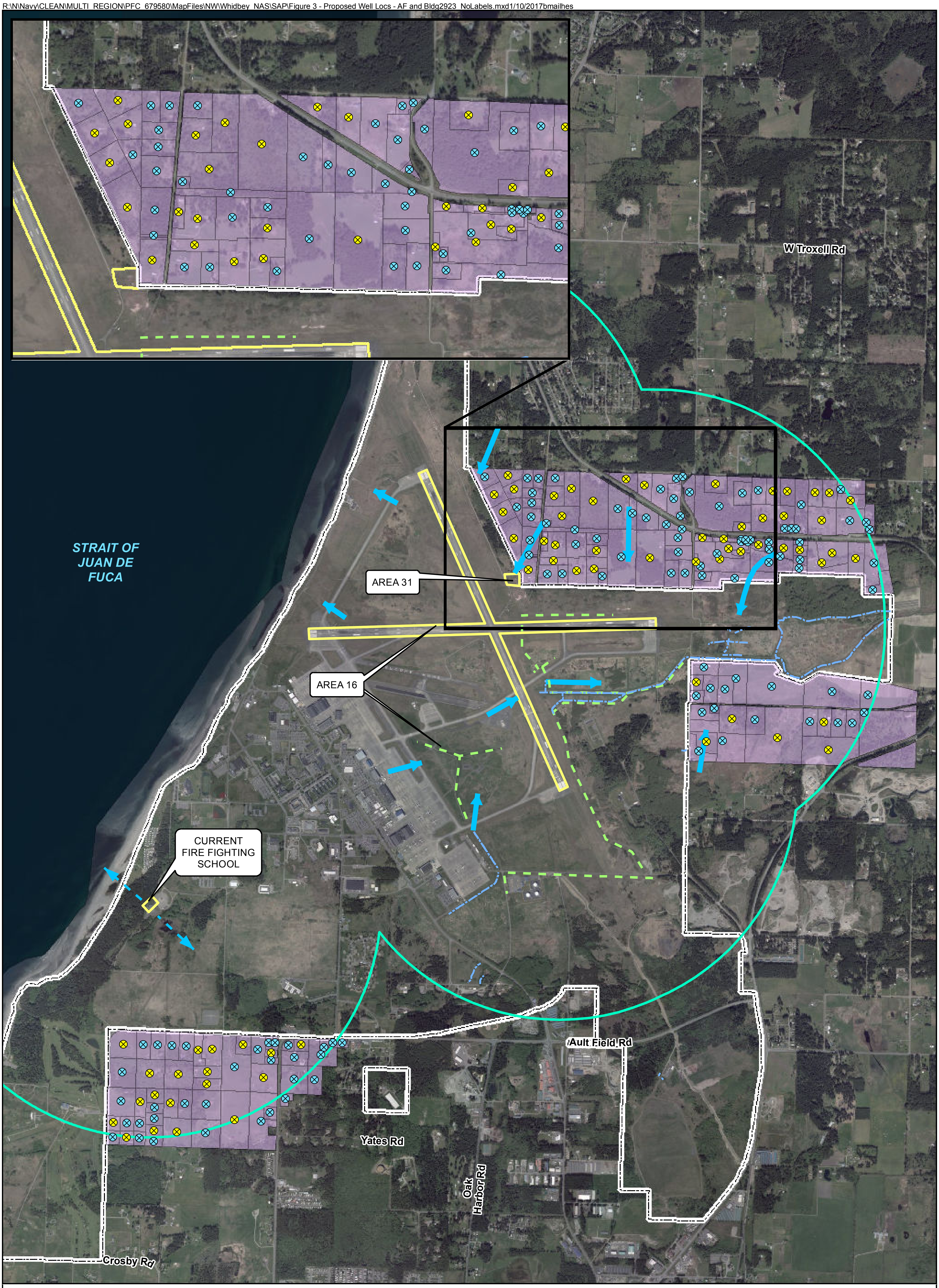
1 inch = 0.4 mile

Imagery Source: Esri

Figure 2
Areas 16 and 31 Layout Map
Naval Air Station Whidbey Island
Oak Harbor, WA

For Official Use Only

R:\N\Navy\CLEAN\MULTI REGION\PFC 679580\MapFiles\NW\Whidbey NAS\SAP\Figure 3 - Proposed Well Locs - AF and Bldg2923 NoLabels.mxd1/10/2017bmailhes



Legend

- ⊗ Proposed Sample Location
- ⊗ Proposed Sample Location (well confirmed)
- -·- Surface Water
- - - Drainage Ditch
- ▭ Ault Field 1-mile zone
- ▭ Suspected Source Area
- ▨ Phase 1 Sample Areas
- ⬚ Base Boundary
- ➡ Assumed Regional Groundwater Flow Direction
- - -➡ Inferred Regional Groundwater Flow Direction

1 inch = 0.4 mile

Imagery Source: Esri

Figure 3
Proposed Sample Location Map
Areas 16, 31, and Building 2923
Naval Air Station Whidbey Island
Oak Harbor, Washington

Appendix A
Field Standard Operating Procedures – CH2M

STANDARD OPERATING PROCEDURE

Chain-of-Custody

I Purpose

The purpose of this SOP is to provide information on chain-of-custody procedures to be used under the CLEAN Program.

II Scope

This procedure describes the steps necessary for transferring samples through the use of Chain-of-Custody Records. A Chain-of-Custody Record is required, without exception, for the tracking and recording of samples collected for on-site or off-site analysis (chemical or geotechnical) during program activities (except wellhead samples taken for measurement of field parameters). Use of the Chain-of-Custody Record Form creates an accurate written record that can be used to trace the possession and handling of the sample from the moment of its collection through analysis. This procedure identifies the necessary custody records and describes their completion. This procedure does not take precedence over region specific or site-specific requirements for chain-of-custody.

III Definitions

Chain-of-Custody Record Form - A Chain-of-Custody Record Form is a printed two-part form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to another custodian. One copy of the form must be retained in the project file.

Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under one’s custody if:

- It is in one’s actual possession.
- It is in one’s view, after being in one’s physical possession.
- It was in one’s physical possession and then he/she locked it up to prevent tampering.
- It is in a designated and identified secure area.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

IV. Procedures

The term "chain-of-custody" refers to procedures which ensure that evidence presented in a court of law is valid. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the courtroom, as well as providing security for the evidence as it is moved and/or passed from the custody of one individual to another.

Chain-of-custody procedures, recordkeeping, and documentation are an important part of the management control of samples. Regulatory agencies must be able to provide the chain-of-possession and custody of any samples that are offered for evidence, or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

A. Sample Identification

The method of identification of a sample depends on the type of measurement or analysis performed. When *in situ* measurements are made, the data are recorded directly in bound logbooks or other field data records with identifying information.

Information which shall be recorded in the field logbook, when in-situ measurements or samples for laboratory analysis are collected, includes:

- Field Sampler(s),
- Contract Task Order (CTO) Number,
- Project Sample Number,
- Sample location or sampling station number,
- Date and time of sample collection and/or measurement,
- Field observations,
- Equipment used to collect samples and measurements, and
- Calibration data for equipment used

Measurements and observations shall be recorded using waterproof ink.

B. Sample Label

Samples, other than for *in situ* measurements, are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often divided into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the Sampling and Analysis Plan. Each sample container is identified by a sample label (see Attachment A). Sample labels are provided, along with sample containers, by the analytical laboratory. The information recorded on the sample label includes:

- Project - CTO Number.

- Station Location - The unique sample number identifying this sample.

- Date - A six-digit number indicating the day, month, and year of sample collection (e.g., 08/21/12).

- Time - A four-digit number indicating the 24-hour time of collection (for example: 0954 is 9:54 a.m., and 1629 is 4:29 p.m.).
- Medium - Water, soil, sediment, sludge, waste, etc.
- Sample Type - Grab or composite.
- Preservation - Type and quantity of preservation added.
- Analysis - VOA, BNAs, PCBs, pesticides, metals, cyanide, other.
- Sampled By - Printed name of the sampler.
- Remarks - Any pertinent additional information.

Using only the work assignment number of the sample label maintains the anonymity of sites. This may be necessary, even to the extent of preventing the laboratory performing the analysis from knowing the identity of the site (e.g., if the laboratory is part of an organization that has performed previous work on the site). The field team should always follow the sample ID system prepared by the project EIS and reviewed by the Project Manager.

C. Chain-of-Custody Procedures

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

D. Field Custody Procedures

- Samples are collected as described in the site Sampling and Analysis Plan. Care must be taken to record precisely the sample location and to ensure that the sample number on the label matches the Chain-of-Custody Record exactly.
- A Chain-of-Custody Record will be prepared for each individual cooler shipped and will include ***only*** the samples contained within that particular cooler. The Chain-of-Custody Record for that cooler will then be sealed in a zip-log bag and placed in the cooler prior to sealing. This ensures that the laboratory properly attributes trip blanks with the correct cooler and allows for easier tracking should a cooler become lost during transit.
- The person undertaking the actual sampling in the field is responsible for the care and custody of the samples collected until they are properly transferred or dispatched.
- When photographs are taken of the sampling as part of the documentation procedure, the name of the photographer, date, time, site location, and site description are entered sequentially in the site logbook as photos are taken. Once downloaded to the server or developed, the electronic files or photographic prints shall be serially numbered, corresponding to the logbook descriptions; photographic prints will be stored in the project files. To identify sample

locations in photographs, an easily read sign with the appropriate sample location number should be included.

- Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions (e.g., a logbook notation would explain that a pencil was used to fill out the sample label if the pen would not function in freezing weather.)

E. Transfer of Custody and Shipment

Samples are accompanied by a Chain-of-Custody Record Form. **A Chain-of-Custody Record Form must be completed for each cooler and should include only the samples contained within that cooler**. A Chain-of-Custody Record Form example is shown in Attachment B. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the Record. This Record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. The Chain-of-Custody Record is filled out as given below:

- Enter header information (CTO number, samplers, and project name).

- Enter sample specific information (sample number, media, sample analysis required and analytical method grab or composite, number and type of sample containers, and date/time sample was collected).

- Sign, date, and enter the time under “Relinquished by” entry.

- Have the person receiving the sample sign the “Received by” entry. If shipping samples by a common carrier, print the carrier to be used in this space (i.e., Federal Express).

- If a carrier is used, enter the airbill number under “Remarks,” in the bottom right corner;

- Place the original (top, signed copy) of the Chain-of-Custody Record Form in a plastic zipper-type bag or other appropriate sample-shipping package. Retain the copy with field records.

- Sign and date the custody seal, a 1-inch by 3-inch white paper label with black lettering and an adhesive backing. Attachment C is an example of a custody seal. The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field. Custody seals shall be provided by the analytical laboratory.

- Place the seal across the shipping container opening (front and back) so that it would be broken if the container were to be opened.

- Complete other carrier-required shipping papers.

The custody record is completed using waterproof ink. Any corrections are made by drawing a line through and initialing and dating the change, then entering the correct information. Erasures are not permitted.

Common carriers will usually not accept responsibility for handling Chain-of-Custody Record Forms; this necessitates packing the record in the shipping container (enclosed with other documentation in a plastic zipper-type bag). As long as custody forms are sealed inside the shipping container and the custody seals are intact, commercial carriers are not required to sign the custody form.

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Record, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis.

V Quality Assurance Records

Once samples have been packaged and shipped, the Chain-of-Custody copy and airbill receipt become part of the quality assurance record.

VI Attachments

A. Sample Label
B. Chain of Custody Form
C. Custody Seal

VII References

USEPA. *User's Guide to the Contract Laboratory Program*. Office of Emergency and Remedial Response, Washington, D.C. (EPA/540/P-91/002), January 1991.

Attachment A

Example Sample Label

QAL

Quality Analytical Laboratories, Inc.
2567 Fairlane Drive
Montgomery, Alabama 36116
PH. (334)271-2440

Client ____________________
Sample No. ____________________
Location ____________________

Analysis ____________________

Preservative HCL
Date ____________ By ____________

CEIMIC
CORPORATION

10 Dean Knauss Drive, Narragansett, R.I. 02882 • (401) 782-8900

SITE NAME	**DATE**
ANALYSIS	**TIME**
	PRESERVATIVE

SAMPLE TYPE

☐ Grab ☐ Composite ☐ Other ____________

COLLECTED BY:

Attachment B

Example Chain-of-Custody Record

CH2M H
APPLIED S ENCES LABORATORY

CHAIN OF CUSTODY CORD AND AGREEMENT TO PERFORM SERVICES

CH2M Hill Project #
☐☐☐☐☐☐☐☐.☐☐.☐☐

Purchase Order #

Project Name

Company Name CH2M HILL Office

Project Manager & Phone #
Mr. [ ]
Ms. [ ]
Dr. [ ]

Report Copy to:

Requested Completion Date:

Sampling Requirements
SDWA ☐ NPDES ☐ RCRA ☐ OTHER ____

Sample Disposal:
Dispose ☐ Return ☐

LAB TEST CODES

ANALYSES REQUESTED

SHADED AREA– FOR LAB USE ONLY

Lab 1 #	Lab 2 #
Quote #	Kit Request #
Project #	
No. of Samples	Page of
Login	LIMS Ver

Sampling		Type		Matrix			CLIENT SAMPLE ID (9 CHARACTERS)	# OF CONTAINERS											REMARKS	LAB 1 ID	LAB 2 ID
Date	Time	COMP	GRAB	WATER	SOIL	AIR															

Sampled By & Title (Please sign and print name)	Date/Time	Relinquished By (Please sign and print name)	Date/Time	QC Level: 1 2 3 Other: ____
Received By (Please sign and print name)	Date/Time	Relinquished By (Please sign and print name)	Date/Time	COC Rec / ICE
Received By (Please sign and print name)	Date/Time	Relinquished By (Please sign and print name)	Date/Time	Ana Req / TEMP Cust Seal / Ph
Received By (Please sign and print name)	Date/Time	Shipped Via UPS BUS Fed-Ex Hand Other ____	Shipping #	
Work Authorized By (Please sign and print name)	Remarks			

Instructions and Agreement Provisions on Reverse Side

DISTRIBUTION: Original - LAB, Yellow - LAB, Pink - Client
REV 3/94 FORM 340

Attachment C

Example Custody Seal

CH2M HILL

CUSTODY SEAL

Date

Signature

STANDARD OPERATING PROCEDURE

Preparing Field Log Books

I. Purpose

This SOP provides general guidelines for entering field data into log books during site investigation and remediation activities.

II. Scope

This is a general description of data requirements and format for field log books. Log books are needed to properly document all field activities in support of data evaluation and possible legal activities.

