June 13, 2018

U.S. Army Corps of Engineers
New England District
696 Virginia Road
Concord, MA 01742-2571
Attn: Ms. Penelope Reddy

Subject: Work Plan for PFAS Sampling of Selected Community and Private Wells
Former Fort Devens Army Installation, Devens, MA
Contract No. W912WJ-18-C-0011

Dear Ms. Reddy,

KOMAN Government Solutions, LLC (KGS) is pleased to provide this work plan for the sampling of per- and polyfluoroalkyl substances (PFAS) from selected community and private water supply wells within or near a 1-mile radius of prior detections of PFAS in groundwater at the Former Fort Devens Army Installation in Devens, Massachusetts.

Although there is no apparent hydrogeologic connection between the locations of known detections of PFAS in groundwater at the Former Fort Devens and the community and private wells selected for sampling, this work plan is proposed at the request of the U.S. Environmental Protection Agency (EPA) as a precautionary measure. Thus, it should be noted that if PFAS are detected in any of the community or private wells, the source of the contamination may be attributable to sources other than the Former Fort Devens.

This work plan includes the selected sampling locations, regional hydrogeology discussion of potential for groundwater from Former Fort Devens to impact adjacent community and private well supplies, planned field activities, laboratory analyses, schedule, and reporting.

REGIONAL HYDROGEOLOGY

Regional groundwater flow and information available regarding geology and hydrogeology in the Devens, Massachusetts area were reviewed to assess potential impacts from PFAS to water supply wells in the surrounding areas. EPA has expressed concern regarding groundwater originating at Former Fort Devens impacting wells located across hydraulic groundwater divides such as Cold Spring Brook and Bowers Brook in Harvard and across the Nashua River in Shirley, Massachusetts. Based on the current review of site topography, overburden soil thickness, overburden soil characteristics, modeled groundwater flow contours, groundwater hydraulic gradients, and aquifer transmissivity, it is unlikely that potential PFAS impacts to these wells would be a result of groundwater originating from the Former Fort Devens; however, site-specific hydrogeology and its relation to the current understanding of the regional hydrogeology will be further investigated during a Remedial Investigation commencing in 2018.

In Shirley, no PFAS impacts are expected at the private wells located along Walker Road. Walker Road, which is on the Ayer/Shirley town line, is located west of Nashua River, and groundwater flow in this area is to the southeast toward the Nashua River. The former Main Post is located on the east side of the river. The groundwater from the former Main Post discharges to the Nashua River and is not anticipated to underflow the river to impact the private wells along Walker Road. Groundwater at Areas of Contamination (AOC) in the former North Post (AOCs 50, 30, 31, 20, and 21) is flowing toward and

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discharging to the Nashua River and is not flowing toward the residential wells located along Walker Road.

In Harvard, Massachusetts, given the location of Cold Spring Brook and Bowers Brook relative to the topographic and bedrock highs to the south and east, the presence of these brooks is attributed to the convergence of flow in the overburden aquifer. Groundwater near Barnum Road at the Former Fort Devens flows to the south and east (i.e., toward the brooks) and shallow groundwater near Ayer Road (Route 110) and Old Mill Road in Harvard presumably flows to the north and west (also toward the brooks). The topographic and bedrock highs and the drainage effect of the brooks likely provide hydraulic control and inhibit the flow of groundwater at the Barnum Road area to flow east of Cold Spring Brook; thus, there is likely no groundwater flow from the Barnum Road area to the public/private supply wells to the east along Ayer Road (Route 110) in Harvard.

SELECTED SAMPLING LOCATIONS

Community supply wells and individual developed parcels, which presumably have a private well, were selected to be sampled based on proximity of the wells or parcels to detections of PFAS in groundwater at Area of Concerns at the Former Fort Devens. Samples will be collected from fifteen community supply wells and from private wells presumably present at the thirteen developed property parcels, as listed below, to determine whether PFAS are present in those wells. The locations of the wells and parcels to be sampled are shown on **Figure 1**.

The owners of the following selected community supply wells will be contacted to request written consent to sample the wells:

- The Appleworks (PWS ID 2125007-01G)
- Foxglove Apartments (PWS ID 2125013-01G)
- Harvard Plaza (PWS ID 2125010-01G)
- Shaker Place offices (PWS ID 2125020-01G)
- Jill Realty Trust (PWS ID 2125003-01G)
- Vanguard Medical/Renaissance (PWS ID 2125012-01G)
- MCI-Shirley Well 1 (PWS ID 2270001-01G)
- MCI-Shirley Well 2 (PWS ID 2270001-02G)
- Harvard Green Condominiums Well 1 (PWS ID 2125014-01G)
- Harvard Green Condominiums Well 2 (PWS ID 2125014-02G)
- Ayer Road Properties LLC Well 1 (PWS ID 2125021-01G)
- Ayer Road Properties LLC Well 2 (PWS ID 2125021-02G)
- Ayer Road Properties LLC Well 3 (PWS ID 2125021-03G)
- Ayer Road Properties LLC Well 4 (PWS ID 2125021-04G)
- Little Leaf Farms (105 Walker Rd, Shirley) (ID to be determined)

The property owners from the selected developed parcels will be contacted to request written consent to sample their private wells:

- 309 Ayer Road, Harvard
- 313 Ayer Road, Harvard
- 354 Ayer Road, Harvard
- 42 Old Mill Road, Harvard
- 62 Old Mill Road, Harvard

- 41 Walker Road, Shirley
- 47 Walker Road, Shirley
- 63 Walker Road, Shirley
- 75 Walker Road, Shirley
- 12 Lawton Road, Shirley
- 27 Lawton Road, Shirley
- 2 Kittredge Road, Shirley
- 92 Great Road, Shirley

An example of the letter and fact sheets that will be sent to the property owners of the wells to be sampled as part of this work plan is included in **Attachment 1**.

FIELD ACTIVITIES

Well owners will be contacted by the Army to obtain consent to access their property to collect water samples from the selected wells. Field activities for sampling of the community and private supply wells will commence upon obtaining written consent from the property owner or community well water system owner. Well owners will be provided with informational PFAS fact sheets (**Attachment 1**) prepared by the Massachusetts Department of Environmental Protection (MassDEP) and EPA.

Private and community wells will be sampled according to KGS standard operating procedures (SOPs), as described in **Attachment 2**. The specific well locations, well owner contact information, description and location of source sampling tap or spigot, well purging information, and well sample collection method will be documented. Information regarding the well construction (e.g., type, depth) will be collected from available town and state databases and/or interviews of the well owners.

Samples will be collected in three 250 milliliter (mL) polypropylene bottles fitted with polypropylene screw caps. The sample containers will include 5 grams/liter (g/L) of Trizma which serves as a buffering agent to remove free chlorine that may be present in the sample. Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Aqueous samples must be extracted within 14 days and extracts should be stored at room temperature and analyzed within 28 days after extraction.

Quality control (QC) samples will include field reagent blanks (FRB), matrix spike/matrix spike duplicates (MS/MSDs), and field duplicates. FRBs will be prepared by pouring a laboratory-supplied sample bottle filled with preserved PFAS-free water into an empty (non-preserved) sample container in the field at the time samples are collected. One FRB will be collected during the community well field sampling activity and one FRB will be collected during the residential well sampling activity. Additional FRB samples may be collected if warranted by site conditions. The FRB will be shipped back to the laboratory with the samples and analyzed to ensure that PFAS were not introduced into samples during sample collection and handling. MS/MSDs will be collected at a rate of 1 per 20 samples (5%) and will be prepared by the laboratory. Field duplicate samples will be prepared at a rate of 1 per 10 samples (10%) by filling a second HDPE bottle at the time of sampling. Samples will be recorded on chains-of-custody, packed in coolers, and shipped on ice to the analytical laboratory in accordance with the 2016 Long Term Monitoring and Maintenance Program (LTMMP) QAPP. The sample register, listing field sample locations and associated quality control samples, is provided in **Table 1**.

LABORATORY ANALYSES

Laboratory analyses will be performed by Alpha Analytical, Inc. of Mansfield, Massachusetts, which is certified under Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) Quality System Manual (QSM) for Environmental Laboratories version 5.1 valid through May 2019. Water samples will be analyzed for PFAS by EPA Method 537 v1.1. EPA Method 537 uses solid-phase extraction (SPE) with liquid chromatography/tandem mass spectrometry (LC/MS/MS) analysis. The sample register is provided in **Table 1** and the target analyte list and project reporting limits are presented in **Table 2.** The laboratory's Standard Operating Procedure (SOP) and certification for PFAS analyses are provided in **Attachment 3**.

KGS will perform Tier 2B data validation in accordance with the general procedures detailed in the *Quality Assurance Project Plan for the Annual Long-Term Monitoring and Maintenance Program (LTMMP)* (KGS, August 2016). The logic outlined in *USEPA National Functional Guidelines for Organic Superfund Methods Data Review* (January 2017) will be used to apply qualifiers to PFAS data. Where specific guidance is not available, the PFAS data will be evaluated using professional judgement in a conservative manner consistent with industry standards.

SCHEDULE

Property access to the community and private wells will be coordinated following regulatory agency approval of the work plan. Written access agreements will be mailed, and authorized signatures of well owners will be obtained prior to sampling their well. Sampling will be conducted soon after access is granted, and a sampling date is coordinated with the property owner. Wells will be grouped as feasible to collect multiple samples each day and to minimize the number of mobilizations. Results will be provided within 60 days of sample collection.

REPORTING

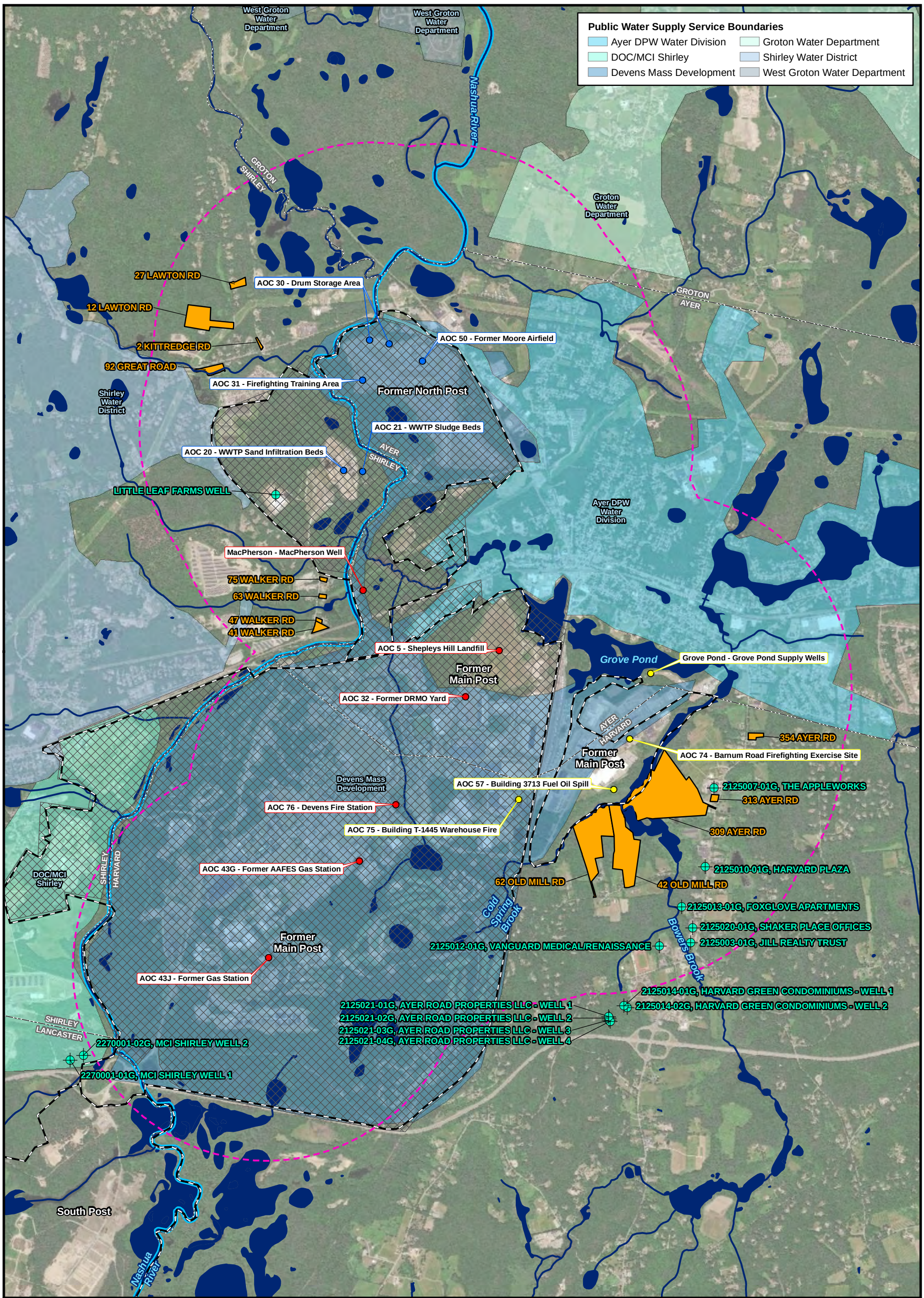
Tabulated results of the analyses will be provided to the well owners, EPA, and MassDEP within 60 days of the sampling event. Individual well owners will receive their well results for PFAS by email or hard copy. EPA and MassDEP will receive results via email for all the wells sampled.

Please contact me at (508) 219-6771 or jropp@komangs.com if you have any questions or require additional information.

Sincerely,
KOMAN Government Solutions, LLC

James Ropp, P.E.
Project Manager

cc: Robert Simeone, BRAC Devens
Mike Kulbersh, USACE
Carol Keating, EPA Region 1
David Chaffin, MassDEP
KGS File
Dan Groher, USACE
Yixian Zhang, USACE
Laurie O'Connor, EPA Region 1
Ron Ostrowski, MassDevelopment



Legend

- Select Public Supply Well
- Area 1 - Grove Pond Supply Wells, AOCs 57, 74, and 75 to be addressed in the RI
- Area 2 - MacPherson Water Supply Well, SHL, AOCs 32, 76, 43G and 43J to be addressed in the RI
- Area 3 - AOCs 20, 21, 30, 31, and 50 to be addressed in the RI
- Select Parcel with Private Well
- 1-Mile Buffer
- Devens - Census Designated Place
- City/Town Boundary
- NHD Waterbody
- NHD Stream/River
- Nashua River
- Former Fort Devens Boundary

N W E S

0 1,250 2,500 Feet

Proposed Community and Private Wells to be Sampled
Work Plan for PFAS Sampling of Select Community and Private Wells

Former Army Installation Devens
Devens, Massachusetts

KOMAN Government Solutions, LLC
293 Boston Post Road West, Suite 100, Marlborough, MA 01752

Date: 06/12/2018

Figure 1

KGS

File: PFAS2018_ComPrivWells.mxd

Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGRID, IGN, and the GIS User Community

Table 1
PFAS Sampling of Community and Residential Well Locations and Method
Former Fort Devens Army Installation, Devens, MA

Community and Private Well Locations			PFAS Target List (EPA Method 537.1)	QC Sample	Date/Time Sampled
Well Type	**Field Sample ID**	**Well Street Address**	3-250 ml poly preserved with Trizma		
Private	309-AR-HAR	309 Ayer Rd, Harvard	X		
	313-AR-HAR	313 Ayer Rd, Harvard	X		
	354-AR-HAR	354 Ayer Rd, Harvard	X		
	42-OMR-HAR	42 Old Mill Rd, Harvard	X		
	62-OMR-HAR	62 Old Mill Rd, Harvard	X		
	HAR-DUP-01		X	FD	
	HAR-FRB-01		X	FRB	
	12-LR-SHI	12 Lawton Rd, Shirley	X		
	27-LR-SHI	27 Lawton Rd, Shirley	X		
	2-KR-SHI	2 Kittredge Rd, Shirley	X		
	92-GR-SHI	92 Great Rd, Shirley	X		
	SHI-DUP-04		X	FD	
	41-WR-SHI	41 Walker Rd, Shirley	X	MS/MSD	
	47-WR-SHI	47 Walker Rd, Shirley	X		
	63-WR-SHI	63 Walker Rd, Shirley	X		
	75-WR-SHI	75 Walker Rd, Shirley	X		
Community	2125007-01G-HAR	325 Ayer Rd, Harvard	X		
	2125013-01G-HAR	253 Ayer Rd, Harvard	X		
	2125010-01G-HAR	275-285 Ayer Rd, Harvard	X		
	2125020-01G-HAR	233 Ayer Rd, Harvard	X		
	2125003-01G-HAR	231 Ayer Rd, Harvard	X		
	2125012-01G-HAR	12-16 Lancaster County Rd, Harvard	X	MS/MSD	
	HAR-DUP-02		X	FD	
	HAR-FRB-02		X	FRB	
	LLFARM-SHI	105 Walker Rd, Shirley	X		
	2270001-01G-LAN	Shirley Rd & Shaker Rd, Lancaster	X		
	2270001-02G-LAN	Shirley Rd & Shaker Rd, Lancaster	X		
	2125014-01G-HAR	35 Lancaster County Rd, Harvard	X		
	2125014-02G-HAR	35 Lancaster County Rd, Harvard	X		
	2125021-01G-HAR	69 Lancaster County Rd, Harvard	X		
	2125021-02G-HAR	69 Lancaster County Rd, Harvard	X		
	HAR-DUP-03		X	FD	
	HAR-FRB-03		X	FRB	
	2125021-03G-HAR	69 Lancaster County Rd, Harvard	X		
	2125021-04G-HAR	69 Lancaster County Rd, Harvard	X		

Notes:
HAR - Harvard
GRO - Groton
LAN - Lancaster
SHI - Shirley
FD - field duplicate
FRB - field reagent blank
MS/MSD - matrix spike/matrix spike duplicate

Table 2
PFAS Target Compound List
PFAS Sampling of Community and Residential Wells
Former Fort Devens Army Installation, Devens, MA

Analyte	CAS Number	RL (ng/L)	LOD (ng/L)	MDL (ng/L)
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	2.0	0.80	0.225
Perfluorooctanoic acid (PFOA)	335-67-1	2.0	0.80	0.261
Perfluorononanoic acid (PFNA)	375-95-1	2.0	0.80	0.257
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	2.0	0.80	0.328
Perfluoroheptanoic acid (PFHpA)	375-85-9	2.0	0.80	0.238
Perfluorobutanesulfonic acid (PFBS)	375-73-5	2.0	1.60	0.650
Perfluorodecanoic acid (PFDA)	335-76-2	2.0	0.80	0.288
Perfluorododecanoic acid (PFDoA)	307-55-1	2.0	0.80	0.284
Perfluorohexanoic acid (PFHxA)	307-24-4	2.0	1.60	0.404
Perfluorotridecanoic Acid (PFTriA)	72629-94-8	2.0	1.60	0.576
Perfluorotetradecanoic acid (PFTeA)	376-06-7	2.0	1.60	0.515
Perfluoroundecanoic acid (PFUnA or PFUdA)	2058-94-8	2.0	0.80	0.218
N-ethyl perfluorooctane sulfonamidoacetic acid (NEtFOSAA)	2991-50-6	2.0	1.60	0.595
N-methyl perfluorooctane sulfonamidoacetic acid (NMeFOSAA)	2355-31-9	2.0	1.60	0.636

Source: Alpha Analytical reporting limits for Method 537.1 (May 14, 2018).

Notes:
ng/L = nanograms per liter
RL = Reporting Limit
LOD = Limit of Detection
MDL = Method Detection Limit

ATTACHMENT 1

EXAMPLE PUBLIC LETTER AND

PFAS INFORMATIONAL FACT SHEETS

DEPARTMENT OF THE ARMY
FORT DEVENS
BASE REALIGNMENT AND CLOSURE OFFICE
30 QUEBEC STREET
DEVENS MA 01434

June x, 2018

Name of Owner
Street Address
Town, State Zip

SUBJECT: Sampling of Drinking Water Well(s)

Dear x:

The Army has detected per- and polyfluoroalkyl (PFAS) compounds in groundwater at some locations of the Former Fort Devens, Devens, Massachusetts. PFAS is a class of chemicals used in firefighting foams as well as several commercial and common household products. Additional information regarding PFAS compounds can be found in the attached fact sheets prepared by the United States Environmental Protection Agency (EPA) and the Massachusetts Department of Environmental Protection (MassDEP).

Although there is no apparent hydrogeologic connection between your water well and the locations of PFAS in groundwater at the Former Fort Devens, the EPA has requested that various wells in the surrounding communities such as yours be sampled out of an abundance of caution.

The Army will sample your well at no cost to you and will provide you the analytical results within 60 days of sampling. Please contact Jim Ropp, our contractor, at Koman Government Solutions (KGS) at jropp@komangs.com or (508) 219-6771 if you would like your well sampled and fill out the attached right of entry form to allow the sampling to be performed, and send it back to the U.S. Army Corps of Engineers in the pre-paid envelope provided. KGS will then contact you to arrange a convenient time to sample your water from an existing tap or spigot. The sample collection should take less than 30 minutes.

If you have any questions or concerns, please do not hesitate to contact me by phone at 978-796-2205 or via email at robert.j.simeone.civ@mail.mil or Penelope Reddy, United States Army Corps of Engineers, at 978-318-8160 or via email at penelope.reddy@usace.army.mil. The Army has also notified the local board of health regarding this sampling effort.

Sincerely,

Robert Simeone.
Army Base Realignment and Closure
Environmental Coordinator

Copies Furnished:

Bridgette Bradley, District Sanitarian, Nashoba Associated Board of Health Agent
David Chaffin, Massachusetts Department of Environmental Protection
John Howlett II, Chair Board of Health, Town of Shirley, Massachusetts
Ira Grossman, District Sanitarian, Nashoba Associated Board of Health Agent
Libby Levison, Chair, Board of Health Town of Harvard, Massachusetts
Carol Keating, United States Environmental Protection Agency
~~Laurie O'Connor, United States Environmental Protection Agency~~
Pamela Papineau, Chair, Board of Health, Town of Ayer, Massachusetts
Penelope Reddy, U.S. Army Corps of Engineers
Jim Ropp, Koman Government Solutions

DEPARTMENT OF THE ARMY
RIGHT-OF-ENTRY

PROJECT NAME; BRAC Former Fort Devens **ROE#**
LOCATION: (fill in town), (fill in county), Massachusetts
ASSESSORS PARCEL NUMBER(s): Map X, Lot X

The undersigned, hereinafter called the "Owner," hereby grant to the UNITED STATES OF AMERICA, hereinafter called the "Government," a right-of-entry upon the following terms and conditions:

1. The Owner hereby grants to the Government the right to enter upon the lands hereinafter described for a one time sampling event of the on-site drinking water well or wells as arranged with the property owner.

2. All tools, equipment, and other property taken upon or placed upon the land by the Government shall remain the property of the Government and may be removed by the Government at any time during the sampling of the well.

3. If any action of the Government's employees or agents in the exercise of this right-of-entry results in damage to the real property, the Government will, in its sole discretion, either repair such damage or make an appropriate settlement with the owner. In no event shall such repair or settlement exceed the fair market value of the fee title to the real property at the time immediately preceding such damage. The Government's liability under this clause is subject to the availability of for such payment, and nothing contained in this agreement may be considered as implying that Congress will at a later date appropriate funds sufficient to meet deficiencies. The provisions of this clause are without prejudice to any rights the Owner may have to make a claim under applicable laws for any other damages than provided herein.

4. The land affected by this right-of-entry is located in (fill in Town, in Massachusetts) x at X Road/Street/Avenue and is presently owned by (fill in owner).

5. All on-site activities will be coordinated in advance with the owner.

WITNESS MY HAND AND SEAL this _____day of _____, 2018.

Owner's Names: Fill in

Mailing Address: X Avenue, Town, Massachusetts

Owner's Phone #: _____
E-mail: _____

Signature: _____

UNITED STATES OF AMERICA

_____
Aaron K. Matthews
Acting District Chief of Real Estate
Real Estate Contracting Officer

United States Environmental Protection Agency

FACT SHEET
PFOA & PFOS Drinking Water Health Advisories

Overview

EPA has established health advisories for PFOA and PFOS based on the agency's assessment of the latest peer-reviewed science to provide drinking water system operators, and state, tribal and local officials who have the primary responsibility for overseeing these systems, with information on the health risks of these chemicals, so they can take the appropriate actions to protect their residents. EPA is committed to supporting states and public water systems as they determine the appropriate steps to reduce exposure to PFOA and PFOS in drinking water. As science on health effects of these chemicals evolves, EPA will continue to evaluate new evidence.

Background on PFOA and PFOS

PFOA and PFOS are fluorinated organic chemicals that are part of a larger group of chemicals referred to as perfluoroalkyl substances (PFASs). PFOA and PFOS have been the most extensively produced and studied of these chemicals. They have been used to make carpets, clothing, fabrics for furniture, paper packaging for food and other materials (e.g., cookware) that are resistant to water, grease or stains. They are also used for firefighting at airfields and in a number of industrial processes.

Because these chemicals have been used in an array of consumer products, most people have been exposed to them. Between 2000 and 2002, PFOS was voluntarily phased out of production in the U.S. by its primary manufacturer. In 2006, eight major companies voluntarily agreed to phase out their global production of PFOA and PFOA-related chemicals, although there are a limited number of ongoing uses. Scientists have found PFOA and PFOS in the blood of nearly all the people they tested, but these studies show that the levels of PFOA and PFOS in blood have been decreasing. While consumer products and food are a large source of exposure to these chemicals for most people, drinking water can be an additional source in the small percentage of communities where these chemicals have contaminated water supplies. Such contamination is typically localized and associated with a specific facility, for example, an industrial facility where these chemicals were produced or used to manufacture other products or an airfield at which they were used for firefighting.