III. Equipment and Materials

- Log book
- Indelible pen

IV. Procedures and Guidelines

Properly completed field log books are a requirement for much of the work we perform under the Navy CLEAN contract. Log books are legal documents and, as such, must be prepared following specific procedures and must contain required information to ensure their integrity and legitimacy. This SOP describes the basic requirements for field log book entries.

A. PROCEDURES FOR COMPLETING FIELD LOG BOOKS

1. Field notes commonly are kept in bound, hard-cover logbooks used by surveyors and produced, for example, by Peninsular Publishing Company and Sesco, Inc. Pages should be water-resistant and notes should be taken only with water-proof, non-erasable permanent ink, such as that provided in Sanford Sharpie® permanent markers.

2. On the inside cover of the log book the following information should be included:

   - Company name and address
   - Log-holders name if log book was assigned specifically to that person
   - Activity or location

- Project name
- Project manager's name
- Phone numbers of the company, supervisors, emergency response, etc.

3. All lines of all pages should be used to prevent later additions of text, which could later be questioned. Any line not used should be marked through with a line and initialed and dated. Any pages not used should be marked through with a line, the author's initials, the date, and the note "Intentionally Left Blank."

4. If errors are made in the log book, cross a single line through the error and enter the correct information. All corrections shall be initialed and dated by the personnel performing the correction. If possible, all corrections should be made by the individual who made the error.

5. Daily entries will be made chronologically.

6. Information will be recorded directly in the field log book during the work activity. Information will not be written on a separate sheet and then later transcribed into the log book.

7. Each page of the log book will have the date of the work and the note takers initials.

8. The final page of each day's notes will include the note-takers signature as well as the date.

9. Only information relevant to the subject project will be added to the log book.

10. The field notes will be copied and the copies sent to the Project Manager or designee in a timely manner (at least by the end of each week of work being performed).

B. INFORMATION TO BE INCLUDED IN FIELD LOG BOOKS

1. Entries into the log book should be as detailed and descriptive as possible so that a particular situation can be recalled without reliance on the collector's memory. Entries must be legible and complete.

2. General project information will be recorded at the beginning of each field project. This will include the project title, the project number, and project staff.

3. Scope: Describe the general scope of work to be performed each day.

4. Weather: Record the weather conditions and any significant changes in the weather during the day.

5. Tail Gate Safety Meetings: Record time and location of meeting, who was present, topics discussed, issues/problems/concerns identified,

and corrective actions or adjustments made to address concerns/ problems, and other pertinent information.

6. Standard Health and Safety Procedures: Record level of personal protection being used (e.g., level D PPE), record air monitoring data on a regular basis and note where data were recording (e.g., reading in borehole, reading in breathing zone, etc). Also record other required health and safety procedures as specified in the project specific health and safety plan.

7. Instrument Calibration; Record calibration information for each piece of health and safety and field equipment.

8. Personnel: Record names of all personnel present during field activities and list their roles and their affiliation. Record when personnel and visitors enter and leave a project site and their level of personal protection.

9. Communications: Record communications with project manager, subcontractors, regulators, facility personnel, and others that impact performance of the project.

10. Time: Keep a running time log explaining field activities as they occur chronologically throughout the day.

11. Deviations from the Work Plan: Record any deviations from the work plan and document why these were required and any communications authorizing these deviations.

12. Heath and Safety Incidents: Record any health and safety incidents and immediately report any incidents to the Project Manager.

13. Subcontractor Information: Record name of company, record names and roles of subcontractor personnel, list type of equipment being used and general scope of work. List times of starting and stopping work and quantities of consumable equipment used if it is to be billed to the project.

14. Problems and Corrective Actions: Clearly describe any problems encountered during the field work and the corrective actions taken to address these problems.

15. Technical and Project Information: Describe the details of the work being performed. The technical information recorded will vary significantly between projects. The project work plan will describe the specific activities to be performed and may also list requirements for note taking. Discuss note-taking expectations with the Project Manager prior to beginning the field work.

16. Any conditions that might adversely affect the work or any data obtained (e.g., nearby construction that might have introduced excessive amounts of dust into the air).

17. Sampling Information; Specific information that will be relevant to most sampling jobs includes the following:

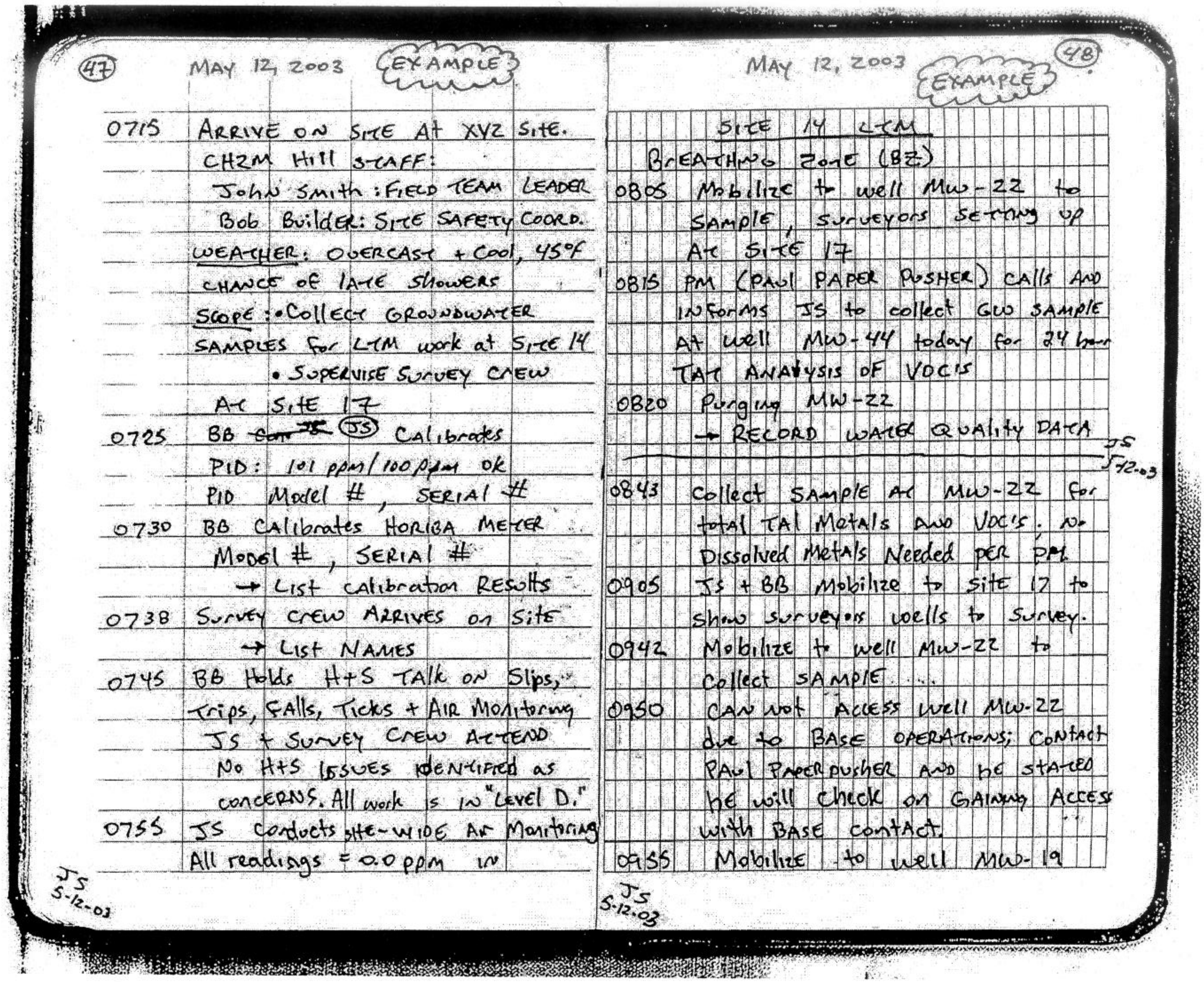
    - Description of the general sampling area – site name, buildings and streets in the area, etc.
    - Station/Location identifier
    - Description of the sample location – estimate location in comparison to two fixed points – draw a diagram in the field log book indicating sample location relative to these fixed points – include distances in feet.
    - Sample matrix and type
    - Sample date and time
    - Sample identifier
    - Draw a box around the sample ID so that it stands out in the field notes
    - Information on how the sample was collected – distinguish between "grab," "composite," and "discrete" samples
    - Number and type of sample containers collected
    - Record of any field measurements taken (i.e. pH, turbidity, dissolved oxygen, and temperature, and conductivity)
    - Parameters to be analyzed for, if appropriate
    - Descriptions of soil samples and drilling cuttings can be entered in depth sequence, along with PID readings and other observations. Include any unusual appearances of the samples.

C. SUGGESTED FORMAT FOR RECORDING FIELD DATA

1. Use the left side border to record times and the remainder of the page to record information (see attached example).
2. Use tables to record sampling information and field data from multiple samples.
3. Sketch sampling locations and other pertinent information.
4. Sketch well construction diagrams.

V. Attachments

Example field notes.



㊼ MAY 12, 2003 EXAMPLE

0715 ARRIVE ON SITE At XYZ SitE.
CH2M Hill STAFF:
John Smith: FIELD TEAM LEADER
Bob Builder: SITE SAFETY COORD.
WEATHER: OVERCAST + Cool, 45°F
CHANCE of lATE ShowERS
SCOPE: • Collect GROUNDWATER
SAMPLES for LTM work at SitE 14
• SUPERVISE SURVEY CREW
At SitE 17
0725 BB ~~Con JS~~ JS Calibrates
PID: 101 ppm/100 ppm ok
PID Model #, SERIAL #
0730 BB CAlibrates HORIBA METER
Model #, SERIAL #
→ List calibration Results
0738 Survey crew ARRIVES on SitE
→ List NAMES
0745 BB Holds H+S TAlk on Slips,
Trips, FAlls, Ticks + AIR Monitoring
JS + SURVEY CREW ATTEND
No H+S ISSUES IDENTIFIED as
CONCERNS. All work is in "Level D."
0755 JS Conducts SitE-WIDE Air Monitoring
All readings = 0.0 ppm in

JS
5-12-03

㊽ MAY 12, 2003 EXAMPLE

SITE 14 LTM
BrEATHING ZONE (BZ)
0805 Mobilize to well MW-22 to
SAMPlE, surveyors setting up
At SitE 17
0815 PM (PAUL PAPER PUSHER) CAlls AND
INFORMS JS to collect GW SAMPlE
At well MW-44 today for 24 hour
TAT ANALYSIS OF VOC's
0820 Purging MW-22
→ RECORD WATER QUALity DATA

JS
5-12-03

0843 Collect SAMPlE At MW-22 for
total TAl Metals AND VOC's. No
Dissolved Metals Needed PER PM
0905 JS + BB Mobilize to SitE 17 to
show surveyors wells to survey.
0942 Mobilize to well MW-22 to
collect SAMPlE ...
0950 CAN not ACCESS well MW-22
due to BASE OPERATIONS; CONTACT
PAUL PAPERPUSHER AND HE STATED
HE will check on GAINING ACCESS
with BASE contAct.
0955 Mobilize to well MW-19

JS
5-12-03

STANDARD OPERATING PROCEDURE – Navy CLEAN PROGRAM

Drinking Water Sampling when Analyzing for Per- and Polyfluoroalklyl Substances (PFASs)

I. Purpose and Scope

This SOP provides guidelines for drinking water sample collection for samples that will be analyzed for Per- and Polyfluoroalklyl Substances (PFASs), aka perfluorinated compounds (PFCs), including perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA) via EPA Method 537 (not modified).

Standard techniques for collecting representative samples are summarized. These procedures are specific to the Navy Comprehensive Long-term Environmental Action Navy (CLEAN) Program under Contract N62470-16-D-9000.

II. Equipment and Materials

Equipment and Materials Required

- Drinking water sample containers (polypropylene bottle with polypropylene screw cap and Trizma preservative)
- Laboratory pre-filled polypropylene bottles containing field reagent blank water and Trizma preservative
- Field Reagent Blank sample containers (polypropylene bottle with polypropylene screw cap and no preservative)
- Shipping supplies (labels, coolers, and ice)
- Loose leaf paper without waterproof coating
- Clip board
- Pen (not Sharpie)
- Nitrile or latex gloves

Equipment and Materials to Avoid During Sampling

Equipment and materials used to collect drinking water samples should not contain any fluorinated compounds, including polytetrafluoroethylene (PTFE), Teflon® or synthetic rubber with fluoropolymer elastomers (e.g., Viton®).

Specifically, the following material should be avoided during sampling:

- Gore-Tex brand or similar high-performance outdoor clothing, clothing treated with ScotchGuard® brand or similar water repellent, fluoropolymer-coated Tyvek®, wrinkle-resistant fabrics, and fire resistant clothing with fluorochemical treatment or anything advertised as water repellant.

- Weather-proof log books with fluorochemical coatings

The sample collection area should be clear of the following items:

- Pre-packaged food wrappers (e.g., fast food sandwich wrappers, pizza boxes, etc.)
- Microwave popcorn bags
- Blue ice containers
- Aluminum foil
- Kim-Wipes
- Sunscreen, insect repellant and other personal hygiene products that may contain PFAS

Sample bottles should be polypropylene in accordance with Method 537. PFASs have a tendency to adhere to glass surfaces. Contact the project manager (PM) if the lab sends glass bottles. Sample vials should not have PTFE/Teflon® lined bottles or caps.

III. Procedures and Guidelines

A. Setup

1. Obtain well construction information from homeowner, if available, in accordance with homeowner questionnaire developed for your project.

2. Record personnel onsite, address, homeowner name, and designated sample ID in the field logbook. Sample IDs should not contain identifying information about the property location due to potential privacy issues, so be sure both address and designated ID are carefully recorded for tracking. Sample IDs and addresses on the sample bottles and in the sample notebook must be checked by both field team members and the address in the field notebook should be confirmed with the homeowner or resident.

3. As feasible, select a sampling collection point prior to any treatment system installed by the homeowner. For example, if the homeowner has a point of use reverse osmosis or granular activated carbon filter in their kitchen sink, collect at the bathroom sink. If there is a point of entry filtration system, ask if there is a sampling port between the well and the system. If there is no way to bypass the existing treatment system without disconnecting pump components or potentially damaging the system, collect a treated sample and note that the sample was collected post-treatment. Avoid collecting samples through hoses. Instead, disconnect the hose and sample from the spigot if an outside collection station is selected.

3. Wash hands before sampling with dish detergent and don nitrile gloves.

4. Open the tap and allow the system to flush for three to five minutes. Do not open bottles until you are ready to sample.