EPA's 2016 Lifetime Health Advisories

EPA develops health advisories to provide information on contaminants that can cause human health effects and are known or anticipated to occur in drinking water. EPA's health advisories are non-enforceable and non-regulatory and provide technical information to states agencies and other public health officials on health effects, analytical methodologies, and treatment technologies associated with drinking water contamination. In 2009, EPA published provisional health advisories for PFOA and PFOS based on the evidence available at that time. The science has evolved since then and EPA is now replacing the 2009 provisional advisories with new, lifetime health advisories.

FACT SHEET
PFOA & PFOS Drinking Water Health Advisories

EPA's 2016 Lifetime Health Advisories, continued

To provide Americans, including the most sensitive populations, with a margin of protection from a lifetime of exposure to PFOA and PFOS from drinking water, EPA established the health advisory levels at 70 parts per trillion. When both PFOA and PFOS are found in drinking water, the combined concentrations of PFOA and PFOS should be compared with the 70 parts per trillion health advisory level. This health advisory level offers a margin of protection for all Americans throughout their life from adverse health effects resulting from exposure to PFOA and PFOS in drinking water.

How the Health Advisories were developed

EPA's health advisories are based on the best available peer-reviewed studies of the effects of PFOA and PFOS on laboratory animals (rats and mice) and were also informed by epidemiological studies of human populations that have been exposed to PFASs. These studies indicate that exposure to PFOA and PFOS over certain levels may result in adverse health effects, including developmental effects to fetuses during pregnancy or to breastfed infants (e.g., low birth weight, accelerated puberty, skeletal variations), cancer (e.g., testicular, kidney), liver effects (e.g., tissue damage), immune effects (e.g., antibody production and immunity), thyroid effects and other effects (e.g., cholesterol changes).

EPA's health advisory levels were calculated to offer a margin of protection against adverse health effects to the most sensitive populations: fetuses during pregnancy and breastfed infants. The health advisory levels are calculated based on the drinking water intake of lactating women, who drink more water than other people and can pass these chemicals along to nursing infants through breastmilk.

Recommended Actions for Drinking Water Systems

Steps to Assess Contamination

If water sampling results confirm that drinking water contains PFOA and PFOS at individual or combined concentrations greater than 70 parts per trillion, water systems should quickly undertake additional sampling to assess the level, scope and localized source of contamination to inform next steps

Steps to Inform

If water sampling results confirm that drinking water contains PFOA and PFOS at individual or combined concentrations greater than 70 parts per trillion, water systems should promptly notify their State drinking water safety agency (or with EPA in jurisdictions for which EPA is the primary drinking water safety agency) and consult with the relevant agency on the best approach to conduct additional sampling.

Drinking water systems and public health officials should also promptly provide consumers with information about the levels of PFOA and PFOS in their drinking water. This notice should include specific information on the risks to fetuses during pregnancy and breastfed and formula-fed infants from exposure to drinking water with an individual or combined concentration of PFOA and PFOS above EPA's health advisory level of 70 parts per trillion. In addition, the notification should include actions they are taking and identify options that consumers may consider to reduce risk such as seeking an alternative drinking water source, or in the case of parents of formula-fed infants, using formula that does not require adding water.

FACT SHEET
PFOA & PFOS Drinking Water Health Advisories

Recommended Actions for Drinking Water Systems, continued

Steps to Limit Exposure

A number of options are available to drinking water systems to lower concentrations of PFOA and PFOS in their drinking water supply. In some cases, drinking water systems can reduce concentrations of perfluoroalkyl substances, including PFOA and PFOS, by closing contaminated wells or changing rates of blending of water sources. Alternatively, public water systems can treat source water with activated carbon or high pressure membrane systems (e.g., reverse osmosis) to remove PFOA and PFOS from drinking water. These treatment systems are used by some public water systems today, but should be carefully designed and maintained to ensure that they are effective for treating PFOA and PFOS. In some communities, entities have provided bottled water to consumers while steps to reduce or remove PFOA or PFOS from drinking water or to establish a new water supply are completed.

Many home drinking water treatment units are certified by independent accredited third party organizations against American National Standards Institute (ANSI) standards to verify their contaminant removal claims. NSF International (NSF®) has developed a protocol for NSF/ANSI Standards 53 and 58 that establishes minimum requirements for materials, design and construction, and performance of point-of-use (POU) activated carbon drinking water treatment systems and reverse osmosis systems that are designed to reduce PFOA and PFOS in public water supplies. The protocol has been established to certify systems (e.g., home treatment systems) that meet the minimum requirements. The systems are evaluated for contaminant reduction by challenging them with an influent of 1.5±30% µg/L (total of both PFOA and PFOS) and must reduce this concentration by more than 95% to 0.07 µg/L or less (total of both PFOA and PFOS) throughout the manufacturer's stated life of the treatment system. Product certification to this protocol for testing home treatment systems verifies that devices effectively reduces PFOA and PFOS to acceptable levels.

Other Actions Relating to PFOA and PFOS

Between 2000 and 2002, PFOS was voluntarily phased out of production in the U.S. by its primary manufacturer, 3M. EPA also issued regulations to limit future manufacturing, including importation, of PFOS and its precursors, without first having EPA review the new use. A limited set of existing uses for PFOS (fire resistant aviation hydraulic fluids, photography and film products, photomicrolithography process to produce semiconductors, metal finishing and plating baths, component of an etchant) was excluded from these regulations because these uses were ongoing and alternatives were not available.

In 2006, EPA asked eight major companies to commit to working toward the elimination of their production and use of PFOA, and chemicals that degrade to PFOA, from emissions and products by the end of 2015. All eight companies have indicated that they have phased out PFOA, and chemicals that degrade to PFOA, from emissions and products by the end of 2015. Additionally, PFOA is included in EPA's proposed Toxic Substance Control Act's Significant New Use Rule (SNUR) issued in January 2015 which will ensure that EPA has an opportunity to review any efforts to reintroduce the chemical into the marketplace and take action, as necessary, to address potential concerns.

FACT SHEET
PFOA & PFOS Drinking Water Health Advisories

Other Actions Relating to PFOA and PFOS, continued

EPA has not established national primary drinking water regulations for PFOA and PFOS. EPA is evaluating PFOA and PFOS as drinking water contaminants in accordance with the process required by the Safe Drinking Water Act (SDWA). To regulate a contaminant under SDWA, EPA must find that it: (1) may have adverse health effects; (2) occurs frequently (or there is a substantial likelihood that it occurs frequently) at levels of public health concern; and (3) there is a meaningful opportunity for health risk reduction for people served by public water systems.

EPA included PFOA and PFOS among the list of contaminants that water systems are required to monitor under the third Unregulated Contaminant Monitoring Rule (UCMR 3) in 2012. Results of this monitoring effort are updated regularly and can be found on the publicly-available National Contaminant Occurrence Database (NCOD) (https://www.epa.gov/dwucmr/occurrence-data-unregulated-contaminant-monitoring-rule#3). In accordance with SDWA, EPA will consider the occurrence data from UCMR 3, along with the peer reviewed health effects assessments supporting the PFOA and PFOS Health Advisories, to make a regulatory determination on whether to initiate the process to develop a national primary drinking water regulation.

In addition, EPA plans to begin a separate effort to determine the range of PFAS for which an Integrated Risk Information System (IRIS) assessment is needed. The IRIS Program identifies and characterizes the health hazards of chemicals found in the environment. IRIS assessments inform the first two steps of the risk assessment process: hazard identification, and dose-response. As indicated in the 2015 IRIS Multi-Year Agenda, the IRIS Program will be working with other EPA offices to determine the range of PFAS compounds and the scope of assessment required to best meet Agency needs. More about this effort can be found at https://www.epa.gov/iris/iris-agenda.

Non-Drinking Water Exposure to PFOA and PFOS

These health advisories only apply to exposure scenarios involving drinking water. They are not appropriate for use, in identifying risk levels for ingestion of food sources, including: fish, meat produced from livestock that consumes contaminated water, or crops irrigated with contaminated water.

The health advisories are based on exposure from drinking water ingestion, not from skin contact or breathing. The advisory values are calculated based on drinking water consumption and household use of drinking water during food preparation (e.g., cooking or to prepare coffee, tea or soup). To develop the advisories, EPA considered non-drinking water sources of exposure to PFOA and PFOS, including: air, food, dust, and consumer products. In January 2016 the Food and Drug Administration amended its regulations to no longer allow PFOA and PFOS to be added in food packaging, which will likely decrease one source of non-drinking water exposure.

Where Can I Learn More?

- EPA's Drinking Water Health Advisories for PFOA and PFOS can be found at: https://www.epa.gov/ground-water-and-drinking-water/drinking-water-health-advisories-pfoa-and-pfos
- PFOA and PFOS data collected under EPA's Unregulated Contaminant Monitoring Rule are available: https://www.epa.gov/dwucmr/occurrence-data-unregulated-con taminant-monitoring-rule
- EPA's stewardship program for PFAS related to TSCA: https://www.epa.gov/assessing-and-managing-chemicals-under-tsca/and -polyfluoroalkyl-substances-pfass-under-tsca
- EPA's research activities on PFASs can be found at: http://www.epa.gov/chemical-research/perfluorinated-chemical-pfc-research
- The Agency for Toxic Substances and Disease Registry's Perflourinated Chemicals and Your Health webpage at: http://www.atsdr.cdc.gov/PFC/

United States Environmental Protection Agency

PFAS in Drinking Water: Questions and Answers for Consumers

Introduction

This fact sheet is intended to inform you about Per- and Polyfluoroalkyl Substances (PFAS) and provide guidance on health protective limits for these chemicals in drinking water.

What are PFAS and how are people exposed to them?

PFAS are fluorinated organic chemicals. Two PFAS chemicals, perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) have been the most extensively produced and studied of these chemicals. PFAS are contained in firefighting foams, which have been used in training exercises and to extinguish oil and gas fires at a variety of locations including airfields. PFAS are also used in a number of industrial processes and have been used to make carpets, clothing, fabrics for furniture, paper packaging for food and other materials (e.g., cookware) that are resistant to water, grease or stains. Because these chemicals have been used in many consumer products, most people have been exposed to them.

While consumer products and food are the largest source of exposure to these chemicals for most people, drinking water can be an additional source of exposure in communities where these chemicals have contaminated water supplies. Such contamination is typically localized and associated with a specific facility, for example, an airfield at which they were used for firefighting or a facility where these chemicals were produced or used.

What are the levels of concern?

There are no enforceable federal or Massachusetts state standards for these substances in public drinking water. However, in June 2018, MassDEP issued a public health guideline to address five PFAS chemicals. These include PFOA, PFOS, perfluorononanoic acid (PFNA), perfluorohexanesulfonic acid (PFHxS) and perfluoroheptanoic acid (PFHpA). This type of guideline, known as an Office of Research and Standards Guideline (ORSG), provides recommended contaminant levels in drinking water and is set to be protective against adverse health effects for all people consuming the water for a lifetime. MassDEP recommends that this ORSG also apply to shorter-term exposures of weeks to months during pregnancy and breast-feeding. Based on this ORSG, MassDEP recommends that:

1) consumers in sensitive subgroups (pregnant women, nursing mothers and infants) not consume water when the level of the five PFAS substances, individually or in combination, is above 0.070 micrograms per liter (µg/L) or 70 parts per trillion (ppt); and,
2) public water suppliers take steps expeditiously to lower levels of the five PFAS, individually or in combination, to below 70 ppt for all consumers.

In May 2016, the United States Environmental Protection Agency (EPA) issued a lifetime Health Advisory (HA) of 0.070 µg/L (70 ppt) for any combination of PFOA and PFOS. The June 2018 MassDEP ORSG and associated recommendations were developed out of an abundance of caution because the five PFAS compounds included in the ORSG share very similar chemical structures and the available data indicates they most likely exhibit similar toxicities.

What health effects are associated with exposure to PFAS?

EPA's 2016 Health Advisory values for PFOS and PFOA were based on recent studies of these substances in laboratory animals and were also informed by studies of exposed people. Overall, these studies indicate that exposure to sufficiently elevated levels of PFOA and PFOS may cause developmental effects in fetuses during pregnancy and in breastfed infants. Effects on the thyroid, the liver, kidneys, hormone levels and the immune system have also been reported. Some studies suggest a cancer risk may exist in people exposed to levels well above the EPA Health Advisory.

It is important to note that consuming water with PFAS above the 70 ppt level does not mean that adverse effects will occur. The degree of risk depends on the level of the chemicals and the duration of exposure. The 70 ppt level assumes that individuals drink only contaminated water, which typically overestimates exposure, and are also exposed to PFAS

from sources beyond drinking water, such as food. To enhance safety, several uncertainty factors are additionally applied to account for the differences between animals and humans and the differences from one human to another human. Scientists are still working to study and better understand the health risks posed by exposures to PFAS. If your water has been found to have PFAS and you have specific health concerns, you may wish to consult with your doctor.

How can I find out about contaminants in my drinking water?

If you get your water from a public water system you should contact them for this information. For a contact list for all public water systems in the Commonwealth you may visit: https://www.mass.gov/lists/drinking-water-health-safety#contacts then under "Contacts" click on "MA Public Water Supplier contacts sorted By Town".

For private well owners, MassDEP recommends the use of a state certified analytical laboratory for all water quality testing. Local Private Well Regulations may specify the use of a state certified lab. A searchable list of MassDEP certified labs can be found at: http://eeaonline.eea.state.ma.us/DEP/Labcert/Labcert.aspx

What options should be considered when PFAS in drinking water is above MassDEP's recommendations?

- ✓ Sensitive subgroups, including pregnant women, nursing mothers and infants, should use bottled water that has been tested for PFAS, for drinking and cooking of foods that absorb water (like pasta).
- ✓ The water should not be used to make infant formula. Bottled water that has been tested for PFAS, or formula that does not require adding water, should be used.
- ✓ For older children and adults, the 70 ppt value is applicable to a lifetime of consuming the water. For these groups, shorter duration exposures present less risk. However, if you are concerned about your exposure while steps are taken to assess and lower the PFAS concentration in your drinking water, use of bottled water that has been tested for PFAS will reduce your exposure.
- ✓ Water contaminated with PFAS can be treated by home water treatment systems that are certified to remove PFAS by an independent testing group such as NSF, UL, Water Quality Association or the CSA Group. These may include point of entry systems, which treat all the water entering a home, or point of use devices, which treat water where it is used, such as at a faucet.
- ✓ In most situations the water can be safely used for washing foods, brushing teeth, bathing and showering. If you have cuts or broken skin, you may want to avoid long showers or baths. If you are concerned about your exposure, even though the risk is very low, you may want to use bottled water for brushing your teeth and cleaning items like dentures, pacifiers, and fruits and vegetables.

- **NOTE ON BOILING WATER:** Boiling water will not destroy these chemicals and will increase their levels somewhat due to water evaporation.
- **NOTE ON BOTTLED WATER:** Even though bottlers are not required to test for PFAS, some bottlers have tested. The best way to know if the bottled water you are drinking or plan to drink has been tested for PFAS is to contact the bottler and ask for the latest testing results. Contact information should be available on the bottle or you may need to search the internet.

Where can I get more information on PFAS?

MassDEP's Office of Research and Standards Guideline for PFAS can be found at: https://www.mass.gov/lists/contaminants#pfas-per-and-polyfluoroalkyl-substances-including-pfos-and-pfoa-

EPA's Drinking Water Health Advisories for PFOA and PFOS can be found at: https://www.epa.gov/ground-water-and-drinking-water/drinking-water-health-advisories-pfoa-and-pfos

The Centers for Disease Control and Prevention's Public Health Statement for PFOS and PFOA can be found at: https://www.atsdr.cdc.gov/pfas/index.html

For additional information on possible health effects, you may contact the Massachusetts Department Environmental Protection, Office of Research and Standards at 617-556-1165.

For information on the MassDEP Drinking Water Program, you may visit https://www.mass.gov/drinking-water-program or contact the program at program.director-dwp@state.ma.us or 617-292-5770.

ATTACHMENT 2

KGS STANDARD OPERATING PROCEDURES

KOMAN Government Solutions, LLC	STANDARD OPERATING PROCEDURE (GUIDANCE)	Number SOP-F009	Page 1 of 3
		Effective Date 15 October 2017	Revision 0
		Applicability KOMAN Government Solutions, LLC	
		Prepared by: Robert Gregory	
Subject: **PFAS SAMPLING**		Approved by: Stephen Deeter	

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1.0 PURPOSE

The purpose of this document is to provide methods, procedures, and guidance for sampling of Per- and Polyfluoroalkyl Substances (PFAS) analysis. Personnel performing PFAS sampling should refer to the appropriate media sampling SOP (e.g., soil, groundwater, sediment, surface water, etc.).

2.0 SCOPE AND APPLICABILITY

This procedure is applicable for sampling efforts for PFAS and where no project/program-specific plan or procedure is in place to direct those activities.

3.0 RESPONSIBILITIES

Personnel performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e., calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

4.0 PROCEDURE

This set of procedures outlines the general steps for collection of samples consistent with the approach for conventional sample collection. Personnel performing PFAS sampling should refer to the appropriate media sampling SOP (e.g., soil, groundwater, sediment, surface water, etc.).

In acknowledgement of the widespread presence of PFAS in the environment (e.g., common household items, packaging, clothing, waterproof paper and pens, etc.) as well as their persistence, significant additional precautions must be taken by sampling personnel to avoid field cross-contamination of environmental samples during PFAS sampling efforts, as follows:

- Sample personnel should not use Post-it Notes® - style adhesive paper products at any time during sample handling, or mobilization/demobilization.
- Sample personnel should wear only old, well laundered (at least six washings since purchase) clothing.
- Sample personnel should not wear water resistant clothing prior to or during sample collection. Tyvek®-style protective clothing must not be worn during sample handling.
- Nitrile glove must be worn at all times while collecting and handling samples.
- Many food and snack products are packaged in wrappers treated with perfluorochemicals. Therefore, hands will be thoroughly washed after handling fast food, carryout food, or snacks.
- Pre-wrapped food or snacks (such as candy bars, microwave popcorn, etc.) must not be in the possession of the sampling personnel during sampling.
- Blue ice must not be used to cool samples or be used in sample coolers.

The following table provides a more detailed summary of items that are likely to contain PFAS and therefore should not be used by sampling teams during sampling efforts. In addition, the

table provides suggestions for items allowable for use as alternatives to items potentially containing PFAS.

Category	**Prohibited Items**	**Allowable Items**
Pumps and Tubing	Polytetrafluoroethylene (PTFE), Teflon®, and other fluoropolymer containing materials. Grundfos™ submersible electric pumps contain Teflon®, and therefore should not be used for purging or sampling.	High-density polyethylene (HDPE), low density polyethylene (LDPE), or silicone tubing. Peristaltic pump or stainless-steel submersible pump (i.e., Proactive Mega-Monsoon 12-volt electric, SamplePro bladder pump, etc.).
Decontamination	Decon 90 Liquid Detergent.	Alconox® or Liquinox®, potable water followed by deionized rinse.
Sample Storage and Preservation	LDPE or glass bottles, PTFE-or Teflon®-lined caps, chemical ice packs.	Laboratory-provided sample container - *preferred* or HDPE bottles, regular ice.
Field Documentation	Waterproof/treated paper or field books, plastic clipboards, Sharpie®-type markers, Post-It® and other adhesive paper products.	Plain Paper, metal clipboard, pens.
Clothing	Clothing or boots made of or with Gore-Tex™ or other synthetic water resistant and/or stain resistant materials, coated Tyvek® material.	Synthetic or cotton material, previously laundered clothing (preferably previously washed greater than six times) without the use of fabric softeners.
Personal Care Products (for day of sample collection)	Cosmetics, moisturizers, hand cream and other related products.	<u>Sunscreens</u>: Alba Organics Natural Yes to Cucumbers Aubrey Organics Jason Natural Sun Block Kiss My Face Baby-safe sunscreens (‘free’ or ‘natural’) <u>Insect Repellents</u>: Jason Natural Quit Bugging Me Repel Lemon Eucalyptus Herbal Armor California Baby Natural Bug Spray BabyGanics <u>Sunscreen and Insect Repellents</u>: Avon Skin So Soft Bug Guard-SPF 30.
Food and Beverage	Pre-packaged food, fast food wrappers or containers.	Bottled water or hydration drinks.

5.0 REFERENCES

U.S. Environmental Protection Agency, 2016. *Technical Advisory-Laboratory Analysis of Drinking Water Samples for Perfluorooctanoic Acid (PFOA) Using EPA Method 537 Rev. 1.1*. EPA 815-B-16-021. September.

U.S. Navy Facilities Engineering Command, 2017. *Interim Per- and Polyfluoroalkyl Substances (PFAS) Site Guidance for NAVFAC Remedial Project Managers (RPMs)*. September Update.

KOMAN Government Solutions, LLC	STANDARD OPERATING PROCEDURE (GUIDANCE)	Number SOP-F016	Page 1 of 4
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1.0 PURPOSE

The purpose of this procedure is to describe the equipment and operations used to collect samples representative of potable private wells and public water supply wells. The procedures are designed to reduce the bias of system related variables (pumps, piping, holding tanks, etc.).

2.0 SCOPE AND APPLICABILITY

Potable water supply investigations are usually conducted as part of a larger investigation such as a spill, leaking tanks, nearby contaminated site, etc. However, an investigation may be conducted independently of a potential contamination source. Potable water supply investigations may include collecting samples directly from public supply wells, distribution systems, private residential wells, etc.

Sampling personnel are responsible for performing the applicable tasks and procedures outlined herein when conducting work related to environmental projects. The Project Leader or an approved designee is responsible for ensuring that performance standards specified by this SOP are achieved. The same sampling techniques used for sampling of other environmental media, including thorough documentation of location, date, time, etc., are to be used during potable water supply sampling.

Special procedures apply when a sample is collected from a private or public potable water supply. All residents will be contacted prior to sampling. At residence, identify yourself and interview the property owner for general well information including: well location, well depth, age, past sample results, holding tank capacity, if water filtration or conditioning unit is used and location and description of septic system in relation to well. Investigators should always obtain the following information from the residents and/or owners in the event contaminants are detected in the sample:

- Resident's and/or owner's name
- Resident's and/or owner's mailing address
- Resident's and/or owner's home and work telephone numbers

The contact information is required in order that the residents or water supply owner/operators can be informed of the need/schedule of sampling and to receive the results of the sampling program.

3.0 SAMPLING SITE SELECTION

The following should be considered when choosing the location to collect a potable water sample:

- Taps selected for sample collection should be supplied with water from a service pipe connected directly to a water main in the segment of interest.
- Whenever possible, choose the tap closest to the water source, and prior to the water lines entering the residence, office, building, etc., and prior to any holding, pressurization or treatment filter systems or water conditioning tanks.
- The sampling tap must be protected from exterior contamination associated with being too

close to a sink bottom or to the ground. Contaminated water or soil from the faucet exterior may enter the bottle during the collection procedure because it is difficult to place a bottle under a low tap without grazing the neck interior against the outside faucet surface. If the tap is too close to the ground for direct collection into the appropriate container, it is acceptable to use a smaller (clean) container to transfer sample to a larger container. The smaller container should be made of glass or stainless steel or other material appropriate for the target constituent list.

- Leaking taps that allow water to discharge from around the valve stem handle and down the outside of the faucet or taps in which water tends to run up on the outside of the lip, are to be avoided as sampling locations.
- Disconnect any hoses, filters, or aerators attached to the tap before sampling. These devices can harbor a bacterial population if they are not routinely cleaned or replaced when worn or cracked.
- Taps where the water flow is not constant should be avoided because temporary fluctuation in line pressure may cause clumps of microbial growth that are lodged in a pipe section or faucet connection to break loose. A smooth flowing water stream at moderate pressure without splashing should be used. The sample should be collected without changing the water flow. It may be appropriate to reduce the flow for the volatile organic compounds aliquot to minimize sample agitation.

Occasionally, samples are collected to determine the contribution of system related variables (e.g., transmission pipes, water coolers, water heaters, holding tanks, pressurization tanks, etc.) to the quality of potable water supplies. In these cases, it may be necessary to ensure that the water source has not been used for a specific time interval (e.g., over a weekend or a three- or four-day holiday period). Sample collection may consist of collecting a sample of the initial flush, collecting a sample after several minutes, and collecting another sample after the system being investigated has been completely purged.

When sampling for bacterial content, the sample container should not be rinsed before use because of possible contamination of the sample container or removal of any dechlorinating agents (if used). When filling any sample container, care should be taken that splashing drops of water from the ground or sink do not enter into either the bottle or cap.

When sampling at a water treatment plant, samples are often collected from the raw water supply and the treated water after chlorination.

4.0 WELL PURGING

Well purging is the process of removing stagnant cold water prior to sample collection. For potable private water supply sampling, it is recommended to purge the system for at least 15 minutes to remove stagnant water volumes from the well piping to the sample intake location. For municipal supply wells that run continuously, no purging is required other than opening a valve and allowing it to flush for a few minutes. In either circumstance, the field team must coordinate with the well/property owner to determine an appropriate location for the discharge of potentially a large volume of water.