B. Sample Collection

Once flushing is complete, samples can be collected.

The steps to be followed for sample collection are as follows:

1. Turn the tap off briefly. Remove the cap from the sample bottle. Position the sample bottle under the tap and turn the tap on.
2. Fill the bottle, taking care not to flush out the sample preservative. Samples do not need to be collected headspace free.
3. After collecting the sample, cap the bottle and agitate by hand until the preservative is dissolved.
4. Pack the sample on ice immediately for shipment to the offsite laboratory.

C. Field Reagent Blank Collection

A field reagent blank is required at each drinking water sampling location and is to be collected immediately following collection of the drinking water sample. The steps to complete collection of the field reagent blank are as follows:

1. A preserved field reagent blank for each sample location will be provided by the laboratory along with empty bottles for the field reagent blanks. While still at the drinking water sample collection point, open the preserved field reagent blank water bottle and an empty unpreserved sample bottle.
2. Pour the preserved reagent blank water from the preserved bottle into the unpreserved blank container.
3. Be sure the field reagent blank bottle is labeled and will be labeled and packed in the same cooler as the associated drinking water sampling for shipment to the offsite laboratory.

V. References

United States Environmental Protection Agency (USEPA), 2009. *Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)*. September

STANDARD OPERATING PROCEDURE

Packaging and Shipping Procedures for Low-Concentration Samples

I. Purpose and Scope

The purpose of this guideline is to describe the packaging and shipping of low-concentration samples of various media to a laboratory for analysis.

II. Scope

The guideline only discusses the packaging and shipping of samples that are anticipated to have low concentrations of chemical constituents. Whether or not samples should be classified as low-concentration or otherwise will depend upon the site history, observation of the samples in the field, odor, and photoionization-detector readings.

If the site is known to have produced high-concentration samples in the past or the sampler suspects that high concentrations of contaminants might be present in the samples, then the sampler should conservatively assume that the samples cannot be classified as low-concentration. Samples that are anticipated to have medium to high concentrations of constituents should be packaged and shipped accordingly.

If warranted, procedures for dangerous-goods shipping may be implemented. Dangerous goods and hazardous materials pose an unreasonable risk to health, safety, or property during transportation without special handling. As a result, only employees who are trained under CH2M HILL Dangerous Goods Shipping course may ship or transport dangerous goods. Employees should utilize the HAZMAT ShipRight tool on the Virtual Office and/or contact a designated CH2M HILL HazMat advisor with questions.

III. Equipment and Materials

- Coolers
- Clear tape
- “This Side Up” labels
- “Fragile” labels
- Vermiculite
- Ziplock bags or bubble wrap
- Ice
- Chain-of-Custody form (completed)
- Custody seals

IV. Procedures and Guidelines

Low-Concentration Samples

A. Prepare coolers for shipment:

- Tape drains shut.
- Affix "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.
- Place mailing label with laboratory address on top of coolers.
- Fill bottom of coolers with about 3 inches of vermiculite or absorbent pads.

B. Arrange decontaminated sample containers in groups by sample number. Consolidate VOC samples into one cooler to minimize the need for trip blanks.

C. Affix appropriate adhesive sample labels to each container. Protect with clear label protection tape.

D. Seal each sample bottle within a separate ziplock plastic bag or bubble wrap, if available. Tape the bag around bottle. Sample label should be visible through the bag.

E. Arrange sample bottles in coolers so that they do not touch.

F. If ice is required to preserve the samples, cubes should be repackaged in zip-lock bags and placed on and around the containers.

G. Fill remaining spaces with vermiculite or absorbent pads.

H. Complete and sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express or the courier.

J Close lid and latch.

K. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.

L. Tape cooler shut on both ends, making several complete revolutions with strapping tape. Cover custody seals with tape to avoid seals being able to be peeled from the cooler.

M. Relinquish to Federal Express or to a courier arranged with the laboratory. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator along with the other documentation.

Medium- and High-Concentration Samples:

Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with potential additional restrictions. If applicable, the sample handler must refer to instructions associated with the shipping of dangerous goods for the necessary procedures for shipping by Federal Express or other overnight carrier. If warranted, procedures for dangerous-goods shipping may be implemented. Dangerous goods and hazardous materials pose an unreasonable risk to health, safety, or property during transportation without special handling. As a result, only employees who are trained under CH2M HILL Dangerous Goods Shipping course may ship or transport dangerous goods. Employees should utilize the HAZMAT ShipRight tool on the Virtual Office and/or contact a designated CH2M HILL HazMat advisor with questions.

V. Attachments

None.

VI. Key Checks and Items

- Be sure laboratory address is correct on the mailing label
- Pack sample bottles carefully, with adequate vermiculite or other packaging and without allowing bottles to touch
- Be sure there is adequate ice
- Include chain-of-custody form
- Include custody seals

Appendix B

Department of Defense Environmental Laboratory Accreditation Program Accreditation Letters

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA LABORATORIES SACRAMENTO
880 Riverside Parkway
West Sacramento, CA 95605
[redacted] [redacted]

ENVIRONMENTAL

Valid To: January 31, 2017 Certificate Number: 2928.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2009 TNI Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.0 of the DoD 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

Inductively Coupled Plasma (ICP), ICP-Mass Spectroscopy, Atomic Absorption Spectroscopy (flame), Gas Chromatography(GC), GC- Mass Spectroscopy, High Resolution Gas Chromatography/High Resolution Mass Spectroscopy, Liquid Chromatography(LC),LC- Mass Spectroscopy, Ion Chromatography, Spectrophotometry, Misc.- Electronic Probes

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Metals			
Aluminum	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Antimony	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Arsenic	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Barium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Beryllium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Boron	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	---------------------
Cadmium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Calcium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Chromium (Total)	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Chromium (Hexavalent)	EPA 7196A	EPA 7196A	---------------------
Cobalt	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Copper	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Iron	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Lead	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Magnesium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Manganese	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Mercury	EPA 7470A	EPA 7471A/7471B	----------------------
Molybdenum	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Nickel	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Phosphorus	EPA 6020/6020A	EPA 6020/6020A	----------------------
Potassium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Selenium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Silica	EPA 6010B/6010C	--	----------------------
Silicon	EPA 6010B/6010C	--	----------------------
Silver	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Sodium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Strontium	EPA 6020/6020A	EPA 6020/6020A	----------------------
Thallium	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	EPA 6020/6020A
Tin	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	----------------------
Titanium	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	----------------------
Uranium	EPA 6020/6020A	EPA 6020/6020A	----------------------
Vanadium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Zinc	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Nutrients			
Nitrate	EPA 353.2/9056A/300.0	EPA 353.2/ 9056A/300.0	----------------------
Nitrate-nitrite	EPA 353.2	EPA 353.2	----------------------
Nitrite	EPA 353.2/9056A/300.0	EPA 353.2/9056A/300.0	----------------------
Orthophosphate	EPA 9056A/300.0	EPA 9056A/300.0	----------------------
Wet Chemistry			
Alkalinity	SM 2320B (1997)	--	----------------------
Chemical Oxygen Demand	EPA 410.4	--	----------------------
Nitrocellulose	WS-WC-0050/353.2 Modified	WS-WC-0050/353.2 Modified	----------------------
Perchlorate	EPA 6850	EPA 6850	----------------------
pH	EPA 9040B/9040C	EPA 9045C/9045D	----------------------
Bromide	EPA 9056A/300.0	EPA 9056A/300.0	----------------------
Chloride	EPA 9056A/300.0	EPA 9056A/300.0	----------------------
Fluoride	EPA 9056A/300.0	EPA 9056A/300.0	----------------------
Sulfate	EPA 9056A/300.0	EPA 9056A/300.0	----------------------
Solids, Total	SM2540B (1997)	--	----------------------
Solids, Total Suspended	SM2540D (1997)	--	----------------------
Solids, Total Dissolved	SM2540C (1997)	--	----------------------
%Moisture	--	ASTM D2216	
TSP (Total Suspended Particulate)	--	--	40CFR Part 50 App B
PM10	--	--	40CFR Part 50 App J

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Hazardous Waste Characteristics			
TCLP Extractables	---------------------------------------	EPA 1311	--------------------
TCLP Inorganics	---------------------------------------	EPA 1311	--------------------
Purgeable Organics (volatiles)			
1,1,1,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	--------------------
1,1,1-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1,2,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1,2-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1-2-Trichloro-1,2-2-trifluorethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15-SIM
1,1-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	---------------------
1,2,3-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2,3-Trichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2,4-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2,4-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2-Dibromo-3-chloropropane	EPA 8260B/8260C	EPA 8260B/8260C	---------------------
1,2-Dibromoethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,3,5-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,3-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,3-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	---------------------
1,4-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,4-Dioxane	---------------------------------------	---------------------------------------	TO14A/TO15/ TO15 SIM
1-Chlorocyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	---------------------
2,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	---------------------
2-Butanone (MEK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
2-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
2-Hexanone (MBK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
2-Methyl-2-propanol (tert-Butyl Alcohol, TBA)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
4-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
4-Ethyltoluene	--	--	TO14A/TO15
4-Isopropyltoluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
4-Methyl-2-pentanone (MIBK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Acetone	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Acrolein	--	--	TO14A/TO15/ TO15 SIM
Allyl Chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Alpha Methyl Styrene	--	--	TO14A/TO15
Benzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Benzyl Chloride	--	--	TO14A/TO15/ TO15 SIM
Bromobenzene	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
Bromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
Bromodichloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Bromoform	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Bromomethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Butadiene (1,3-Butadiene)	--	--	TO14A/TO15/ TO15 SIM
Butane	--	--	TO14A/TO15
Carbon Disulfide	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Carbon Tetrachloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Chlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Chlorodifluoromethane	--	--	TO14A/TO15
Chloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Chloroform	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Chloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
cis-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
cis-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Cyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Dibromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Dibromomethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Dichlorodifluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Diisopropyl Ether (DIPE)	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
Dimethyl Disulfide	WS-MS-0003	WS-MS-0003	EPA 15/16