5.0 WELL SUPPLY SAMPLING METHODOLOGY

Samples should be collected following purging from a valve or cold water tap as near to the well as possible and preferably prior to any storage/pressure tanks or physical/chemical treatment system, if present.

- The sample should be collected from a tap or spigot located at or near the well head or pump house and before the water supply is introduced into any storage tank or treatment unit. Remove any aeration or sediment trap device from the tap prior to sample collection.
- Purge the system for a minimum of 15 minutes.

6.0 REFERENCES

USEPA, 2013. Field Branches Quality System and Technical Procedures - Potable Water Supply Sampling (SESDPROC-305-R3). Region 4. May.

ATTACHMENT 3

LABORATORY STANDARD OPERATING PROCEDURE AND CERTIFICATION

Determination of Selected Perfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)

Reference: EPA Method 537, Version 1.1, September 2009, EPA Document #: EPA/600/R-08/092

Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.1, 2016

1. Scope and Application

Matrices: Drinking Water, Non-potable water, Soil

Definitions: Refer to Alpha Analytical Quality Manual.

1.1 This is a liquid chromatography/tandem mass spectrometry (LC/MS/MS) method for the determination of selected perfluorinated alkyl substances (PFASs) in drinking water. Accuracy and precision data have been generated in reagent water, and finished ground and surface waters for the compounds listed in Table 1.

1.2 The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

1.3 This method is restricted to use by or under the supervision of analysts experienced in the operation of the LC/MS/MS and in the interpretation of LC/MS/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Table 1

Parameter	Acronym	CAS
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	-
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	-
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnA	2058-94-8

2. Summary of Method

2.1 A 250-mL water sample is fortified with surrogates and passed through a solid phase extraction (SPE) cartridge containing polystyrenedivinylbenzene (SDVB) to extract the method analytes and surrogates. The compounds are eluted from the solid phase with a small amount of methanol. The extract is concentrated to dryness with nitrogen in a heated water bath, and then adjusted to a 1-mL volume with 96:4% (vol/vol) methanol:water after adding the IS(s). A 10-µL injection is made into an LC equipped with a C18 column that is interfaced to an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the internal standard technique. Surrogate analytes are added to all Field and QC Samples to monitor the extraction efficiency of the method analytes.

2.2 Method Modifications from Reference

2.2.1 None.

3. Reporting Limits

3.1 The reporting limit for PFAS's is 2 ng/L.

4. Interferences

4.1 All glassware must be meticulously cleaned. Wash glassware with detergent and tap water, rinse with tap water, followed by a reagent water rinse. Non-volumetric glassware can be heated in a muffle furnace at 400 °C for 2 hours or solvent rinsed. Volumetric glassware should be solvent rinsed and not be heated in an oven above 120 °C. Store clean glassware inverted or capped. Do not cover with aluminum foil because PFASs can be potentially transferred from the aluminum foil to the glassware.

4.1.1 **NOTE:** PFAS standards, extracts and samples should not come in contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAS analyte, IS and SUR standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers.

4.2 Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The method analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, SPE sample transfer lines, etc. All items such as these must be routinely demonstrated to be free from interferences (less than 1/3 the RL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent blanks as described in Section 9.2. **Subtracting blank values from sample results is not permitted.**

4.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending

upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample. Under the LC conditions used during method development, matrix effects due to total organic carbon (TOC) were not observed.

4.4 Relatively large quantities of the preservative (Sect. 6.2.1) are added to sample bottles. The potential exists for trace-level organic contaminants in these reagents. Interferences from these sources should be monitored by analysis of laboratory reagent blanks (Sect. 9.2.1), particularly when new lots of reagents are acquired.

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

5. Health and Safety

5.1 The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

5.2 All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.3 PFOA has been described as "likely to be carcinogenic to humans." Pure standard materials and stock standard solutions of these method analytes should be handled with suitable protection to skin and eyes, and care should be taken not to breathe the vapors or ingest the materials.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

6.1.1 Samples must be collected in three (3) 250-mL polypropylene bottles fitted with a polypropylene screw-cap.

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

6.1.3 Open the tap and allow the system to flush until the water temperature has stabilized (approximately 3 to 5 min). Collect samples from the flowing system.

6.1.4 Fill sample bottles, taking care not to flush out the sample preservation reagent. Samples do not need to be collected headspace free.

6.1.5 After collecting the sample, cap the bottle and agitate by hand until preservative is dissolved. Keep the sample sealed from time of collection until extraction.

6.1.6 Field Reagent Blank (FRB)

6.1.6.1 A FRB must be handled along with each sample set. The sample set is composed of samples collected from the same sample site and at the same time. At the laboratory, fill the field blank sample bottle 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. At the sampling site, the sampler must open the shipped FRB and pour the preserved reagent water into the empty shipped sample bottle, seal and label this bottle as the FRB. The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFASs were not introduced into the sample during sample collection/handling.

6.1.6.2 The same batch of preservative must be used for the FRBs as for the field samples.

6.1.6.3 The reagent water used for the FRBs must be initially analyzed for method analytes as a MB and must meet the MB criteria in Section 9.2.1 prior to use. This requirement will ensure samples are not being discarded due to contaminated reagent water rather than contamination during sampling.

6.2 Sample Preservation

6.2.1 The preservation reagent, listed in the table below, is added to each sample bottle as a solid prior to shipment to the field (or prior to sample collection).

Table 2

Compound	Amount	Purpose
Trizma	5.0 g/l	Buffering reagent and removes free chlorine

6.3 Sample Shipping

6.3.1 Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10 °C when the samples are received at the laboratory. Samples stored in the lab must be held at or below 6 °C until extraction, but should not be frozen.

NOTE: Samples that are significantly above 10° C, at the time of collection, may need to be iced or refrigerated for a period of time, in order to chill them prior to shipping. This will allow them to be shipped with sufficient ice to meet the above requirements.

6.4 Sample Handling

6.4.1 Holding Times

6.4.1.1 Water samples should be extracted as soon as possible but must be extracted within 14 days. Extracts must be stored at room temperature and analyzed within 28 days after extraction.

7. Equipment and Supplies

7.1 SAMPLE CONTAINERS – 250-mL polypropylene bottles fitted with polypropylene screw caps. Sample bottles must be discarded after use.

7.2 POLYPROPYLENE BOTTLES – 4-mL narrow-mouth polypropylene bottles.

7.3 CENTRIFUGE TUBES – 15-mL conical polypropylene tubes with polypropylene screw caps for storing standard solutions and for collection of the extracts.

7.4 AUTOSAMPLER VIALS – Polypropylene 0.3-mL autosampler vials with polypropylene caps.

7.4.1 NOTE: Polypropylene vials and caps are necessary to prevent contamination of the sample from PTFE coated septa. However, polypropylene caps do not reseal, so evaporation occurs after injection. Thus, multiple injections from the same vial are not possible.

7.5 POLYPROPYLENE GRADUATED CYLINDERS – Suggested sizes include 25, 50, 100 and 1000-mL cylinders.

7.6 MICRO SYRINGES – Suggested sizes include 5, 10, 25, 50, 100, 250, 500 and 1000-µL syringes.

7.7 PLASTIC PIPETS – Polypropylene or polyethylene disposable pipets.

7.8 ANALYTICAL BALANCE – Capable of weighing to the nearest 0.0001 g.

7.9 SOLID PHASE EXTRACTION (SPE) APPARATUS FOR USING CARTRIDGES

7.9.1 SPE CARTRIDGES – 0.5 g, 6-mL SPE cartridges containing styrenedivinylbenzene (SDVB) sorbent phase.

7.9.2 VACUUM EXTRACTION MANIFOLD – A manual vacuum manifold with Visiprep large volume sampler for cartridge extractions, or an automatic/robotic sample preparation system designed for use with SPE cartridges, may be used if all QC requirements discussed in Section 9 are met. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. Care must be taken with automated SPE systems to ensure the PTFE commonly used in these systems does not contribute to unacceptable analyte concentrations in the MB (Sect. 9.2.1).

7.9.3 SAMPLE DELIVERY SYSTEM – Use of a polypropylene transfer tube system, which transfers the sample directly from the sample container to the SPE cartridge, is recommended, but not mandatory. Standard extraction manifolds come equipped with PTFE transfer tube systems. These can be replaced with 1/8" O.D. x 1/16" I.D. polypropylene or polyethylene tubing cut to an appropriate length to ensure no sample contamination from the sample transfer lines. Other types of non-PTFE tubing may be used provided it meets the MB (Sect. 9.2.1) and LCS (Sect. 9.3) QC requirements. The PTFE transfer tubes may be used, but an MB must be run on each PFTE transfer tube and the QC requirements in Section 13.2.2 must be met. In the case of automated SPE, the removal of PTFE lines may not be feasible; therefore, MBs will need to be rotated among the ports and must meet the QC requirements of Sections 13.2.2 and 9.2.1.

7.10 EXTRACT CONCENTRATION SYSTEM – Extracts are concentrated by evaporation with nitrogen using a water bath set no higher than 65 °C.

7.11 LABORATORY OR ASPIRATOR VACUUM SYSTEM – Sufficient capacity to maintain a vacuum of approximately 10 to 15 inches of mercury for extraction cartridges.

7.12 LIQUID CHROMATOGRAPHY (LC)/TANDEM MASS SPECTROMETER (MS/MS) WITH DATA SYSTEM

7.12.1 LC SYSTEM – Instrument capable of reproducibly injecting up to 10-µL aliquots, and performing binary linear gradients at a constant flow rate near the flow rate used for development of this method (0.3 mL/min). The LC must be capable of pumping the water/methanol mobile phase without the use of a degasser which pulls vacuum on the mobile phase bottle (other types of degassers are acceptable). Degassers which pull vacuum on the mobile phase bottle will volatilize the ammonium acetate mobile phase causing the analyte peaks to shift to earlier retention times over the course of the analysis batch. The usage of a column heater is optional.

NOTE: During the course of method development, it was discovered that while idle for more than one day, PFASs built up in the PTFE solvent transfer lines. To prevent long delays in purging high levels of PFASs from the LC solvent lines, they were replaced with PEEK tubing and the PTFE solvent frits were replaced with stainless steel frits. It is not possible to remove all PFAS background contamination, but these measures help to minimize their background levels.

7.12.2 LC/TANDEM MASS SPECTROMETER – The LC/MS/MS must be capable of negative ion electrospray ionization (ESI) near the suggested LC flow rate of 0.3 mL/min. The system must be capable of performing MS/MS to produce unique product ions for the method analytes within specified retention time segments. A minimum of 10 scans across the chromatographic peak is required to ensure adequate precision.

7.12.3 DATA SYSTEM – An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software should have the capability of processing stored LC/MS/MS data by recognizing an LC peak within any given retention time window. The software must allow integration of the ion abundance of any specific ion within specified time or scan number limits. The software must be able to calculate relative response factors, construct linear regressions or quadratic calibration curves, and calculate analyte concentrations.

7.12.4 ANALYTICAL COLUMN – An LC C_{18} column (2.1 x 150 mm) packed with 5 µm d_p C_{18} solid phase particles was used. Any column that provides adequate resolution, peak shape, capacity, accuracy, and precision (Sect. 9) may be used.

8. Reagents and Standards

8.1 GASES, REAGENTS, AND SOLVENTS – Reagent grade or better chemicals should be used.

8.1.1 REAGENT WATER – Purified water which does not contain any measurable quantities of any method analytes or interfering compounds greater than 1/3 the RL for each method analyte of interest. Prior to daily use, at least 3 L of reagent water should be flushed from the purification system to rinse out any build-up of analytes in the system's tubing.

8.1.2 METHANOL (CH_3OH, CAS#: 67-56-1) – High purity, demonstrated to be free of analytes and interferences.

8.1.3 AMMONIUM ACETATE ($NH_4C_2H_3O_2$, CAS#: 631-61-8) – High purity, demonstrated to be free of analytes and interferences.

8.1.4 20 mM AMMONIUM ACETATE/REAGENT WATER – To prepare 1 L, add 1.54 g ammonium acetate to 1 L of reagent water. This solution is prone to volatility losses and should be replaced at least every 48 hours.

8.1.5 TRIZMA PRESET CRYSTALS, pH 7.0 – Reagent grade. A premixed blend of Tris [Tris(hydroxymethyl)aminomethane] and Tris HCL [Tris(hydroxymethyl)aminomethane hydrochloride]. Alternatively, a mix of the two components with a weight ratio of 15.5/1 Tris HCL/Tris may be used. These blends are targeted to produce a pH near 7.0 at 25 °C in reagent water. Trizma functions as a buffer, and removes free chlorine in chlorinated finished waters (Sect. 6.2.1).

8.1.6 NITROGEN – Used for the following purposes: Nitrogen aids in aerosol generation of the ESI liquid spray and is used as collision gas in some MS/MS instruments. The nitrogen used should meet or exceed instrument manufacturer's specifications. In addition, Nitrogen is used to concentrate sample extracts (Ultra High Purity or equivalent).

8.1.7 ARGON – Used as collision gas in MS/MS instruments. Argon should meet or exceed instrument manufacturer's specifications. Nitrogen gas may be used as the collision gas provided sufficient sensitivity (product ion formation) is achieved.

8.2 STANDARD SOLUTIONS – When a compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. PFAS analyte, IS and SUR standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers. Standards for sample fortification generally should be prepared in the smallest volume that can be accurately measured to minimize the addition of excess organic solvent to aqueous samples.

NOTE: Stock standards (Sect. 8.2.1, 8.2.3 and 8.2.5) are stored at ≤4 °C. Primary dilution standards (Sect. 8.2.2 and 8.2.4) are stored at room temperature to prevent adsorption of the method analytes onto the container surfaces that may occur when refrigerated. Storing the standards at room temperature will also minimize daily imprecision due to the potential of inadequate room temperature stabilization.

8.2.1 IS STOCK STANDARD SOLUTIONS - IS stock standard solutions are stable for at least 6 months when stored at 4 °C. The stock solution is purchased at a concentration range of 1-4 ng/µl.

8.2.2 INTERNAL STANDARD PRIMARY DILUTION (IS PDS) STANDARD (0.5-2 ng/µL) – Prepare the IS PDS at a concentration of 0.5-2 ng/µL. The IS PDS is prepared in 96:4% (vol/vol) methanol:water. The IS PDS is stable for at least two months when stored in polypropylene centrifuge tubes at room temperature.

Table 3

Internal Standard	Conc. of IS Stock (ng/uL)	Vol. of IS Stock (mL)	Final Vol. of IS PDS (mL)	Final Conc. of IS PDS (ng/µL)
^{13}C-PFOA	1	1.0	2.0	0.5
^{13}C-PFOS	3	1.0	2.0	1.5
D_3-NMeFOSAA	4	1.0	2.0	2.0

8.2.3 SUR STOCK STANDARD SOLUTIONS – SUR stock standard solutions are stable for at least 6 months when stored at 4 °C.

8.2.4 SURROGATE PRIMARY DILUTION STANDARD (SUR PDS) (0.5-2 ng/µL) – Prepare the SUR PDS at a concentration of 0.5-2 ng/µL. The SUR PDS is prepared in 96:4% (vol/vol) methanol:water. This solution is used to fortify all QC and Field Samples. The PDS is stable for one year when stored in polypropylene centrifuge tubes at room temperature.

Table 4

Surrogate	Conc. of SUR Stock (ng/µL)	Vol. of SUR Stock (mL)	Final Vol. of SUR PDS (,L)	Final Conc. of SUR PDS (ng/µL)
^{13}C-PFHxA	1.0	1.0	2.0	0.5
^{13}C-PFDA	1.0	1.0	2.0	0.5
d_5-NEtFOSAA	4.0	1.0	2.0	2.0

8.2.5 ANALYTE STOCK STANDARD SOLUTION – Analyte stock standards are stable for at least 6 months when stored at -15 °C. When using these stock standards to prepare a PDS, care must be taken to ensure that these standards are at room temperature and adequately vortexed.

Table 5

Analyte	Analyte Stock Solvent	Concentration (ug/mL)
PFHxA	96:4% (vol/vol) methanol:water	1.0
PFHpA	96:4% (vol/vol) methanol:water	1.0
PFOA	96:4% (vol/vol) methanol:water	1.0
PFNA	96:4% (vol/vol) methanol:water	1.0
PFDA	96:4% (vol/vol) methanol:water	1.0
PFUnA	96:4% (vol/vol) methanol:water	1.0
PFDoA	96:4% (vol/vol) methanol:water	1.0
PFTrDA	100% ethyl acetate	1.0
PFTA	100% ethyl acetate	1.0
PFBS	100% methanol	1.0
PFHxS	100% methanol	1.0
PFOS	100% methanol	1.0
NEtFOSAA	100% methanol	1.0
NMeFOSAA	100% methanol	1.0

8.2.6 LOW, MEDIUM AND HIGH LEVEL LCS – The LCS's will be prepared at the following concentrations and rotated per batch; 2 ng/L, 40 ng/L, 500 ng/l. The analyte PDS contains all the method analytes of interest at various

concentrations in methanol containing 4% water. The analyte PDS has been shown to be stable for 6 months when stored at room temperature.

8.2.7 CALIBRATION STANDARDS (CAL) –

Current Concentrations (ng/mL): 0.5, 1.0, 2.0, 5.0, 10.0, 20.0, 30.0, 40.0, 50.0, 125 and 150 (optional)

Prepare the CAL standards over the concentration range of interest from dilutions of the analyte PDS in methanol containing 4% reagent water. The IS and SUR are added to the CAL standards at a constant concentration (10-40 ng/L). The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may depend on system sensitivity. The CAL standards may also be used as CCVs (Sect. 9.9). The CAL standards are stable for at least two weeks when stored at room temperature. Longer storage times are acceptable provided appropriate QC measures are documented demonstrating the CAL standard stability.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 REPORTING LIMIT (RL) CONFIRMATION

9.1.1 Fortify, extract, and analyze seven replicate LCSs at 2 ng/l. These LCSs must contain all method preservatives described in Section 6.2.1. Calculate the mean measured concentration (*Mean*) and standard deviation for these replicates. Determine the Half Range for the prediction interval of results (HR_{PIR}) using the equation below

$$HR_{PIR} = 3.963s$$

Where:

s = the standard deviation
3.963 = a constant value for seven replicates.

9.1.2 Confirm that the upper and lower limits for the Prediction Interval of Result ($PIR = Mean \pm HR_{PIR}$) meet the upper and lower recovery limits as shown below

The Upper PIR Limit must be ≤150% recovery.

$$\frac{Mean + HR_{PIR}}{Fortified\ Concentration} \times 100\% \leq 150\%$$

The Lower PIR Limit must be ≥ 50% recovery.

$$\frac{Mean - HR_{PIR}}{Fortified\ Concentration} \times 100\% \geq 50\%$$

9.1.3 The RL is validated if both the Upper and Lower PIR Limits meet the criteria described above. If these criteria are not met, the RL has been set too low and must be determined again at a higher concentration.

9.2 Blank(s)

9.2.1 **METHOD BLANK (MB)** - A Method Blank (MB) is required with each extraction batch to confirm that potential background contaminants are not interfering with the identification or quantitation of method analytes. If more than 20 Field Samples are included in a batch, analyze an MB for every 20 samples. If the MB produces a peak within the retention time window of any analyte that would prevent the determination of that analyte, determine the source of contamination and eliminate the interference before processing samples. Background contamination must be reduced to an acceptable level before proceeding. Background from method analytes or other contaminants that interfere with the measurement of method analytes must be below 1/3 of the RL. Blank contamination is estimated by extrapolation, if the concentration is below the lowest CAL standard. This extrapolation procedure is not allowed for sample results as it may not meet data quality objectives. If the method analytes are detected in the MB at concentrations equal to or greater than this level, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch. Because background contamination is a significant problem for several method analytes, it is highly recommended that the analyst maintain a historical record of MB data.

9.2.2 **FIELD REAGENT BLANK (FRB)** - The purpose of the FRB is to ensure that PFASs measured in the Field Samples were not inadvertently introduced into the sample during sample collection/handling. Analysis of the FRB is required only if a Field Sample contains a method analyte or analytes at or above the RL. The FRB is processed, extracted and analyzed in exactly the same manner as a Field Sample. If the method analyte(s) found in the Field Sample is present in the FRB at a concentration greater than 1/3 the RL, then all samples collected with that FRB are invalid and must be recollected and reanalyzed.

9.3 Laboratory Control Sample (LCS)

9.3.1 An LCS is required with each extraction batch. The fortified concentration of the LCS must be rotated between low, medium, and high concentrations from batch to batch.

9.3.2 The low concentration LCS must be as near as practical to, but no more than two times, the RL. Similarly, the high concentration LCS should be near the high end of the calibration range established during the initial calibration (Sect. 10.6).

9.3.3 Results of the low-level LCS analyses must be 50-150% of the true value. Results of the medium and high-level LCS analyses must be 70-130% of the true value. If the LCS results do not meet these criteria for method analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.

9.3.4 It is the responsibility of the extraction chemist to view the previous extraction batch to determine the next spiking concentration. (Low → Medium → High)

9.4 Internal Standards (IS)

The analyst must monitor the peak areas of the IS(s) in all injections during each analysis day. The IS responses (peak areas) in any chromatographic run must be within 70-140% of the response in the most recent CCV and must not deviate by more than 50% from the average area measured during initial analyte calibration. If the IS areas in a chromatographic run do not meet these criteria, inject a second aliquot of that extract aliquoted into a new capped autosampler vial. Random evaporation losses have been observed with the polypropylene caps causing high IS(s) areas.

9.4.1 If the reinjected aliquot produces an acceptable IS response, report results for that aliquot.

9.4.2 If the reinjected extract fails again, the analyst should check the calibration by reanalyzing the most recently acceptable CAL standard. If the CAL standard fails the criteria of Section 9.9, recalibration is in order per Section 10.6. If the CAL 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 reinjected extract, but annotate as suspect. Alternatively, collect a new sample and re-analyze.

9.5 Surrogate Recovery

The SUR standard is fortified into all samples, CCVs, MBs, LCSs, MSs, MSDs, FD, and FRB prior to extraction. It is also added to the CAL standards. The SUR is a means of assessing method performance from extraction to final chromatographic measurement. Calculate the recovery (%R) for the SUR using the following equation

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

Where:

A = calculated SUR concentration for the QC or Field Sample
B = fortified concentration of the SUR.

9.5.1.1 SUR recovery must be in the range of 70-130%. When SUR recovery from a sample, blank, or CCV is less than 70% or greater than 130%, check 1) calculations to locate possible errors, 2) standard solutions for degradation, 3) contamination, and 4) instrument performance. Correct the problem and reanalyze the extract.

9.5.1.2 If the extract reanalysis meets the SUR recovery criterion, report only data for the reanalyzed extract.

9.5.1.3 If the extract reanalysis fails the 70-130% recovery criterion, the analyst should check the calibration by injecting the last CAL standard that passed. If the CAL standard fails the criteria of Section 10.7, recalibration is in order per Section 10.6. If the CAL standard is acceptable, extraction of the sample should be repeated provided the sample is still within the holding time. If the re-extracted sample also fails the recovery criterion, report all data for that sample as suspect/SUR recovery to inform the data user that the results are suspect due to SUR recovery. Alternatively, collect a new sample and re-analyze.

9.6 Matrix Spike (MS)

9.6.1 Analysis of an MS is required in each extraction batch and is used to determine that the sample matrix does not adversely affect method accuracy. Assessment of method precision is accomplished by analysis of a Field Duplicate (FD) (Sect. 9.7); however, infrequent occurrence of method analytes would hinder this assessment. If the occurrence of method analytes in the samples is infrequent, or if historical trends are unavailable, a second MS, or MSD, must be prepared, extracted, and analyzed from a duplicate of the Field Sample. Extraction batches that contain MSDs will not require the extraction of a field sample duplicate. If a variety of different sample matrices are analyzed regularly, for example, drinking water from groundwater and surface water sources, method performance should be established for each. Over time, MS data should be documented by the laboratory for all routine sample sources.

9.6.2 Within each extraction batch, a minimum of one Field Sample is fortified as an MS for every 20 Field Samples analyzed. The MS is prepared by spiking a sample with an appropriate amount of the Analyte Stock Standard (Sect. 8.2.5). Use historical data and rotate through the low, mid and high concentrations when selecting a fortifying concentration. Calculate the percent recovery (%*R*) for each analyte using the equation

$$\%R = \frac{(A - B)}{C} \times 100$$

Where:

A = measured concentration in the fortified sample
B = measured concentration in the unfortified sample
C = fortification concentration.

9.6.3 Analyte recoveries may exhibit matrix bias. For samples fortified at or above their native concentration, recoveries should range between 70-130%, except for low-level fortification near or at the RL (within a factor of 2-times the RL concentration) where 50-150% recoveries are acceptable. If the accuracy of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCVs, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.7 Laboratory Duplicate

9.7.1 FIELD DUPLICATE OR LABORATORY FORTIFIED SAMPLE MATRIX DUPLICATE (FD or MSD) – Within each extraction batch (not to exceed 20 Field Samples), a minimum of one FD or MSD must be analyzed. Duplicates check the precision associated with sample collection, preservation, storage, and laboratory procedures. If method analytes are not routinely observed in Field Samples, an MSD should be analyzed rather than an FD.