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Ethyl Acetate	--	--	TO14A/TO15
Ethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Ethylmethacrylate	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
Ethyl tert-butyl Ether (ETBE)	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
Hexachlorobutadiene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Hexane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Iodomethane	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
Isobutanol (2-Methyl-1-propanol)	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
Isooctane (2,2,4-Trimethylpentane)	--	--	TO14A/TO15
Isopropyl Alcohol	--	--	TO14A/TO15
Isopropylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
m & p Xylene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Methyl tert-butyl Ether (MTBE)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Methylene Chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Naphthalene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
n-Butanol	--	--	TO14A/TO15
n-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
n-Heptane	--	--	TO14A/TO15
n-Nonane	--	--	TO14A/TO15
n-Octane	--	--	TO14A/TO15
n-Propylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
o-Xylene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Pentane	--	--	TO14A/TO15
Propene	--	--	TO14A/TO15
sec-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Styrene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
t-Amyl methyl Ether (TAME)	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
t-1,4-Dichloro-2-Butene	EPA 8260B/8260C	EPA 8260B/8260C	----------------------
tert-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Tetrachloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Tetrahydrofuran	--	--	TO14A/TO15
Toluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
trans-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
trans-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Trichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Trichlorofluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Vinyl Acetate	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Vinyl Bromide	---------------------------------------	---------------------------------------	TO14A/TO15
Vinyl Chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Xylenes, Total	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Carbon Dioxide	---------------------------------------	---------------------------------------	ASTM1946D/3C
Nitrogen	---------------------------------------	---------------------------------------	ASTM1946D/3C
Oxygen	---------------------------------------	---------------------------------------	ASTM1946D/3C
Helium	---------------------------------------	---------------------------------------	ASTM1946D/3C
Hydrogen	---------------------------------------	---------------------------------------	ASTM1946D/3C
Methane	---------------------------------------	---------------------------------------	ASTM1946D/3C
Gasoline Range Organics (GRO)	EPA 8260B/AK101MS	EPA 8260B/AK101MS	TO14A/TO15
TPH as Gasoline	EPA 8260B/AK101MS	EPA 8260B/AK101MS	TO14A/TO15
Extractable Organics (semivolatiles)			
1,2,4,5-Tetrachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	---------------------
1,2,4-Trichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1,2-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1,2-Diphenylhydrazine (as Azobenzene)	EPA 8270C/8270D	EPA 8270C/8270D	---------------------
1,3-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1,3-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1,4-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1-Methylnaphthalene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
2,3,4,6-Tetrachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4,5-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4,6-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4-Dimethylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4-Dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,6-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,6-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Chloronaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Chlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Methylnaphthalene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
2-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
3&4-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
3,3'-Dichlorobenzidine	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
3-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4,6-Dinitro-2-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Bromophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Chloro-3-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Chloroaniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Chlorophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Acenaphthene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Acenaphthylene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Aniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Anthracene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(a)anthracene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(a)pyrene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(b)fluoranthene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(g,h,i)perylene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(k)fluoranthene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzoic Acid	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Benzyl Alcohol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Benzyl butyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Biphenyl	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Bis(2-chloroethoxy) Methane	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Bis(2-chloroethyl) Ether	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Bis(2-chloroisopropyl) Ether	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Carbazole	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Chrysene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
			TO-13A Modified
Bis (2-ethylhexyl) Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Dibenz(a,h)anthracene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Dibenzofuran	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Diethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Dimethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Di-n-butyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Di-n-octyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Fluoranthene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Fluorene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Hexachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Hexachlorobutadiene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Hexachlorocyclopentadiene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Hexachloroethane	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Indeno(1,2,3-c,d) Pyrene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Isophorone	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Naphthalene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Nitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
n-Nitrosodimethylamine	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
n-Nitrosodi-n-propylamine	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
n-Nitrosodiphenylamine	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Pentachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Phenanthrene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM/	WS-MS-0006/ TO-13A/ TO-13A Modified
Phenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Pyrene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Pyridine	WS-MS-0005	WS-MS-0005	---------------------
1,4-Dioxane	WS-MS-0011/8270C-SIM Modified	WS-MS-0011/8270C-SIM Modified	----------------------
Diesel Range Organics (DRO)	EPA 8015B/8015C/8015D/ AK102	EPA 8015B/8015C8015D/ AK102	----------------------
Residual Range Organics	AK103	AK103	----------------------
Motor Oil Range Organics (MRO)	EPA 8015B/8015C8015D	EPA 8015B/8015C8015D	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Dioxins			
2,3,7,8-TeCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,7,8-PeCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,4,7,8-HxCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,6,7,8-HxCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,7,8,9-HxCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,4,6,7,8-HpCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
OCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
2,3,7,8-TeCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,7,8-PeCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
2,3,4,7,8-PeCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,4,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,6,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,7,8,9-HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
2,3,4,6,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,4,6,7,8-HpCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
1,2,3,4,7,8,9-HpCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
OCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
Total TCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
Total PeCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
Total HxCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
Total HeptaCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
Total TCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
Total PeCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
Total HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------
Total HpCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Chemical Warfare Degradates			
1,4-Dithiane	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
Benzothiazole	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
p-Chlorophenyl methylsulfide	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
p-Chlorophenyl methylsulfoxide	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
p-Chlorophenyl methylsulfone	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
Chloropicrin	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
Acetophenone	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
2-Chloroacetophenone	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
1,4-Oxathiane	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------------------
Diisopropylmethylphosphate (DIMP)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------------------
Dimethylmethylphosphonate (DMMP)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------------------
Ethyl methylphosphonic acid (EMPA)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------------------
Isopropyl methylphosphonic acid (IMPA)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------------------
Methylphosphonic acid (MPA)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------------------
Thiodiglycol (2,2'-Thiodiethanol) (TDG)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------------------
Nitroaromatics			----------------------
2-Amino-4,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
4-Amino-2,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
3,5-Dinitroaniline	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
1,3-Dinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
2,4-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
2,6-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Glycerol trinitrate (Nitroglycerin)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Hexahydro-1,3,5-trinitro-1,3,5-triazine (Hexogen)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Nitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
2-Nitrotoluene (o-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
3-Nitrotoluene (m-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
4-Nitrotoluene (p-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Octahydro-1,3,5,7-tetranitro1,3,5,7-tetracine (Octogen)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Picric acid	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Pentaerythritol Tetranitrate	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
1,3,5-Trinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
2,4,6-Trinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Hexahydro-1,3-dinitroso-5-nitro-1,3,5,triazine (DNX)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Hexahydro-1,3,5-trinitroso-1,3,5-triazine (TNX)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
1-Nitroso-3,5-dinitro-1,3,5-triazacyclohexane (MNX)	EPA 8330A/8330B	EPA 8330A/8330B	----------------------
Nitroguanidine	WS-LC-0010/8330A Modified	WS-LC-0010/8330A Modified	----------------------
Nitrosamines			
N-Nitrosodimethyl amine (NDMA)	WS-MS-0012/521 Modified	WS-MS-0012/521 Modified	----------------------
Perfluoro Compounds			----------------------
6:2 Fluorotelomer sulfonate (6:2 FTS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
8:2 Fluorotelomer sulfonate (8:2 FTS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol [N-Et-FOSE]	WS-ID-0021/8290 Modified	WS-ID-0021/8290 Modified	----------------------
2-(N-Methylperfluoro-1-octanesulfonamido)-ethanol [N-Me-FOSE]	WS-ID-0021/8290 Modified	WS-ID-0021/8290 Modified	----------------------
N-Ethyl perfluorooctane sulfonamide (EtFOSA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
N-Methyl perfluorooctane sulfonamide (MeFOSA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
N-Methyl perfluorooctanesulfon amidoacetic acide (MeFOSAA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorooctanoic acid	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorooctane Sulfonate	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorobutyric acid (PFBA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluoropentanoic acid (PFPA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Perfluorohexanoic acid (PFHxA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluoroheptanoic acid (PFHpA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorononanoic acid (PFNA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorodecanoic acid (PFDA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluoroundecanoic acid (PFUDA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorododecanoic acid (PFDoDA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorotridecanoic acid (PFTriA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorotetradecanoic acid (PDTeA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorobutane Sulfonate (PFBS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorohexane Sulfonate (PFHxS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluoroheptane Sulfonate (PFHpS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluorodecane Sulfonate (PFDS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Perfluoroocatane Sulfonamide (FOSA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------------------
Pesticides/PCBs			
Aldrin	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
a-BHC	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
b-BHC	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
d-BHC	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
g-BHC (Lindane)	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
a-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
g-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
4,4'-DDD	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
4,4'-DDE	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
4,4'-DDT	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Dieldrin	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Endosulfan I	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Endosulfan II	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Endosulfan sulfate	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Endrin	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Endrin Aldehyde	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Endrin Ketone	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Heptachlor	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Heptachlor Epoxide	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Methoxychlor	EPA 8081A/8081B	EPA 8081A/8081B	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Toxaphene	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
Chlordane (technical)	EPA 8081A/8081B	EPA 8081A/8081B	----------------------
PCB (Aroclors)			
PCB-1016	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1221	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1232	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1242	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1248	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1254	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1260	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1262	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1268	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB (congeners) – removed references to BZ and IUPAC numbers.			
PCB 1	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 2	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 3	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 4	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 5	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 6	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 7	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 8	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 9	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 10	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 11	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 12	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 13	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 14	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 15	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 16	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 17	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 18	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 19	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 20	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 21	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 22	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 23	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 24	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 25	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 26	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 27	EPA 1668A/1668C	EPA 1668A/1668C	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 28	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 29	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 30	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 32	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 31	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 33	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 34	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 35	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 36	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 37	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 38	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 39	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 40	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 41	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 42	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 43	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 44	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 45	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 46	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 47	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 48	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 49	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 50	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 51	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 52	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 53	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 54	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 55	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 56	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 57	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 58	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 59	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 60	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 61	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 62	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 63	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 64	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 65	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 66	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 67	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 68	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 69	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 70	EPA 1668A/1668C	EPA 1668A/1668C	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 71	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 72	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 73	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 74	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 75	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 76	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 77	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 78	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 79	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 80	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 81	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 82	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 83	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 84	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 85	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 86	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 87	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 88	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 89	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 90	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 91	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 92	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 93	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 94	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 95	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 96	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 97	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 98	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 99	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 100	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 101	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 102	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 103	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 104	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 105	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 106	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 107	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 108	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 109	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 110	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 111	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 112	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 113	EPA 1668A/1668C	EPA 1668A/1668C	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 114	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 115	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 116	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 117	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 118	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 119	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 120	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 121	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 122	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 123	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 124	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 125	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 126	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 127	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 128	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 129	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 130	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 131	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 132	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 133	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 134	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 135	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 136	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 137	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 138	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 139	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 140	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 141	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 142	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 143	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 144	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 145	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 146	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 147	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 148	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 149	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 150	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 151	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 152	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 153	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 154	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 155	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 156	EPA 1668A/1668C	EPA 1668A/1668C	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 157	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 158	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 159	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 160	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 161	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 162	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 163	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 164	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 165	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 166	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 167	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 168	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 169	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 170	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 171	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 172	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 173	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 174	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 175	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 176	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 177	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 178	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 179	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 180	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 181	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 182	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 183	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 184	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 185	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 186	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 187	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 188	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 189	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 190	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 191	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 192	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 193	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 194	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 195	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 196	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 197	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 198	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 199	EPA 1668A/1668C	EPA 1668A/1668C	----------------------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 200	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 201	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 202	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 203	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 204	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 205	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 206	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 207	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 208	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
PCB 209	EPA 1668A/1668C	EPA 1668A/1668C	----------------------
Metals Digestion			
Acid Digestion Total Rcoverable or Dissolved Metals	EPA 3005A	--------------------------------------	----------------------
Acid Digestion for Total Metals	EPA 3010A	--------------------------------------	----------------------
Acid Digestion of Sediments, Sludges and Soils	--------------------------------------	EPA 3050B	EPA 3050B
Organic Preparation Methods			
Separatory Funnel Liquid-Liquid Extraction	--------------------------------------	EPA 3510C	----------------------
Ultrasonic Extraction	--------------------------------------	EPA 3550B/3550C	----------------------
Waste Dilution	EPA 3580A	EPA 3580A	----------------------
Solid-Phase Extraction	EPA 3535A	--------------------------------------	----------------------
Volatiles Purge and Trap	EPA 5030B/5030C	EPA 5030B	----------------------
Volatiles Purge and Trap for Solids	--------------------------------------	EPA 5035/5035A	----------------------
Semivolatiles in Air	--------------------------------------	--------------------------------------	TO-13
Chemical Warfare Degradates (in solid)	--------------------------------------	WS-OP-0005	----------------------
Microwave Assisted Extraction	--------------------------------------	EPA 3546	----------------------
Organic Cleanup Procedures			
Florisil Cleanup	EPA 3620B/3620C	EPA 3620B/3620C	EPA 3620B/ 3620C
Sulfur Cleanup	EPA 3660A	EPA 3660A	EPA 3660A
Sulfuric Acid Cleanup	EPA 3665A	EPA 3665A	EPA 3665A
Silica Gel Cleanup	EPA 3630C	EPA 3630C	----------------------

Parameter/Analyte	Potable Water
Perfluoro Compunds	
Perfluorobutane Sulfonate (PFBS)	EPA 537
Perfluoroheptanoic acid (PFHpA)	EPA 537
Perfluorohexane Sulfonate (PFHxS)	EPA 537
Perfluorononanoic acid (PFNA)	EPA 537
Perfluorooctanoic acid (PFOA)	EPA 537
Perfluoroocatane Sulfonate (PFOS)	EPA 537

Accredited Laboratory

A2LA has accredited

TESTAMERICA SACRAMENTO

West Sacramento, 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 17025:2005, 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 5.0 of the DoD 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 8 January 2009).

Presented this 30th day of December 2015.

Senior Director of Quality & Communications
For the Accreditation Council
Certificate Number 2928.01
Valid to January 31, 2017
Revised May 6, 2016

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

Appendix C
Scoping Session Meeting Minutes

Project Name: PFC Exposure Evaluation and Rapid Response

PM: Katie Tippin

Date of Session: August 2, 2016

Scoping Session Purpose: Project chartering to gain consensus on the path forward for the PFC exposure evaluation and rapid response. Define and agree to the project scope and objectives.

Name	Title/Project Role	Affiliation
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

The project team reviewed and discussed the procedure for the perfluorinated compound (PFC) exposure evaluation and rapid response.

The following were endorsed by the team:

- Develop a generic Sampling and Analysis Plan (SAP) with the laboratory worksheets and generic information included prior to identifying sites for investigation. The generic SAP will be developed and reviewed by the Navy for all laboratories procured. NAVFAC suggests using more than one laboratory, if possible.
- Once sites are identified, a desktop review will be completed to determine if there is a potential source for PFCs and potential receptors located downgradient offsite.
- Environmental questions and project action levels (PALs) were discussed and agreed upon.
- Sites identified as having potential sources for PFCs in addition to potential offsite receptors of drinking water will be investigated with the approach outlined below. A site-specific SAP for these sites will be developed, and edited worksheets will be submitted to the Base and the Navy for review.
- The following investigation approach was endorsed:
  - If PFC concentrations in groundwater at the suspected source exceed the PALs, then groundwater samples (from existing or new monitoring wells) will be collected downgradient at the facility boundary.
  - If PFC concentrations in groundwater at the facility boundary exceed the PALs, then drinking water samples will be collected at all potentially impacted drinking water sources within ½ mile downgradient of the PAL exceedances.
  - If PFC concentrations in drinking water exceed the PALs within ½ mile downgradient of the facility boundary, then potentially impacted drinking water sources within ½ mile downgradient of the furthest PAL exceedance will be sampled. This step-out process will be conducted until no additional drinking water sources impacted by PFCs above the PALs are identified.

  - If PFC concentrations in drinking water exceed the PALs within ½ mile downgradient, then alternate drinking water sources will be supplied.
- Groundwater and drinking water samples will be analyzed for perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS), and perfluorobutane sulfonate (PFBS).

Action Items

Endorse the project; NAVFAC chemists to confirm DOD path forward for using linear vs. branched, or both, for testing, calibration, etc. at laboratories. NAVFAC chemists also to confirm use of USEPA Method 537 vs. modified method.

Follow-up

Ken Bowers/NAVFAC clarified instruction on methodology and reporting requirements. For groundwater, USEPA Method 537 (Modified) will be used. For drinking water, USEPA Method 537 will be used. Branched and linear isomers will be reported for both PFOA and PFOS.

Project Name: PFC Exposure Evaluation and Rapid Response **PM:** Katie Tippin		
Date of Session: August 31, 2016 **Scoping Session Purpose:** Discussion on revisions to the proposed investigation approach discussed during the August 2, 2016 scoping session		
Name	**Title/Project Role**	**Affiliation**
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

The project team reviewed and discussed the revised procedure for the perfluorinated compound (PFC) exposure evaluation and rapid response as a result of updated policy guidance from ASN:

The following changes were made to the path forward:

- The collection of off-Base drinking water samples is the priority for this investigation. Drinking water samples will be collected from offsite drinking water supplies within 1 mile downgradient, if hydrogeology is known, or within a 1-mile radius, if hydrogeology is unknown, of suspected source area(s). If PFC concentrations in drinking water exceed the project action limits (PALs), then the investigation will be extended for an additional ½-mile downgradient from the exceeding location. The investigation area will be extended until there are no detections of PFCs in drinking water above the PALs.
- If PFC concentrations in drinking water collected from off-Base samples do not exceed the PALs then the PFC groundwater investigation will go back to the FECs for applicable regulatory investigation process.
- If PFCs are detected in drinking water above the PALs, then an alternate drinking water source will be supplied (consistent with August 2, 2016 scoping session). Additionally, onsite groundwater samples may be collected at the suspected source area(s) and downgradient at the Base boundary to determine the nature and extent of PFC concentrations in groundwater.

Appendix D
Laboratory Standard Operating Procedure

Title: Determination of Selected Perfluorinated Alkyl Acids (PFAA) in Drinking Water by Solid Phase Extraction (SPE) and Analysis by Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)

[Method 537]

Approvals (Signature/Date):

Robert Hrabak Operations Manager	10/24/16 Date	Joe Schairer Health & Safety Manager / Coordinator	10/25/16 Date
Lisa Stafford Quality Assurance Manager	10/26/2016 Date	for Crystal Pollock Laboratory Director	10/31/16 Date

Facility Distribution No. Uncontrolled **Distributed To:** Sacram,ento Biuds Folder

1. SCOPE AND APPLICATION

1.1. This procedure is based upon EPA Method 537 by J.A. Shoemaker, P.E. Grimmett, and B.K. Boutin, EPA Document # EPA/600/R-08/092.

1.2. This method covers the determination for six selected Perfluorinated alkyl acids (PFAA) in drinking water using Liquid Chromatography with tandem Mass Spectrometry (LC/MS/MS). A complete list of the PFAA for method 537 is listed on Table 1. The six compounds used for the validation and the minimum reporting limit (MRL) under the 537 UCMR3 program are listed below.