9.7.2 Calculate the relative percent difference (*RPD*) for duplicate measurements (*FD1* and *FD2*) using the equation

$$RPD = \frac{|FD1 - FD2|}{(FD1 + FD2) / 2} \times 100$$

9.7.3 RPDs for FDs should be ≤30%. Greater variability may be observed when FDs have analyte concentrations that are within a factor of 2 of the RL. At these

concentrations, FDs should have RPDs that are ≤50%. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCV, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.7.4 If an MSD is analyzed instead of a FD, calculate the relative percent difference (RPD) for duplicate MSs (MS and MSD) using the equation

$$RPD = \frac{|MS - MSD|}{(MS + MSD) / 2} \times 100$$

9.7.5 RPDs for duplicate MSs should be ≤30% for samples fortified at or above their native concentration. Greater variability may be observed when MSs are fortified at analyte concentrations that are within a factor of 2 of the RL. MSs fortified at these concentrations should have RPDs that are ≤50% for samples fortified at or above their native concentration. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCV, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.8 Initial Calibration Verification (ICV)

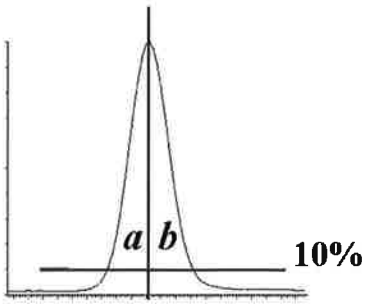
9.8.1 As part of the IDC (Sect. 13.2), each time a new Analyte Stock Standard solution (Sect. 8.2.5) is used, and at least quarterly, analyze a QCS sample from a source different from the source of the CAL standards. If a second vendor is not available, then a different lot of the standard should be used. The QCS should be prepared and analyzed just like a CCV. Acceptance criteria for the QCS are identical to the CCVs; the calculated amount for each analyte must be ± 30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem.

9.9 Continuing Calibration Verification (CCV)

9.9.1 CCV Standards are analyzed at the beginning of each analysis batch, after every 10 Field Samples, and at the end of the analysis batch. See Section 10.7 for concentration requirements and acceptance criteria.

9.10 Method-specific Quality Control Samples

9.10.1 PEAK ASYMMETRY FACTOR – A peak asymmetry factor must be calculated using the equation below during the IDL and every time a calibration curve is generated. The peak asymmetry factor for the first two eluting peaks in a midlevel CAL standard (if only two analytes are being analyzed, both must be evaluated) must fall in the range of 0.8 to 1.5. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted. See



guidance in Section 10.6.4.1 if the calculated peak asymmetry factors do not meet the criteria.

$$A_s = b / a$$

Where:

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

9.11 Method Sequence

ICV

CCV-LOW

MB

LCS

LCSD

MS

Duplicate or MSD

Field Samples (1-10)

CCV-MID

Field Samples (11-20)

CCV-HIGH

10. Procedure

10.1 Equipment Set-up

10.1.1 This procedure may be performed manually or in an automated mode using a robotic or automatic sample preparation device. If an automated system is used to prepare samples, follow the manufacturer's operating instructions, but all extraction and elution steps must be the same as in the manual procedure. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. If an automated system is used, the MBs should be rotated among the ports to ensure that all the valves and tubing meet the MB requirements (Sect. 9.2).

10.1.2 Some of the PFASs adsorb to surfaces, including polypropylene. Therefore, the aqueous sample bottles must be rinsed with the elution solvent (Sect 10.3.4) whether extractions are performed manually or by automation. The bottle rinse is passed through the cartridge to elute the method analytes and is then collected (Sect. 10.3.4).

10.1.3 **NOTE:** The SPE cartridges and sample bottles described in this section are designed as single use items and should be discarded after use. They may not be refurbished for reuse in subsequent analyses.

10.2 Sample Preparation

10.2.1 Samples are preserved, collected and stored as presented in Section 6. All Field and QC Samples, including the MB, LCS and FRB, must contain the dechlorinating agent listed in Section 6.2.1. Determine sample volume. An indirect measurement may be done in one of two ways: by marking the level of the sample on the bottle or by weighing the sample and bottle to the nearest 10 g. After extraction, proceed to Section 10.5 for final volume determination. Some of the PFASs adsorb to surfaces, thus the sample volume may **NOT** be transferred to a graduated cylinder for volume measurement. The MB, LCS and FRB may be prepared by measuring 250 mL of reagent water with a polypropylene graduated cylinder or filling a 250-mL sample bottle to near the top.

The entire sample that is received must be sent through the SPE cartridge. In addition, the bottle must be solvent rinsed and this rinse must be sent through the SPE cartridge as well. The method blank (MB) and laboratory control sample (LCS) must be extracted in exactly the same manner (i.e., must include the bottle solvent rinse). It should be noted that a water rinse alone is not sufficient. This does not apply to samples with high concentrations of PFAS that are prepared using serial dilution and not SPE.

10.2.2 Add 20 µL of the SUR PDS (Sect. 8.2.4) to each sample, cap and invert to mix for a final concentration of 10 ng/L for ^{13}C-PFHxA and ^{13}C-PFDA and 40 ng/L for d_5-NEtFOSAA.

10.2.3 In addition to the SUR(s) and dechlorination agent, if the sample is an LCS, MS, or MSD, add the necessary amount of analyte PDS (Sect. 8.2.5). Cap and invert each sample to mix.

10.3 Cartridge SPE Procedure

10.3.1 CARTRIDGE CLEAN-UP AND CONDITIONING – DO NOT allow cartridge packing material to go dry during any of the conditioning steps. Rinse each cartridge with 15 mL of methanol. Next, rinse each cartridge with 18 mL of reagent water, without allowing the water to drop below the top edge of the packing. If the cartridge goes dry during the conditioning phase, the conditioning must be started over. Add 4-5 mL of reagent water to each cartridge, attach the sample transfer tubes (Sect. 7.2.3), turn on the vacuum, and begin adding sample to the cartridge.

10.3.2 SAMPLE EXTRACTON – Adjust the vacuum so that the approximate flow rate is 10-15 mL/min. Do not allow the cartridge to go dry before all the sample has passed through.

10.3.3 SAMPLE BOTTLE AND CARTRIDGE RINSE – After the entire sample has passed through the cartridge, rinse the sample bottles with two 7.5-mL aliquots of reagent water and draw each aliquot through the sample transfer tubes and the cartridges. Draw air or nitrogen through the cartridge for 5 min at high vacuum (10-15 in. Hg).

NOTE: If empty plastic reservoirs are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be

treated like the transfer tubes. After the entire sample has passed through the cartridge, the reservoirs must be rinsed to waste with reagent water.

10.3.4 SAMPLE BOTTLE AND CARTRIDGE ELUTION – Turn off and release the vacuum. Lift the extraction manifold top and insert a rack with collection tubes into the extraction tank to collect the extracts as they are eluted from the cartridges. Rinse the sample bottles with 4 mL of methanol and elute the analytes from the cartridges by pulling the 4 mL of methanol through the sample transfer tubes and the cartridges. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion. Repeat sample bottle rinse and cartridge elution with a second 4-mL aliquot of methanol.

NOTE: If empty plastic reservoirs are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be treated like the transfer tubes. After the reservoirs have been rinsed in Section 10.3.3, the elution solvent used to rinse the sample bottles must be swirled down the sides of the reservoirs while eluting the cartridge to ensure that any method analytes on the surface of the reservoirs are transferred to the extract.

10.4 Extract Concentration

10.4.1 Concentrate the extract to dryness under a gentle stream of nitrogen in a heated water bath (60-65 °C) to remove all the water/methanol mix. Add the appropriate amount of 96:4% (vol/vol) methanol:water solution and the IS PDS (Sect. 8.2.2) to the collection vial to bring the volume to 1 mL and vortex. Transfer a small aliquot with a plastic pipet (Sect. 7.6) to a polypropylene autosampler vial.

NOTE: It is recommend that the entire 1-mL aliquot not be transferred to the autosampler vial because the polypropylene autosampler caps do not reseal after injection. Therefore, do not store the extracts in the autosampler vials as evaporation losses can occur occasionally in these autosampler vials. Extracts can be stored in 15-mL centrifuge tubes (Sect. 7.3).

10.5 Sample Volume Determination

10.5.1 If the level of the sample was marked on the sample bottle, use a graduated cylinder to measure the volume of water required to fill the original sample bottle to the mark made prior to extraction. Determine to the nearest 10 mL. If using weight to determine volume, weigh the empty bottle to the nearest 10 g and determine the sample weight by subtraction of the empty bottle weight from the original sample weight (Sect. 10.2.1). Assume a sample density of 1.0 g/mL. In either case, the sample volume will be used in the final calculations of the analyte concentration (Sect. 11.2).

10.6 Initial Calibration - Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed. After the initial calibration is successful, a CCV is required at the beginning and end of each period in which analyses are performed, and after every tenth Field Sample.

10.6.1 ESI-MS/MS TUNE

10.6.1.1 Calibrate the mass scale of the MS with the calibration compounds and procedures prescribed by the manufacturer.

10.6.1.2 Optimize the [M-H]- for each method analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.3 mL/min). This tune can be done on a mix of the method analytes. The MS parameters (voltages, temperatures, gas flows, etc.) are varied until optimal analyte responses are determined. The method analytes may have different optima requiring some compromise between the optima.

10.6.1.3 Optimize the product ion for each analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.3 mL/min). This tune can be done on a mix of the method analytes. The MS/MS parameters (collision gas pressure, collision energy, etc.) are varied until optimal analyte responses are determined. Typically, the carboxylic acids have very similar MS/MS conditions and the sulfonic acids have similar MS/MS conditions.

10.6.2 Establish LC operating parameters that optimize resolution and peak shape. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

Cautions: LC system components, as well as the mobile phase constituents, contain many of the method analytes in this method. Thus, these PFASs will build up on the head of the LC column during mobile phase equilibration. To minimize the background PFAS 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.

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 in Sect. 10.9.2 can be met over a period of two weeks. During method development, retention times shifted to shorter and shorter times as days progressed when mobile phases with less than 20 mM ammonium acetate were used.

10.6.3 Inject a mid-level CAL standard under LC/MS conditions to obtain the retention times of each method analyte. If analyzing for PFTA, ensure that the LC conditions are adequate to prevent co-elution of PFTA and the mobile phase interferants. These interferants have the same precursor and products ions as PFTA, and under faster LC conditions may co-elute with PFTA. Divide the chromatogram into retention time windows each of which contains one or more chromatographic peaks. During MS/MS analysis, fragment a small number of selected precursor ions ([M-H]-) for the analytes in each window and choose the most abundant product ion. For maximum sensitivity, small mass windows of ±0.5 daltons around the product ion mass were used for quantitation. If sufficient sensitivity exists to meet the RL, wider mass ranges may be used to obtain more confirmation ions.

10.6.3.1 As recommended by the EPA Advisory on September 2016, both linear and branched isomers should e included in the quantitation. NOTE: As the NOTE in Section 10.6.4.1 indicates, PFOS has linear and branched isomers. There have been reports that not all the products ions in the linear PFOS are produced in all the branched PFOS isomers. (This phenomenon probably exists for PFHxS and PFBS also, although it has not been studied to date.) Thus, in an attempt to reduce PFOS bias, it is required that the *m/z* 499 → *m/z* 80 transition be used as the quantitation transition. Some MS/MS instruments, such as conventional ion traps, may not be able to scan a product ion with such a wide mass difference from the precursor ion; therefore, they may not be used for this method if PFOS, PFBS, or PFHxS analysis is to be conducted. Literature reports indicate for the most abundant PFOS isomer, which is the linear isomer, that all the products ions obtained on an ion trap have less than 10% relative abundance. In addition, there is not a single ion trap MS/MS transition that encompasses the linear isomer and the majority of the branch isomers; thus, the bias would be unacceptably high.

10.6.4 Inject a mid-level CAL standard under optimized LC/MS/MS conditions to ensure that each method analyte is observed in its MS/MS window and that there are at least 10 scans across the peak for optimum precision.

10.6.4.1 If broad, split or fronting peaks are observed for the first two eluting chromatographic peaks (if only two analytes are being analyzed, both must be evaluated), change the initial mobile phase conditions to higher aqueous content until the peak asymmetry ratio for each peak is 0.8 – 1.5. The peak asymmetry factor is calculated as described in Section 9.10.1 on a mid-level CAL standard. The peak asymmetry factor must meet the above criteria for the first two eluting peaks during the IDL and every time a new calibration curve is generated. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

NOTE: PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 5 due to chromatographic resolution of the linear and branched isomers of these compounds. According to the EPA Advisory, September 2016, the branched isomers are identified by analyzing a qualitative/semi-qualitative mixed PFOA standard and the quantitation of PFOA is accomplished by integration the total response which includes peaks identified as linear and branched isomers. Most PFASs are produced by two different processes. One process gives rise to linear PFASs only while the other process produces both linear and branched isomers. Thus, both branched and linear PFASs can potentially be found in the environment. For the aforementioned compounds that give rise to more than one peak, all the chromatographic peaks observed in the standard must be integrated and the areas totaled. Chromatographic peaks in a sample must be integrated in the same way as the CAL standard.