Analyte	CAS No.	MRL (ng/L)	Acronym
Perfluorobutanesulfonate, potassium salt	29420-49-3 375-73-5	90	PFBS
Perfluorohexansulfonate, sodium salt	82382-12-5 355-46-4	30	PFHxS
Perfluorooctanesulfonate, sodium salt	4021-47-0 1763-23-1	40	PFOS
Perfluorooctanoic acid	335-67-1	20	PFOA
Perfluoroheptanoic acid	375-85-9	10	PFHpA
Perfluorononanoic acid	375-95-1	20	PFNA

Note: Two CAS No. shown for the sulfonate compound; first is for the salt and second is the anion of the sulfonate compound.

1.3. The estimated detection limit (EDL) in aqueous sample is 4.0 ng/L for the PFAA compounds, however, the reporting limits for the six PFAA compounds monitored under the UCMR^3 program are listed above, which range from 10 to 90 ng/L (or 0.010 to 0.090 ug/L).

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

2. SUMMARY OF METHOD

2.1. A 250 mL aliquot of aqueous sample, preserved with dechlorinating agent, is loaded onto a 6-cc SPE cartridge containing 500mg of polystyrenedivinylbenezene (SDVB) packing. The PFAA compounds are eluted with methanol and the methanol eluent is concentrated to dryness with nitrogen in a heated water bath, spiked with internal standard, and adjusted to 1.0 mL final volume with 96:4 methanol-water.

2.2. A fixed volume, such as 10 µL, is injected on the HPLC equipped with a C_{18} column interfaced to a tandem mass spectrometer (LC/MS/MS). The compounds are identified by comparing the acquisition of the mass transition and retention times to reference spectra and retention times for the calibration standards acquired under identical LC/MS/MS conditions.

2.3. The concentration of each analyte is determined by using internal standard technique. Surrogate analytes are added to all Field and QC Samples to monitor the extraction efficiency of the method analytes and are quantitate by internal standard technique.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottle and caps, glassware, solvent bottles, squirt bottles, and other processing apparatus.. The method analytes in this method can also be found in many common laboratory supplies and equipment, such as polytetrafluoroethylene (PTFE) products, LC solvent lines, methanol, aluminum foil, and SPE manifold and sample transfer lines. All items such as these must be routinely demonstrated to be free from interferences under the conditions of the analysis by analyzing laboratory reagent blanks**. Subtracting blank values from the sample results is not permitted.**

4.2. Matrix interferences may also be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature of the sample. Humic and/or fulvic material is co-extracted by this method and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample.

4.3. The potential exists for trace level organic contaminants in preservatives that are added to sample bottles. Trizma® is the chemical buffering and dechlorinating agent used for this method. Interference from these sources should be monitored by analysis of the laboratory reagent blanks, particularly when new lots of reagents are acquired.

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

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. When briskly shaking the pH test strip and free-chlorine test strip after dipping, ensure that the test strip is only shaken away from you (into the hood). Do not shake the test strip back towards you.

5.1.2. Ensure that the vacuum exhaust hose used during the filtering is securely anchored inside of a fume hood so that vapors are not pumped into the working environment.

5.1.3. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. PVC gloves provide adequate levels of protection against the chemicals used in this SOP, except for acetonitrile. Nitrile gloves provide adequate levels of protection against acetonitrile.

5.1.4. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.5. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of SPE device and other laboratory equipment represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetonitrile	Flammable Poison	40 ppm -TWA	Early symptoms may include nose and throat irritation, flushing of the face and chest tightness Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption cam occur: symptoms may parallel inhalation exposure. Irritant to the eyes.
Trizma® preset crystals, pH 7.0	Irritant	None listed	Inhalation, ingestion, eye or skin contact can cause irritation, coughing, shortness of breath, nausea, vomiting, redness and itching.
Ammonium Acetate	Irritant	5 to 15 mg/m^3 - TWA	Hazardous in case of eye contact (irritant), ingestion, and inhalation – High level of ingestation may cause irritation of the gastrointestinal tract with generalized discomfort, diarrhea, nausea, vomiting abdominal pain. Slightly hazardous in case of skin contact (irritant) and inhalation - May affect behavior/central nervous system(tremor, anxiety, impairment of motor performance or recognition, flaccidity of facial muscles) and respiratory system (dyspnea). Inhalation of dust can irritate the respiratory tract (nose, throat, lungs) with symptoms of coughing, wheezing, and/or shortness of breath. Repeated or prolonged exposure to the substance can produce target organs damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Analytical column: Waters Atlantis dC$_{18}$, 3.5 µm, 150 x 2.1 mm, or equivalent.

6.2. Autosampler vials, 300-µL, polypropylene, with polypropylene screw caps or Agilent PTFE lined LC screw caps.

NOTE: Polypropylene vials and caps are necessary to prevent contamination of the sample from PTFE lined septa. If Agilent PTFE lined screw caps are determined to be free from contamination they may be used. However, polypropylene caps do not reseal and evaporation occurs after injection. Multiple injections from the same vial is not recommended unless it is immediate and for qualitative purpose only.

6.3. Balance – Analytical capable of accurately weighing to 0.0001g.

6.4. Bottles, 4-mL, 8-mL, and 250-mL size polypropylene, with polypropylene screw caps.

6.5. Extract concentrator or nitrogen manifold with water bath heating to 50 - 55°C.

6.6. Free-Chlorine Test Strips, designed to resist the interference from mono-chloramines and detection to 0.01 ppm, Fisher PN NC9116053, or equivalent

6.7. Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) – This method uses LC instrumentation and a tandem mass Spectrometer with Waters Quatro Premiere with MassLynx version 4.1 software or equivalent. The liquid chromatograph is equipped with a refrigerated autosampler, an injection valve, a pump capable of variable flow rate. The use of a column heater is required to maintain a stable temperature throughout the analytical run. The Tandem MS/MS is operated in negative ion electrospray (ESI) for method 537.

6.8. Wide range pH test paper, 0-14, or equivalent.

6.9. Pipettes, auto-pipets, and other equipment used to prepare standards and reagents.

6.10. Solid phase extraction (SPE) system.

6.10.1. SPE Cartridges – 500 milligram/6-cc SPE cartridges containing polystyrenedivinylbenzene (SDVB) sorbent phase (Agilent PN 12255021), or equivalent.

6.10.2. Vacuum extraction manifold – Supelco Visiprep, or equivalent. A manual vacuum manifold with column adapters, and column reservoirs for cartridge extraction.

6.10.3. A robotic or automated system may be used, however care must be taken to ensure the PTFE commonly used in these systems do not contribute to unacceptable concentrations in the laboratory reagent blanks. *NOTE: Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system and each port must pass PFC background check.*

6.11. Volumetric flask, Class A, various size 2.0-mL to 100-mL, as appropriate.

6.12. Test tubes, 15 mL, polypropylene, with polypropylene screw caps.

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit use without lessening the accuracy of the determination.

7.2. Acetonitrile, HPLC Grade, or equivalent.

7.3. Ammonium Acetate, $NH_4C_2H_3O_2$, HPLC Grade, Fisher PN A639-500, or equivalent.

7.4. Ammonium Acetate/Reagent water, 20mM; – Prepared by weighing 0.77 grams of ammonium acetate and dissolving in 500 mL of water. The resultant solution is filtered through a 0.22µm filter before use, and this solution is prone to volatility and should be replaced every 7 days or sooner.

7.5. Methanol, HPLC Grade, or equivalent.

7.6. Methanol-Water, 96:4 vol/vol., prepared by mixing 960mL methanol and 40 mL reagent water. Stored in polypropylene bottle and sealed with polypropylene screw cap.

7.7. Trizma® preset crystals, pH 7.0 (Sigma Aldrich Cat. No. T7193-250G or equivalent), a mixed blend of Tris [Tris(hydroxymethyl) aminomethane] and Tris HCl [Tris(hydroxymethyl)aminomethane hydrochloride] which function as a buffer and removes chlorine in chlorinated finished water. It is added to the sample bottle at a concentration of 5g/L (or 1.25g per 250 mL polypropylene bottle).

7.8. Reagent Water - HPLC grade water, distilled or de-ionized water, free of chemicals of interest.

7.9. **Standard neat and stock solutions** –

7.9.1. Standard neat and solutions are available from reputable vendor, such as Wellington Laboratories. However, source for PFOS and PFHxS, according to Method 537, must contain both the linear and branched isomers. Wellington standards do not meet this criteria, therefore, these standards must be purchased from other sources such as Santa Cruz Biotechnology and Sigma-Aldrich.

7.9.2. Neat materials are weighed and diluted in methanol at high concentrations such as 0.1 to 2 mg/mL in methanol. They are stored in a polypropylene bottle at 2-6° C and the solution is valid for 12 months.

7.9.3. Manufacturer will usually have an expiration date on the standards. If not, a two year from receipt of the standards is used for native compounds and five years for labeled or deuterated compounds.

7.9.4. Stock solutions purchased from vendors such as Wellington Laboratories are typically at 50 ug/mL in methanol.

7.9.5. A mixture of PFAA and PFAS from Wellington Laboratories is used as a second source and the concentration is at 2 µg/mL, however, if branched and linear isomers of PFC standards are available, they should be purchased and used for this method. Second source standards are purchased as neat or as solution and are from different vendors and/or different lots.

7.9.5.1. A technical (qualitative) grade PFOA standard which contains both linear and branched isomers is used as a retention time (RT) marker. This is used to integrate the total response for both linear and branched isomers of PFOA in environmental samples while relying on the initial calibration with the linear isomer quantitative standard

7.9.6. **537A Working Standard and 537A Spike Mix** – An appropriate amount of the stock solution is added to 10 mL volumetric flask and dilute to mark with methanol. The resultant mixture of the 537A UCMR3 IM Standard is 2.0 - 9.0 µg/mL and is stored in a polypropylene bottle at 2-6° C. The solution is valid for 6 months.

Analyte	**537 Stock Conc. (mg/mL)**	**Aliquot (mL)**	**537A Working Standard Conc. (µg/mL)**	**Aliquot (mL)**	**537A Spike Mix Final Conc. (µg/mL)**
PFBS(1)	2.0	0.45	90	1.0	9.0
PFHxS(1)	2.0	0.15	30		3.0
PFOA	2.0	0.10	20		2.0
PFOS(1)	2.0	0.20	40		4.0
PFHpA	0.050	--	--	0.40	2.0
PFNA	0.050	--	--	0.20	2.0

NOTE (1); The standard is received as salt form, the concentration is corrected as anion form

7.9.7. **537A Spike Mix** – Dilute the 537A Spike Mix in methanol to prepare the following:

537-SP	50-450 ng/mL	12.5 µL to 2.5 mL
537-SP2	200-1800 ng/mL	500 µL to 25 mL
537-SP3	380-3420 ng/mL	380 µL to 10 mL

. The final concentration is 0.20 to 0.90 µg/mL. The solution is stored in a polypropylene bottle at 2-6° C. and is valid for 6 months.

7.9.8. **537A IM Surrogate solution** – Prepare a 537A IM Surrogate Mix by adding appropriate amount of the stock solution to a 10.0 mL volumetric flask and dilute to mark with methanol. The resultant mixture is 1.0 ug/mL. The solution is stored in a polypropylene bottle at 2-6° C. and is valid for 6 months.

7.9.9. **537A Surrogate Mix** – Dilute 2.0 mL of the 537A IM Surrogate Solution to a final volume of 10 mL in methanol for a 5x dilution. The resultant mixture is 0.20 ug/mL. The solution is stored in a polypropylene bottle at 2-6° C. and is valid for 6 months.

537A Surrogate Analyte	Stock Conc. (mg/mL)	Aliquot (mL) to 10mL final volume	537A IM Surr Solution Conc. (ug/mL)	Aliquot (mL) to 10mL final volume	537A Surr Mix Conc. (ug/mL)
13C2-PFHxA	0.050	0.20	1.0	2.0	0.20
13C2-PFDA	0.050	0.20	1.0		0.20

7.9.10. **537A IS solution** – A 537A Internal Standard Mix is prepared by diluting the appropriate amount of stock solution to a 10.0 mL volumetric flask to mark with methanol. The resultant mixture is 1.0 – 2.868 ug/mL, stored in a polypropylene bottle at 2-6° C, and is valid for 6 months.

537A Internal Standard Analyte	Stock Conc, (mg/mL)	Aliquot (mL) to 10mL final volume	537A IS Solution Conc. (ug/mL)
13C2-PFOA	0.050	0.20	1.0
13C4-PFOS(2)	0.0478	0.60	2.868

NOTE (2); The standard is received as salt form, the concentration is listed as anion form.

7.9.11. **537A Calibration curve** – Refer to the schematics below for the preparation of the calibration curve to achieve the following concentrations in 96% methanol / 4% water. The calibration curve is stored in a polypropylene bottle at 2-6° C. and is valid for 6 months.

*** NOTE: L1 – L6 is used for UCMR3 program**

537A Standards	**Volume (uL) to add in 10 mL FV (+ 400uL water for a final composition of 96:4 Methanol:Water, and dilute to mark with Methanol)**								
	L1(*)	**L2(*)**	**L3(*)**	**L4(*)**	**L5(*)**	**L6(*)**	**L7**	**L8**	**L9**
537A Spike Mix (1.0-9.0 ug/mL)	10	25	50	100	150	200	250	400	500
537A IM Surr Mix (1.0 ug/mL)	100	100	100	100	100	100	100	100	100
537A IS Mix (1.0 ug/mL)	100	100	100	100	100	100	100	100	100

Analyte		**Standard Level - Concentration as ng/mL**								
		L1 1.0-9.0	**L2** 2.5-22.5	**L3** 5.0-45	**L4** 10-90	**L5** 15-135	**L6** 20-180	**L7** 25-225	**L8** 40-360	**L9** 50-450
PFBS		9.0	22.5	45	90	135	180	225	360	450
PFHxS		3.0	7.5	15	30	45	60	75	120	150
PFOA		2.0	5.0	10	20	30	40	50	80	100
PFOS		4.0	10	20	40	60	80	100	160	200
PFHpA		1.0	2.5	5.0	10	15	20	25	40	50
PFNA		2.0	5.0	10	20	30	40	50	80	100
SU	**13C2-PFHxA**	10	10	10	10	10	10	10	10	10
SU	**13C2-PFDA**	10	10	10	10	10	10	10	10	10
IS	**13C2-PFOA**	10	10	10	10	10	10	10	10	10
IS	**13C4-PFOS**	28.68	28.68	28.68	28.68	28.68	28.68	28.68	28.68	28.68

7.9.12. Second source standard is either from a separate vendor or a separate lot number from the primary stock standards. The standard is stored at 2-6°C. Unless specified by the manufacturer, an expiration date of six months from receipt of the standards is used.

7.9.13. Second source standard is not needed for the surrogate and IS compounds.

7.9.14. **Second Source Stock Solution** –Stock solutions prepared from neat are diluted in methanol at 2.0 mg/mL. They are stored in a polypropylene bottle at 2-6° C and the solution is valid for six months.

7.9.15. **537A ICV Working Solutio**n is prepared by diluting the second source stock standards in a 10 mL volumetric flask with methanol. They are stored in a polypropylene bottle at 2-6° C and the solution is valid for 6 months.

Volume for the preparation of the 537A ICV Mix (+400 µL water for a final composition of 96:4 Methanol: Water, and dilute to mark with Methanol)

Level	**Final Volume**	**537A ICV Working Std (10-50µg/mL)**	**537A IM Surrogate Mix (1.0 µg/mL)**	**537A IS Mix (1.0 – 2.868 µg/mL)**
ICV	10 mL	20 µL	100 µL	100 µL

Analyte	**537A ICV Stock Conc. (mg/mL)**	**Aliquot (mL)**	**537A ICV Working Standard Conc. (ug/mL)**
PFBS(1)	2.0	0.25	50
PFHxS(1)	2.0	0.05	10
PFOA	2.0	0.05	10
PFOS(1)	2.0	0.05	10
PFHpA	2.0	0.025	5.0
PFNA	2.0	0.05	10

7.9.16. **537A ICV Standard Mix** – is prepared by diluting the second source working solution in a 10 mL volumetric flask. The Surrogate and IS solutions, and 400 µL of water are added and diluted to mark with methanol. The 537A ICV Standard Mix is stored in a polypropylene bottle at 2-6° C and the solution is valid for 6 months.

Analyte		ICV Concentration (ng/mL)
PFBS		100
PFHxS		20
PFOA		20
PFOS		20
PFHpA		10
PFNA		20
SU	**13C2-PFHxA**	10
SU	**13C2-PFDA**	10
IS	**13C2-PFOA**	10
IS	**13C4-PFOS**	28.68

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE FOR PFC

8.1. A 250 mL or larger polypropylene bottle with a polypropylene screw cap is recommended for sample collection. High-density polyethylene (HDPE) containers with HDPE screw caps may also be used, based on the availability of the containers. Prior to shipment to the field, the buffering and dechlorinating reagent Trizma® must be added as a dry solid to each bottle so that the final amount is 5g/L. For 250 mL size bottle, 1.25 g of Trizma® is added. For larger sizes the amount of Trizma® added is increased proportionally.

8.2. The sample handler must wash their hands before sampling and wear nitrile gloves while filling and sealing the sample bottles. PFAA contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile glove will aid in minimizing this type of accidental contamination of the samples.

8.3. Sample bottles must not be pre-rinsed with sample before collection.

8.4. Fill sample bottles, taking care not to flush out the sample preservative. Samples do not need to be collected headspace free.

8.5. After collecting the sample cap carefully to avoid spillage, and agitate by inverting a few times. Keep samples sealed until analysis.

8.6. Field reagent blanks (FRB) must accompany with the sample set. The sample set is composed of samples collected from the sample site and at the same time.

8.6.1. At the laboratory, a sample bottle is filled with reagent water and preservatives, seal, and ship to the sampling site along with the sample bottles. For each FRB shipped, an empty sample bottle (no preservatives) must also be shipped.

8.6.2. At the sampling site, the sampler must open the shipped FRB and pour the reserved reagent water into the empty shipped bottle, seal and label this bottle as the FRB.

8.6.3. The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFAAs were not introduced into the sample during sample collection and handling.

8.6.4. The preservative used for the FRB must be the same batch used for the samples.

8.7. Samples must be chilled during shipment and must not exceed 10°C during the first 48 hours following collection. Sample temperature must be confirmed to be at or below 10°C when samples are received at the laboratory.

8.8. Free chlorine test must also be performed when samples are received at the laboratory to verify that free chlorine has been neutralized. If free chlorine is present, then the sample is not acceptable for this method – the client must be notified for possible re-sampling.

8.9. Samples stored in the laboratory must be stored below 6°C until extraction, but it should not be frozen. Do not freeze.

8.10. Aqueous samples must be extracted within 14 days of sampling when samples are properly preserved, shipped, and stored.

8.11. Extracts must be stored at room temperature and analyzed within 28 days after extraction.

8.11.1. Extracts and standards stored at 2-6°C has no impact, however, they must be at room temperature and vortexed or mixed well before aliquoting.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.2. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.

9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/matrix spike duplicate (MS/MSD), a laboratory control sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD an LCSD may be substituted if required by the program or client. In the event that multiple MS/MSD are run with a batch due to client requirements, the additional MS/MSD do not count toward the maximum 20 samples in a batch.

9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. The method blank is processed in the same manner, including the addition of dechlorinating agent, anti-microbial agents, and pH adjustments on, and at the same time, as the associated samples. Corrective actions must be documented on a Non-Conformance memo and implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside of the control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable.

9.3.1. If the MB produces a peak within the retention time window of any of the analytes determine the source of the contamination and eliminate the interference before processing samples.

9.3.2. The method blank must not contain any analyte at or above 1/3 of the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be taken in consultation with the client.

9.3.4. Re-extraction and re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.4. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS fortification level must be rotated between low to high from batch to batch.

9.4.1. The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner (including the addition of dechlorinating and anti-microbial chemicals) and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable.

9.4.2. Similarly, the high concentration LCS should be near the high end of the calibration range established during the initial calibration.

9.4.3. The percent recovery (%R) for each analyte is calculated using the equation.

$$\%R = (A-B)/C \cdot 100$$

Where

A = measured concentration in the fortified sample.

B = measured concentration in the unfortified sample

C = expected fortified concentration.

9.4.4. Analyte recoveries may exhibit matrix bias. For samples fortified at mid or high levels, the analyte recoveries should range between 70-130%. For low level at or near the MRL (within 2x MRL) the analyte recoveries should range between 50 to 150%.

9.4.5. If the analyte(s) recovery do not meet the criterion and the CCV standard is in control, a second injection should be done to determine if there was a bad injection.

9.4.6. If the analyte(s) is in control, report the valid injection

9.4.7. If the analyte(s) is out of control, the data for this problem analyte(s) must be considered invalid for all samples in the extraction batch.

9.5. Surrogate recoveries for the Method Blank, LCS, and samples must be within the limits of 70% to 130%.

9.5.1. Surrogate recovery is calculated using the following equation

$$\% R = (A/B) \cdot 100$$

Where
A = calculated SURR concentration for the QC or field sample
B = Fortified concentration of the SURR.

9.5.2. If the surrogate recovery is less than 70% or greater than 130%, check the calculation to locate possible errors, check for standard degradation, contamination, and instrument performance. Correct the problem and re-analyze the extract.

9.5.3. If the extracts reanalysis fails the 70-130% criterion, the analyst should check the calibration by injecting and evaluating the last CCV that passed.

9.5.4. If the CCV standard failed, then recalibration is in order.

9.5.5. If the CCV standard passed, then extraction of the sample should be repeated provided the sample is still within the holding time.

9.5.6. If the re-extraction also fails the recovery criterion, report all data for that sample as suspect/SURR recovery to inform the data user that the results are suspect due to SUR recovery.

9.5.7. Alternatively, collect a new sample and re-analyze.

9.6. Internal Standard (IS) - must be monitored during each analysis day. Internal Standards is added to all blanks, standards and samples before analysis.

9.6.1. The IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration and must not deviate by more than 70-140% from the most recent CCV standard.

9.6.1.1. This is performed by assigning all non-CCV samples to be compared to both the last CCV and last ICIS sample within the Chrom method. (Method Editor, Limit Group, Edit, ISTD/Tune/Tail tab)

9.6.2. If the IS areas do not meet the criteria for samples, re-analyze the affected sample in a new capped autosampler vial. Random evaporation loss has been observed with PP caps causing high IS areas.

9.6.3. A poor injection, as well as matrix enhancement or suppression could cause the IS area to exceed these criteria. A second injection should be done to determine if there was a bad injection.

9.6.4. If the re-injected aliquot produces an acceptable IS response, report results for that aliquot.

9.6.5. If the re-injected aliquot failed again, the analyst should check the calibration by reanalyzing the most recently acceptable CCV standard.

9.6.6. If the CCV standard fails, recalibration is in order.

9.6.7. If the CCV standard is acceptable, extraction of the sample may need to be repeated provided the sample is still within the holding time. Otherwise, report results obtained from the re-injected extract, but annotate as suspect.

9.6.8. Alternatively, collect a new sample and re-analyze.

9.7. Continuing Calibration Verification (CCV) – is analyzed at the beginning of a run, the end of a run, and after every 10 samples to determine if the calibration is valid.

9.7.1. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required.

9.7.2. The CCV should vary throughout the run. A low level CCV is analyzed first, then subsequent CCV alternate between the mid and high level.

9.7.3. At the beginning of a run and if a curve has not been run that day, two levels of CCV should be run. One CCV should be run at the reporting limit and the other CCV at the mid point of the curve.

9.7.4. The reporting limit CCV should be within 50% of the true value and the mid and high level CCV should be within 30% of the true value.

9.8. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples.

9.8.1. The MS/MSD fortification level must be rotated between low to high from batch to batch.

- The low concentration MS/MSD must be no greater than 2x MRL.

9.8.2. Spiked analytes with recoveries or precision outside of the control limits may be reported if result for the LCS is in control. Corrective actions must be documented on a nonconformance memo and implemented when recoveries of any spiked analyte are outside of the control limits provided by the LIMS or by the client.

9.8.3. A duplicate control sample (LCSD or DCS) may be substituted when insufficient sample volume is provided to process an MS/MSD pair if required by the program or client. The LCSD is evaluated in the same manner as the LCS.

9.9. Initial calibration verification (ICV) – When available, a second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve.

Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.

9.10. A typical run sequence for 537 would be as follows:

1. Primer (A number of primers until the instrument is stable)
2. Blank
3. Calibration Curve (A minimum of 5 calibration levels)
4. Blank + IS
5. CCV low-level (at MRL)
6. ICV
7. PFOA RT marker, as needed
8. MB
9. LCS at (rotated between low to high fortification level from batch to batch).
10. Sample1
11. Sample1 MS
12. Sample1 MSD
13. Sample 2
14 etc. (up to 10 field samples between CCVs).
15. CCV mid or high-level (Rotate mid, and high level CCV throughout the sequence).

10. CALIBRATION

10.1. Mass tuning is performed by the service engineer following major maintenance to the mass spectrometer.

10.2. Initial Tune:

10.2.1. Mass calibration is verified on an as-needed basis, when conditions have been optimized, by acquiring a full-scan mass spectrum of the target analytes ions. The target analyte ions should be within 0.3 m/z of the expected mass.

10.2.2. Optimize the $[M-H]^-$ for each target analyte by infusing approximately 1.0 µg/mL of each analyte into a tee fitting with eluent flowing at about 0.1 ml/min. Set the entrance and exit slits to about 45, the collision to 0 and the collision gas off. Working with the parent ions for each analyte the MS parameters capillary voltage, temperatures, gas flows, etc.) is varied until optimal analyte responses are determined. The target analytes may have different optimal conditions requiring some compromise on some of the conditions for the precursor ions.

10.2.3. With the infusion still running next turn on the collision gas, set the entrance to about -5, the exit to about 1.5, and vary the collision voltage and collision gas to get optimal conditions for each analyte. A compromise setting for the collision gas will have to be made for product ions.

10.3. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to new columns or pump seals. A new calibration is not required after minor maintenance.

10.4. With the exception of the circumstances delineated in policy CA-T-P-002, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Average Response Factor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points.

10.5. The following requirements must be met for the initial calibration to be used.

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve must be less than ± ½ the reporting limit for the analyte.
- The internal standard area count must be within criteria for the standards.
- The origin must be forced through zero (specified by method 537).
- There must be no carryover at or above 1/3 MRL after a high calibration standard.
- At least five calibration levels are used to prepare the initial calibration curve, however, for quadratic fit at least six calibration levels are used.

10.6. A fixed injection volume is used for quantitation purposes and is to be the same for samples and standards in the analytical sequence.

10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL and the use of peak area.

10.8. **Initial Calibration -**At least five calibration levels are used to prepare the initial calibration curve, except for quadratic fit at least six calibration levels are used. Larger concentration range will require more calibration points. Each standard is injected once to obtain the peak area for each analyte at each concentration and the internal standard area count must be within criteria from each standard.

10.8.1. The lowest concentration of the calibration standard must be at or below the MRL, which may depend on system sensitivity. It is recommended that at least four of the calibration standards are at a concentration greater than or equal to the MRL.

10.8.2. The quantitation is performed using the Internal Standard technique. The data system software is used to generate the best fit; average response factor, a linear regression, or a quadratic fit for each of the analytes. The linear regression or quadratic fit **must always** be forced through zero and may be concentration weighted, if necessary. Forcing through zero allows for a better estimate of the background levels of the method analytes.

10.8.3. Peak Asymmetry Factor – a measure of peak tailing. The peak asymmetry factor must be calculated for the first two eluting peaks (if only two analytes are being analyzed, both must be evaluated) on a mid-level calibration standard.

10.8.4. The peak asymmetry factors must fall in the range of 0.8 to 1.5 using the equation below when the initial calibration curve is generated and during the initial demonstration is performed.

Note: Modifying the standard or extract composition to more aqueous content to prevent poor peak shape is not permitted.

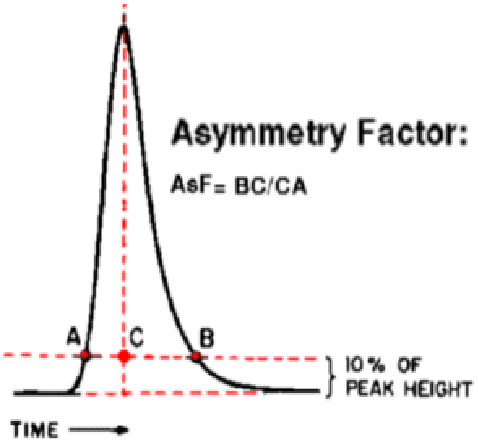


Figure 1 $^{\text{ref. 16.4.6}}$

Where:

AsF = peak asymmetry factor

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

AC = 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 peak apex. Attach this document to the calibration standard used for evaluation by scanning the document and associating it to the file as a document type of High Res MS Tune in TALS.

10.8.5. If peak asymmetry factor is not met or if peak broad, split or fronting peaks are observed for the first two eluting chromatographic peaks, change the initial mobile phase to higher aqueous content until the peak symmetry factor is met (AsF = 0.8 to 1.5).

10.8.6. The calibration acceptance criteria for the initial calibration are as follow:

- Each calibration point, except the lowest point, of each analyte should be calculated to be within 70-130% of the true value. The lowest calibration point that is at or below the MRL should be within 50-150% of its true value.

537 ICAL Criteria			
Standard Concentration	**Criteria for Native Analyte**	**Criteria for Surrogate**	**Criteria for IS**
Lowest calibration standard ≤ MRL	± 50% of true value	± 30% of true value	20% RPD
> MRL	± 30% of true value	± 30% of true value	20% RPD

- If these criteria cannot be met, it is recommended that corrective action is taken to reanalyze the calibration standards, restrict the range of calibrations, or select an alternate method of calibration (however, forcing the curve through zero is still required).

- Method 537 specifies five calibration concentrations of the initial calibration curve spanning a 20-fold concentration range. Standard preparation consists of 9 calibration levels; however, higher concentration levels are not required and may not meet the ICAL criteria's. Standard levels 1- 6 were used for the method 537 validation.

10.9. **Continuing calibration verification standard (CCV)** – is injected prior to sample analysis (with the exception of after a curve and ICV a CCV is not needed until 10 field samples have been run), following every 10 field samples or fewer, and at the end of the analytical run. *See section 9.10 for a typical run sequence.*

10.9.1. The method blank (MB), laboratory control sample (LCS), and matrix spike/spike duplicate (MS/SD) are not counted as field samples.

10.9.2. The first CCV injected must be the low level standard at or below the MRL to verify instrument sensitivity prior to any analysis. If standards have been prepared such that all low calibration points are not in the same calibration solution, it may be necessary to analyze two calibration standards to meet this requirement.

10.9.3. Subsequent CCV should alternate between the mid-level and high-level standards. If the initial calibration curve was not performed on the day of sample analysis, two CCV are required prior to analysis, the first one at or below the MRL, and the second one near the mid-level of the calibration curve.

537 CCV Criteria			
Standard Concentration	**Criteria for Native Analyte**	**Criteria for Surrogate**	**Criteria for IS**
First CCV of batch (≤ MRL)	± 50% of true value	± 30% of true value	± 50% of true value and 70-140% from most recent CCV.
First CCV of batch (> MRL)	± 30% of true value	± 30% of true value	± 50% of true value and 70-140% from most recent CCV.
Subsequent CCVs (>MRL)	Within ± 30% of true value	± 30% of true value	± 50% of true value and 70-140% from most recent CCV.

10.9.4. If the CCV standard is out of control, another injection of the standard may be injected singly to determine if the system is back in control. If it is still out of control, then there may be a problem with the standards, the instrument, or the column. If the column is replaced, then a new curve is necessary.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, and chemical characteristics, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviation from this procedure must also be documented as a nonconformance, with a cause and corrective action described. See WS-QA-0023 for additional information on the established procedures for the identification and documentation of non-conformances and corrective actions.

11.3. Samples must be checked for verification of temperature when samples are received at the laboratory and free chlorine check and pH as soon as possible after samples have arrived.

11.3.1. Check each sample for pH and presence of free chlorine. Record the observations.

11.3.2. **For pH determination**, dip a pH test strip briefly into an aliquot of the sample in a disposable container.

11.3.3. Remove and shake the test strip once briskly to remove the excess sample.

WARNING: Ensure that the pH and free-chlorine test strips are shaken away from you (into the hood) and not toward you.

11.3.4. Compare the test strip to the reference chart to determine the pH of the sample.

11.3.5. Note the pH on the bench-sheet. If the value is different by 1 pH unit from 7.0, the sample was not sufficiently preserved. The Project Manager must be notified and file a non-conformance memo as well.

11.3.6. If pH was not within the range, Trizma® was either not used as a preservative in the sample or the sampling was not performed correctly. Continue with the free chlorine determination on the samples.

11.3.7. **For free chlorine determination**, dip a free-chlorine test strip into the aliquot for 20 seconds with a constant gentle back and forth motion.

11.3.8. Remove and shake the test strip once briskly to remove the excess sample.

WARNING: Ensure that the pH and free-chlorine test strips are shaken away from you (into the hood) and not toward you.

11.3.9. Wait 20 seconds and compare the test strip to the reference chart to determine the concentration (ppm or mg/L) of free chlorine. Complete the comparison within one minute.

11.3.10. Note and record the concentration on the bench-sheet. If the value is greater than or equal to 0.1 mg/L, the sample is not sufficiently dechlorinated. The Project Manager must be notified immediately and file a non-conformance memo.

11.4. If free chlorine is present, Trizma® was either not used as preservative in the samples or the sampling was improperly performed. If free chlorine is present, then the sample is not acceptable for this method – the client must be notified for possible re-sampling.

11.4.1. Aqueous samples must be preserved and dechlorinated, collected and stored as presented in Section 8. All field and QC samples included the MB and LCS must contain the chemical agent listed in Section 8.1. If the sample was collected in 250 mL bottles just mark the bottles where the sample level is and the sample amount can be determined later. The sample amount can also be determined by weight difference of the bottle before and after it is emptied. If the sample was collected in larger bottles, pour 250mL of sample into a 250mL propylene bottle. For MB and LCS de-ionized water with dechlorinating agent added at the appropriate level is used (approximately 25 to 30 mg per 250 mL).

11.5. Add the 537A-Surrogate to all samples, including the MB, LCS, and MS/SD;

Note: Surrogate solution should be at room temperature and adequately vortex to ensure contents are mixed well before using.

- Add 50µL of the 0.20 ug/mL 537A-Surr Mix to a 250 mL sample for a final concentration of 40 ng/L.

11.6. One laboratory control sample (LCS) must be prepared with every batch – the LCS is rotated between low to high levels between subsequent batches.

Note: Spike solution should be at room temperature and adequately vortexed to ensure contents are mixed well before using.

LCS Fortification				
Amount Added		**Standard Concentration**		**Sample Concentration**
Low	50 µL	537-SP SPIKE	50 - 450 ng/mL	10 - 90 ng/L
Mid	50 µL	537-SP2 SPIKE	200 – 1800 ng/mL	40 - 360 ng/L
High	50 µL	537-SP3 SPIKE	380 – 3420 ng/mL	76 - 684 ng/L

11.7. The MS/MSD is rotated between low to high levels between subsequent batches.

MS/SD Fortification				
Amount Added		**Standard Concentration**		**Sample Concentration**
Low	50 µL	537A -SP SPIKE	50 - 450 ng/mL	10 - 90 ng/L
Mid	50 µL	537A SP2 SPIKE	200 – 1800 ng/mL	40 - 360 ng/L

Fortification levels for 537A on LCS and MS/SD (ng/L)

Analytes	Low Level (ng/L) 50uL	Mid Level (ng/L) 50uL	High Level (ng/L) 50uL
PFBS[1]	90	360	648
PFHpA	10	40	72
PFHxS[1]	30	120	216
PFOA	20	80	144
PFOS[1]	40	160	288
PFNA	20	80	144

11.8. After the addition of the surrogate solution and spike solution, seal the sample bottles with the screw caps and mix contents well before proceeding by inverting and shaking.

11.9. Solid Phase Extraction (SPE).

Note 1: Avoid the use of Teflon products for this method, especially with the Teflon squirt bottles. The polyethylene (PE) bottle has been QC'ed and found to be acceptable for the PFC analysis.

Note 2: Sample volume may be determined by volume or by weight. Do not transfer sample to graduated glass cylinders for volume determination as PFAAs may absorb to the glass surface, a plastic graduated cylinder may be used. If volume is determined by weight, measure the mass to the nearest 10 grams and density of 1.0g/mL, and determine volume by the difference in mass of the emptied bottle after SPE and sample bottle before SPE.

Note 3: Cartridge conditioning and loading – DO NOT allow cartridge packing material to go dry during any of the conditioning steps. If they do go dry, then the conditioning step must be started over.

11.9.1. Set up SPE cartridge for conditioning - 500 milligram/6-cc SDVB sorbent phase (Agilent PN 12255021) was used for the method validation of 537.

11.9.2. Document the manufacturer and lot number of the SPE cartridge.

- New SPE lots should be QC for PFC recovery and background check before using.
- New source for the sorbent phase must undergo the IDOC, verify that all Quality Control (QC) acceptance criteria are met, and that acceptable method performance can be verified in a real sample matrix.

<u>Warning:</u> Ensure that the vacuum exhaust hose used during the filtering is securely anchored inside of a fume hood so that vapors are not pumped into the working environment.

11.9.3. Condition the cartridge with 15 mL of methanol followed by 18 mL of water with vacuum. Stop the flow when the water reaches the top of the packing. If it goes dry, repeat the conditioning.

11.9.4. Add ~5 mL of water to each cartridge, and attach the adapter and column reservoir.

11.9.5. Turn on the vacuum and begin loading the sample to the reservoir. The vacuum must be adjusted so that the flow rate is approximately 10-15 mL/min. DO NOT allow sample to go dry before the completion of the sample loading and rinsing steps.

11.9.6. After the entire 250mL sample has been loaded onto the column, rinse the sample bottle with two 7.5mL aliquots of reagent water and pour onto the column reservoir.

11.9.7. After the entire sample has passed, rinse the reservoir with an additional 5.0 mL of reagent water.

11.9.8. After the last rinsing, allow the cartridge to dry under high vacuum (10-15 inch Hg) for 5 minutes (leave reservoirs on at this stage).

11.9.9. After drying, turn off vacuum pump and release vacuum.

11.9.10. Set up collection tubes to the SPE manifold using 15-mL polypropylene centrifuge tubes.

11.9.11. Rinse sample bottles with 4 mL of methanol and transfer to the column reservoir onto the cartridge. Elute the analytes from the cartridge by pulling the methanol through using low vacuum such that the solvent exits the cartridge in a drop wise fashion. Repeat sample bottle-to-column reservoir rinse and cartridge elution with a second 4-mL aliquot of methanol. A total of 8 mL methanol is collected.

11.10. Extract Concentration

11.10.1. Concentrate the extract volume under gentle stream of nitrogen with water bath at 60-65ºC to dryness.

11.10.2. Add 1.0 mL of the 96:4 Methanol:Water solution, and mix the contents well using a vortex mixer.

11.10.3. Add the appropriate amount of 537A-IS to all samples;

- Add 10uL of the 1.0-2.868 ug/mL 537A-IS solution for a final concentration of 10-28.68 ng on a 1.0mL extract.

11.10.4. Mix the contents well before transferring a portion of the extracts to polypropylene autosampler vials.

- Do not transfer the entire 1-mL aliquot to the autosampler vial because the polypropylene autosampler caps does not reseal after injection. The extracts in the autosampler vial after injection is lost due to evaporation, it is not recommended to be reused for further analysis. Extracts can be stored in the 15-mL centrifuge tubes.

11.10.5. Transfer approximately 60uL (8 – 10 drops) of the sample to a 300uL polypropylene autosampler vial with a plastic pipet or with an automatic pipet / plastic tip. Seal the PP autosampler vial with the polypropylene screw.

11.10.6. The extracts in polypropylene vials are ready for LC/MS/MS analysis.

11.10.7. Seal the rest of the extract in the 15-mL PP centrifuge tube with the PP screw cap and stored the extract at 2-6ºC.

Note: If further process of the extracts is necessary, such as re-injection or dilution, the extract should be equilibrated to room temperature and adequately vortexed to ensure contents are mixed well before proceeding.

11.11. **Instrument Conditions for method 537**

11.11.1. Instrument used in the method validation of 537 – Waters Premier XE with an Acquity UPLC.

11.11.2. Column recommendation: Waters Atlantis dC18, 5u, 150x2.1 mm (PN 186001301).

11.11.3. LC Solvent System

Reservoir	Solvent
A	20mM Ammonium Acetate in water
B	Methanol
Strong Wash	1:1 Acetonitrile-Methanol
Weak Wash	10% Methanol in Water
Seal Wash	20% Methanol in Water

11.11.4. LC Program[1] for Method 537A.

Time (min)	A%	B%	Flow Rate	Curve
0	80	20	0.10	initial
2	80	20	0.01	6
12	20	80	0.10	6
19	10	90	0.10	6
19.5	0	100	0.10	6
22	0	100	0.10	6
22.1	80	20	0.10	6
27.0	80	20	0.10	end

Run Time (minutes)	27
Strong Wash Volume (uL)	800
Weak Wash Volume (uL) Temperature (ºC)	2500
Autosampler Temperature (ºC)	10
Column Temperature (ºC)	35
Injection Volume (uL)	10
Loop Option – Partial Loop with Needle Overfill	
High Pressure Limit (psi)	5000

Footnote 1: Instrument conditions used in validation of 537A on instrument A6. Conditions may change to improve resolutions and responses.

NOTE 2: Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

NOTE 3: Mobile phase modifiers other than 20 mM ammonium acetate may be used at the discretion of the analyst, provided that the retention time stability criteria is met over a period of two weeks.

NOTE 4: LC system components, as well as the mobile phase constituents, contain many of the method analytes in this method. Thus, these PFAAs will build up on the head of the LC column during mobile phase equilibration. To minimize the background PFAA peaks and to keep background levels constant, the time the LC column sits at initial conditions must be kept constant and as short as possible (while ensuring reproducible retention times). In addition, prior to daily use, flush the column with 100% methanol for at least 20 min before initiating a sequence. It may be necessary on some systems to flush other LC components such as wash syringes, sample needles or any other system components before daily use.

NOTE 5: The PFC kit supplied by Waters consists of a PFC isolator trap, PEEK solvent lines, and non-fluorinated solvent filters helps minimize PFAA background in the LC system.

11.11.5. **Tandem MS conditions -** The following instrument parameters must be optimized whenever necessary in order to provide good performance.

Parameter	Setting
Ionization Mode	ESI -
Capillary Voltage	2.80 Kilovolts
Cone Voltage	Varies on compounds

Extractor Voltage	3 Volts
RF lens	0.2 Volts
Source Temperature	130 °C.
Desolvation Temperature	350 °C.
Cone Gas Flow (Nitrogen)	98 L/hr
Desolvation Gas Flow (Nitrogen)	1100 L/hr
Collision Energy	Varies on compounds
Collision Gas flow (mL/min, Argon)	0.25
Collision Gas Pressure (m bar)	2.88e-3
Collision T-Wave Parameters	Automatic
Multiplier Voltage	750

11.11.6. Mass Spectrometer (MS) scanning conditions - Conditions of the analysis in negative-ion multiple-reaction-monitoring (MRM) mode listed below;

Mass Spectrometer Conditions for MRM negative ion ESI mode

The RT of the PFAA using the UPLC conditions and column as listed in this method.

Desc. = Description - N = native
S = surrogate
IS = internal standard

Function Number (F-#);Acquisition time (minutes)
F-1; 10.0 to 14.0
F-2; 12.5 to 15.5
F-3; 13.5 to 16.5
F-4; 14.5 to 17.5
F-5; 15.0 to 18.0
F-6; 16.0 to 19.0
F-7; 16.5 to 19.5
F-8; 16.5 to 19.5
F-9; 17.5 to 21.0

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Desc.	F-#	Analyte	Precursor Ion	Product Ion	Dwell sec.	Cone Voltage	Collision Energy	RT (min)
N-1	**F-1**	**PFBS**	299	80	0.025	40	30	12.36
N-2	F-2	PFHxA	313	269	0.025	10	30	13.74
S-1	**F-2**	**13C2-PFHxA**	315	270	0.025	15	10	13.74
N-3	**F-3**	**PFHpA**	363	319	0.025	15	10	14.86
N-4	**F-3**	**PFHxS**	399	80	0.025	45	33	14.81
N-5	**F-4**	**PFOA**	413	369	0.025	16	10	15.74
IS-1	**F-4**	**13C2-PFOA**	415	370	0.025	10	10	15.74
N-6	**F-5**	**PFNA**	463	419	0.025	16	10	16.48
N-7	**F-5**	**PFOS**	499	80	0.025	55	45	16.39
IS-2	**F-5**	**13C4-PFOS**	503	80	0.025	50	45	16.39
N-8	F-6	PFDA	513	469	0.025	16	10	17.10
S-2	**F-6**	**13C2-PFDA**	515	470	0.025	12	12	17.10
N-9	F-7	N-Me-FOSAA	569.9	419.1	0.025	25	20	17.44
IS-3	F-7	d3-N-MeFOSAA	572.9	419.0	0.025	25	20	17.43
N-10	F-8	PFUdA	563	519	0.025	16	10	17.64
N-11	F-8	N-EtFOSAA	584	419	0.025	25	20	17.72
S-3	F-8	d5-N-EtFOSAA	589	419	0.025	25	20	17.71
N-12	F-9	PFDoA	613	569	0.025	16	10	18.14
N-13	F-9	PFTrDA	663	619	0.025	15	10	18.71
N-14	F-9	PFTeDA	713	669	0.025	15	10	19.41

Bold = UCMR^3 program

12. DATA ANALYSIS AND CALCULATION

12.1. Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentration using MS/MS. Concentrations were calculated by measuring the product ions listed. Other ions may be selected at the discretion of the analyst.

12.2. The LC/MS/MS system is calibrated using the IS technique. Calculate analyte and Surrogate concentrations using the multiple point calibration established in the initial calibration. Do not use daily calibration verification (CCV) standard to quantitate analyte in samples (unless it is a qualitative determination and is not for reporting the data such as screening).

12.3. Prior to reporting the data, the chromatogram should be reviewed for any incorrect peak identification and poor integration.

12.4. PFBS, PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions specified in the method due to the linear and branch isomers of these compounds. Most PFAA compounds are produced by two different processes. One gives rise to linear PFAA only while the other process produces both linear and branched isomers. Both branched and linear

PFAA compounds can potentially be found in the environment. For the aforementioned compounds that give rise to more than one peak, all chromatographic peaks observed in the standard must be integrated and the areas totaled. Chromatographic peaks in the sample must be integrated in the same way as the calibration standard and concentrations reported as a total for each of these analytes.

12.4.1. A technical (qualitative) grade PFOA standard is analyzed initially, after an initial calibration when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times.

12.5. If the concentration of the analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.6. Calculations

12.6.1. Peak areas are used as a measure of response.

12.6.2. Average Response Factor, Linear fit, or quadratic fit is used for the calibration curve. Quadratic fit is used if the response is non-linear.

12.6.3. The average response of the internal standard in the calibration curve is used to correct for the response of the reported analytes.

12.6.4. The calibration is automatically performed by the Micromass MassLynx software, based on the equations:

Equation 1 Average Response Factor:

$$x = \frac{y}{RF_a}$$

Where

x = ratio of the sample concentration to the IS concentration
y = ratio of the sample response to the IS response
RF_a = average response factor

Equation 2 Linear Calibration:

$$x = \frac{(y-b)}{m}$$

Where

x = ratio of the sample concentration to the IS concentration
y = ratio of the sample response to the IS response

b = y intercept of the curve
m = slop of the curve

Equation 3 Quadratic calibration:

$$x = \frac{-b \pm \sqrt{b^2 - 4a(c-y)}}{2a}$$

Where

a = second order coefficient (curvature)
b = first order coefficient (slope)
c = zero order coefficient (intercept)
x = ratio of the sample concentration to the IS concentration
y = ratio of the sample response to the IS response

12.7. Reporting Requirements

12.7.1. Reporting limits and units are described in Section 1.5.

12.7.2. Sample results are entered into a LIMS system in accordance with current QA policies.

12.7.3. Footnotes and anomalies when applicable must be included in the data package and data reduction process. Exceeded holding times must be immediately communicated to the project managers and followed by an electronically filed non-conformance memo

13. METHOD PERFORMANCE

13.1. The laboratory must make a one time initial demonstration of capability for each individual method. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests is may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2. Initial Demonstration of Low System Background - Prepare and analyze a method blank containing preservatives using the same procedure to extract and analyze samples. Confirm that the method blank is reasonably free of contamination and that the criteria in Section 9.3 are met.

13.2.1. Anytime a new lot of SPE cartridge, solvents, centrifuge tubes, disposable pipets, and autosampler vials are used, it must be demonstrated that the background is reasonably free of contamination.

13.3. Prepare and analyze four to seven replicates of laboratory control samples (LCS) fortified near the mid range of the initial calibration curve The reagent water must contain the same preservatives used in the method.

13.3.1. Initial Demonstration of Precision (IDP) – the result of the replicate analysis must be less than 20% relative standard deviation (RSD) to be valid.

13.3.2. Initial Demonstration of Accuracy (IDA) – the average recovery of the replicate values must be within ± 30% of the true value to be valid.

Demonstration of Capability Criteria		
Matrix	**IDP**	**IDA**
Reagent Water containing preservatives and fortified at mid level.	20 % RSD	70 to 130% Recovery

13.3.3. If any analyte does not meet the acceptance criteria, then the test must be repeated. Only those analytes that did not meet criteria in the first test need to be re-evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.4. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.5. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be at or below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006 and policy CA-Q-S-006. MDL studies are reviewed and maintained in the Quality Assurance Department.

13.6. The reporting limit is validated using the procedure outlined below. The lowest calibration standard in the initial calibration and the low-level CCV must be less than or equal to the reporting limit.

13.7. Spike and analyze seven replicates at the target reporting limit concentration. Process all samples adding the preservative as well. Calculate the mean and standard deviation for these replicates. Calculate the Half-Range for the prediction of interval of results (HRPIR) as follows:

Equation 5

$$HR_{PIR} = 3.963S$$

Where S is standard deviation, and 3.963 is a constant value for 7 replicates.

13.8. Calculate the upper and lower limits for the Prediction of Interval Result.

Equation 6

$$PIR = \text{Mean Recovery} \pm HR_{PIR}$$

Equation 7

$$Upper\ PIR = \frac{Mean\ \mathrm{Recovery} + HR_{PIR}}{Fortified\ Concentraton} \leq 150\%$$

$$Lower\ \ PIR = \frac{Mean\ \mathrm{Recov}ery - HR_{PIR}}{Fortified\ \ Concentration} \geq 50\%$$

Equation 8

13.9. The MRL is validated if both the Upper and Lower PIR limits meet the criteria described above. If these criteria are not met, the MRL has either been set too low and must be determined again at a higher concentration or the preparation must be carefully repeated.

13.10. MDL fortification performed at 8.0 to 72 ng/L for instrument A6 has met criteria for Method 537A.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Assorted test tubes, autovials, disposable gloves. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Waste Acetonitrile/Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.3. Aqueous waste from the LCMS instrument contaminated with methanol and acetonitrile. This is collected in a 1-gallon carboy at the instrument. When the carboy is full or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full or after no more than 75 days, move the drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. EPA Method 537, EPA Document # EPA/600/R-08/092 537 by J.A. Shoemaker, P.E. Grimmett, and B.K. Boutin, Version 1.1, September 2009.

16.2. UCMR3 Laboratory Approval Requirements and Information Document, Technical Support Center, U.S. Envirnomental Protection Agency, Version 2.0, January 2012

16.3. SW-846 Method 8321A, "Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection", Revision 1, December 1996, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

16.4. List of other SOPs cross-referenced in SOP.

16.4.1. WS-QA-0041, Calibration and Calibration Check of Balances.

16.4.2. WS-PQA-008, Data Recording Requirements.

16.4.3. WS-PQA-003, Quality Control Program.

16.4.4. WS-QA-0023, Nonconformance and Corrective Action System

16.4.5. WS-QA-0006, Method Detection Limits and Instrument Detection

16.4.6. The HPLC Troubleshooting Wizard, LC Resources, Inc. © 2006.

17. METHOD MODIFICATIONS

17.1. None – new method.

18. ATTACHMENTS

18.1. Table 1–UCMR3 Monitoring Analytes, Surrogates, and Internal Standards for 537, its CAS No., MRL, Acronym, and Molecular Formula.

19. REVISION HISTORY

19.1. WS-DW-0004, Rev. 1.4, Effective 111/04/2016

19.1.1. Section 2.2, changed 5.0 µL to 10 µL.

19.1.2. Section 10,2.2, deleted "or $[M+H]^+$" and replaced "hormones" with "target analytes".

19.1.3. Editorial changes.

19.2. WS-DW-0004 Rev. 1.3, Effective 10/07/2016

19.2.1. Added Section 7.9.5.1 – "A technical (qualitative) grade PFOA standard which contains both linear and branched isomers is used as a retention time (RT) marker. This is used to integrate the total response for both linear and branched isomers of PFOA in environmental samples while relying on the initial calibration with the linear isomer quantitative standard."

19.2.2. Section 8.1, inserted the second sentence, "High-density polyethylene (HDPE) containers with HDPE screw caps may also be used, based on the availability of the containers." to reflect the current container availability.

19.2.3. Section 9.10 Item 7 – Added "PFOA RT marker, as needed"

19.2.4. Added Section 12.4.1 – "A technical (qualitative) grade PFOA standard is analyzed initially, after an initial calibration when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times."

19.2.5. Editorial changes.

19.3. WS-DW-0004 Rev. 1.2, Effective 09/18/2015

19.3.1. Updated copyright statement on Cover page.

19.3.2. Section 1.4 – added "…Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated."

19.3.3. Section 6.2 – changed to "Autosampler vials, 300-µL, polypropylene, with polypropylene screw caps or Agilent PTFE lined LC screw caps." And added to Note -: If Agilent PTFE lined screw caps are determined to be free from contamination they may be used.

19.3.4. Editorial changes.

19.4. WS-DW-0004 Rev. 1.1, Effective 02/01/2013

19.4.1. Section 9.8.3 – Updated LCS spiking requirements per laboratory practice.

19.4.2. Editorial changes.

19.5. WS-DW-0004 Rev. 1

19.5.1. Updated spiking levels in Section 7.9.

TABLE 1 - Method 537 Analytes, Surrogates, and Internal Standards

Analyte	CAS No.	Acronym	Molecular Formula (as anions)
Perfluorobutanesulfonate, potassium salt	29420-49-3 **375-73-5**	**PFBS**	$C_4HF_9sO_3$
Perfluorohexanesulfonate, sodium salt	82382-12-5 **355-46-4**	**PFHxS**	$C_6HF_{13}sO_3$
Perfluorooctanesulfonate, sodium salt	4021-47-0 **1763-23-1**	**PFOS**	$C_8HF_{17}SO_3$
Perfluorooctanoic acid	**335-67-1**	**PFOA**	$C_8HF_{15}O_2$
Perfluoroheptanoic acid	**375-85-9**	**PFHpA**	$C_7HF_{13}O_2$
Perfluoronanoic acid	**375-95-1**	**PFNA**	$C_9HF_{17}O_2$
Perfluorohexanoic acid	307-24-4	PFHxA	$C_6HF_{11}O_2$
Perfluorodecanoic acid	335-76-2	PFDA	$C_{10}HF_{18}O_2$
Perfluoroundecanoic acid	2058-94-8	PFUdA	$C_{11}HF_{21}O_2$
Perfluorododecanoic acid	307-55-1	PFDoA	$C_{12}HF_{23}O_2$
Perfluorotridecanoic acid	72629-94-8	PFTrDA	$C_{13}HF_{26}O_2$
Perfluorotetradecanoic acid	376-06-7	PFTeDA	$C_{14}HF_{27}O_2$
N-Methylperfluoro-1-octanesulfonamidoacetic acid	NA	N-MeFOSAA	$C_{11}H_6F_{17}NO_4S$
N-Ethylperfluoro-1-octanesulfonamidoacetic acid	NA	N-EtFOSAA	$C_{12}H_8F_{17}NO_4S$
SU1 Perfluoro-n-[1,2-$^{13}C_2$]hexanoic acid	NA	**$^{13}C_2$-PFHxA**	$^{13}C_2{}^{12}C_4HF_{11}O_2$
SU2 Perfluoro-n-[1,2-$^{13}C_2$]decanoic acid	NA	**$^{13}C_2$-PFDA**	$^{13}C_2{}^{12}C_8HF_{19}O_2$
SU3 N-deuterioethylperfluoro-1-octanesulfonamidoacetic acid	NA	d_5-N-EtFOSAA	$C_{12}D_5H_3F_{17}NO_4S$
IS1 Perfluoro-[1,2-$^{13}C_2$]octanoic acid	NA	**$^{13}C_2$-PFOA**	$^{13}C_2{}^{12}C_6HF_{15}O_2$
IS2 Perfluoro-[1,2,3,4-$^{13}C_4$]octanesulfonate, sodium salt	NA	**$^{13}C_4$-PFOS**	$^{13}C_4{}^{12}C_4HF_{17}SO_3$
IS3 N-deuteriomethylperfluoro-1-octanesulfonamidoacetic acid	NA	d_3-N-MeFOSAA	$C_{11}D_3H_3F_{17}NO_4S$

19.5.2. Editorial changes

Bold indicates the analyte are under the UCMR[3] program.