10.6.5 Prepare a set of CAL standards as described in Section 8.2.7. The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may

depend on system sensitivity. It is recommended that at least four of the CAL standards are at a concentration greater than or equal to the RL.

10.6.6 The LC/MS/MS system is calibrated using the IS technique. Use the LC/MS/MS data system software to generate a linear regression or quadratic calibration curve for each of the analytes. This curve **must always** be forced through zero and may be concentration weighted, if necessary. Forcing zero allows for a better estimate of the background levels of method analytes.

10.6.6.1 The isotopically labeled IS(s) in this method may undergo suppression in the ESI source if the concentration of the co-eluting unlabeled method analyte(s) is too high. The analyte concentration at which suppression may occur can vary depending on the instrument, LC conditions, ESI conditions, IS concentration, etc. To evaluate whether suppression is occurring during calibration, calculate the relative percent difference (*RPD*) between the high (H) and low (L) areas for each IS using the equation

$$RPD = \frac{(H - L)}{(H + L) / 2} \times 100$$

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

10.6.7 CALIBRATION ACCEPTANCE CRITERIA – When quantitated using the initial calibration curve, each calibration point, except the lowest point, for each analyte should calculate to be within 70-130% of its true value. The lowest CAL point should calculate to be within 50-150% of its true value. If these criteria cannot be met, the analyst will have difficulty meeting ongoing QC criteria. It is recommended that corrective action is taken to reanalyze the CAL standards, restrict the range of calibration, or select an alternate method of calibration (forcing the curve through zero is still required).

10.6.7.1 **CAUTION:** When acquiring MS/MS data, LC operating conditions must be carefully reproduced for each analysis to provide reproducible retention times. If this is not done, the correct ions will not be monitored at the appropriate times. As a precautionary measure, the chromatographic peaks in each window must not elute too close to the edge of the segment time window.

10.7 CONTINUING CALIBRATION CHECK (CCV) – Minimum daily calibration verification is as follows. Verify the initial calibration at the beginning and end of each group of analyses, and after every tenth sample during analyses. In this context, a "sample" is considered to be a Field Sample. MBs, CCVs, LCSs, MSs, FDs FRBs and MSDs are not counted as samples. The beginning CCV of each analysis batch must be at or below the RL in order to verify instrument sensitivity prior to any analyses. If standards have been prepared such that all low CAL points are not in the same CAL solution, it may be necessary to analyze two CAL standards to meet this requirement. Alternatively, the analyte concentrations in the analyte PDS may be customized to meet this criteria. Subsequent CCVs should alternate between a medium and high concentration CAL standard.

10.7.1 Inject an aliquot of the appropriate concentration CAL standard and analyze with the same conditions used during the initial calibration.

10.7.2 Determine that the absolute areas of the quantitation ions of the IS(s) are within 70-140% of the areas measured in the most recent continuing calibration check, and within 50-150% from the average areas measured during initial calibration. If any of the IS areas has changed by more than these amounts, adjustments must be made to restore system sensitivity. These adjustments may include cleaning of the MS ion source, or other maintenance as indicated in Section 10.7.4. Major instrument maintenance requires recalibration (Sect 10.6) and verification of sensitivity by analyzing a CCV at or below the RL (Sect 10.7). Control charts are useful aids in documenting system sensitivity changes.

10.7.3 Calculate the concentration of each analyte and SUR in the CCV. The calculated amount for each analyte and SUR for medium and high level CCVs must be within ± 30% of the true value. The calculated amount for the lowest calibration point for each analyte must be within ± 50% and the SUR must be within ± 30% of the true value. If these conditions do not exist, then all data for the problem analyte must be considered invalid, and remedial action should be taken (Sect. 10.7.4) which may require recalibration. Any Field or QC Samples that have been analyzed since the last acceptable calibration verification should be reanalyzed after adequate calibration has been restored, with the following exception. **If the CCV fails because the calculated concentration is greater than 130% (150% for the low-level CCV) for a particular method analyte, and Field Sample extracts show no detection for that method analyte, non-detects may be reported without re-analysis.**

10.7.4 REMEDIAL ACTION – Failure to meet CCV QC performance criteria may require remedial action. Major maintenance, such as cleaning the electrospray probe, atmospheric pressure ionization source, cleaning the mass analyzer, replacing the LC column, etc., requires recalibration (Sect 10.6) and verification of sensitivity by analyzing a CCV at or below the RL (Sect 10.7).

10.8 EXTRACT ANALYSIS

10.8.1 Establish operating conditions equivalent to those summarized in Tables 5-8 of Section 16. Instrument conditions and columns should be optimized prior to the initiation of the IDC.

10.8.2 Establish an appropriate retention time window for each analyte. This should be based on measurements of actual retention time variation for each method analyte in CAL standard solutions analyzed on the LC over the course of time. A value of plus or minus three times the standard deviation of the retention time obtained for each method analyte while establishing the initial calibration and completing the IDC can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily on the determination of the appropriate retention window size.

10.8.3 Calibrate the system by either the analysis of a calibration curve (Sect. 10.6) or by confirming the initial calibration is still valid by analyzing a CCV as described in Section 10.7. If establishing an initial calibration, complete the IDC as described in Section 13.2.

10.8.4 Begin analyzing Field Samples, including QC samples, at their appropriate frequency by injecting the same size aliquots (10 µL was used in method development), under the same conditions used to analyze the CAL standards.

10.8.5 At the conclusion of data acquisition, use the same software that was used in the calibration procedure to identify peaks of interest in predetermined retention time windows. Use the data system software to examine the ion abundances of the peaks in the chromatogram. Identify an analyte by comparison of its retention time with that of the corresponding method analyte peak in a reference standard.

10.8.6 Comparison of the MS/MS mass spectra is not particularly useful given the limited ±0.5 dalton mass range around a single product ion for each method analyte.

10.8.7 The analyst must not extrapolate beyond the established calibration range. If an analyte peak area exceeds the range of the initial calibration curve, the extract may be diluted with 96%:4% vol/vol) methanol:water solution and the appropriate amount of IS added to match the original concentration. Re-inject the diluted extract. Incorporate the dilution factor into the final concentration calculations. Acceptable SUR performance (Sect. 9.5.1.1) should be determined from the undiluted sample extract. The resulting data should be documented as a dilution, with an increased RL.

11. Data Evaluation, Calculations and Reporting

11.1 Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations using MS/MS. In validating this method, concentrations were calculated by measuring the product ions listed in Table 8. Other ions may be selected at the discretion of the analyst.

11.2 Calculate analyte and SUR concentrations using the multipoint calibration established in Section 10.6. Do not use daily calibration verification data to quantitate analytes in samples. Adjust final analyte concentrations to reflect the actual sample volume determined in Section 10.5.

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

11.4 PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 5 due to the linear and branch isomers of these compounds (Sect. 10.6.4.1). The areas of all the linear and branched isomer peaks observed in the CAL standards for each of these analytes must be summed and the concentrations reported as a total for each of these analytes.

11.5 Calculations must utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty), typically two, and not more than three significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Section 9.0 outlines sample batch QC acceptance criteria. If non-compliant organic compound results are to be reported, the Organic Section Head and/or the Laboratory Director, and the Operations Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis.

The analyst, Data Reviewer, or Department Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

12.2 All results for the organic compounds of interest are reportable without qualification if extraction and analytical holding times are met, preservation requirements (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

13.1.1 The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

13.2.1 The IDC must be successfully performed prior to analyzing any Field Samples. Prior to conducting the IDC, the analyst must first generate an acceptable Initial Calibration following the procedure outlined in Section 10.6.

13.2.2 INITIAL DEMONSTRATION OF LOW SYSTEM BACKGROUND – Any time a new lot of SPE cartridges, solvents, centrifuge tubes, disposable pipets, and autosampler vials are used, it must be demonstrated that an MB is reasonably free of contamination and that the criteria in Section 9.2.1 are met. If an automated extraction system is used, an MB should be extracted on each port to ensure that all the valves and tubing are free from potential PFAS contamination.

13.2.3 INITIAL DEMONSTRATION OF PRECISION (IDP) – Prepare, extract, and analyze four to seven replicate LCSs fortified near the midrange of the initial calibration curve according to the procedure described in Section 10. Sample preservatives as described in Section 6.2.1 must be added to these samples. The relative standard deviation (RSD) of the results of the replicate analyses must be less than 20%.

13.2.4 INITIAL DEMONSTRATION OF ACCURACY (IDA) – Using the same set of replicate data generated for Section 13.2.3, calculate average recovery. The average recovery of the replicate values must be within ± 30% of the true value.

13.2.5 INITIAL DEMONSTRATION OF PEAK ASYMMETRY FACTOR – Peak asymmetry factors must be calculated using the equation in Section 9.10.1 for the first two eluting peaks (if only two analytes are being analyzed, both must be evaluated) in a mid-level CAL standard. The peak asymmetry factors must fall in the range of 0.8 to 1.5. See guidance in Section 10.6.4.1 if the calculated peak asymmetry factors do not meet the criteria.

13.2.6 Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.7 The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

14.1.1 Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

14.1.2 This method utilizes SPE to extract analytes from water. It requires the use of very small volumes of organic solvent and very small quantities of pure analytes, thereby minimizing the potential hazards to both the analyst and the environment as compared to the use of large volumes of organic solvents in conventional liquid-liquid extractions.

14.1.3 The analytical procedures described in this method generate relatively small amounts of waste since only small amounts of reagents and solvents are used. The matrices of concern are finished drinking water or source water. However, laboratory waste management practices must be conducted consistent with all applicable rules and regulations, and that laboratories protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations, particularly the hazardous waste identification rules and land disposal restrictions.

15. Referenced Documents

15.1.1 Chemical Hygiene Plan – ID 2124

15.1.2 SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

15.1.3 SOP ID 1739 Demonstration of Capability (DOC) Generation SOP

15.1.4 SOP ID 1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 6: LC Method Conditions

Time (min)	% 20 mM Ammonium Acetate	% Methanol
Initial	60.0	40.0
1.0	60.0	40.0
25.0	10.0	90.0
32.0	10.0	90.0
32.1	60.0	40.0
37.0	60.0	40.0
Waters Atlantis ® dC_{18} 2.1 x 150 mm packed with 5.0 µm C_{18} stationary phase Flow rate of 0.3 mL/min 10 µL injection		

Table 7: ESI-MS Method Conditions

ESI Conditions	
Polarity	Negative ion
Capillary needle voltage	-3 kV
Cone Gas Flow	98 L/hr
Nitrogen desolvation gas	1100 L/hr
Desolvation gas temp.	350 °C

Table 8: Method Analyte Source, Retention Times (RTs), and IS References

Analyte	Peak #	IS# Ref
PFBS	1	2
PFHxA	2	1
PFHpA	4	1
PFHxS	5	2
PFOA	6	1
PFNA	8	1
PFOS	9	2
PFDA	11	1
NMeFOSAA	13	3
NEtFOSAA	15	3
PFUnA	17	1
PFDoA	18	1
PFTrDA	19	1
PFTA	20	1
^{13}C-PFHxA	3	1
^{13}C-PFDA	12	1
d_5-NEtFOSAA	16	3
^{13}C-PFOA-IS#1	7	-
^{13}C-PFOS-IS#2	10	-
d_3-NMeFOSAA-IS#3	14	-

Table 9: MS/MS Method Conditions

Segment[a]	Analyte	Precursor Ion [b] (m/z)	Product Ion [b,c] (m/z)	Cone Voltage (v)	Collision Energy [d] (v)
1	PFBS	299	80	40	25
2	PFHxA	313	269	15	10
3	PFHpA	363	319	12	10
3	PFHxS [e]	399	80	40	40
4	PFOA	413	369	15	10
4	PFNA	463	419	12	10
4	PFOS [e]	499	80	40	40
5	PFDA	513	469	15	10
5	NMeFOSAA [e]	570	419	25	20
5	NEtFOSAA [e]	584	419	25	20
5	PFUnA	563	519	15	10
5	PFDoA	613	569	15	10
6	PFTrDA	663	619	15	10
6	PFTA	713	669	15	10
2	^{13}C-PFHxA	315	270	15	10
5	^{13}C-PFDA	515	470	12	12
5	d_5-NEtFOSAA	589	419	25	20
4	^{13}C-PFOA	415	370	15	10
4	^{13}C-PFOS	503	80	40	40
5	d_3-NMeFOSAA	573	419	25	20

[a] Segments are time durations in which single or multiple scan events occur.
[b] Precursor and product ions listed in this table are nominal masses. During MS and MS/MS optimization, the analyst should determine the precursor and product ion masses to one decimal place by locating the apex of the mass spectral peak place. These precursor and product ion masses (with one decimal place) should be used in the MS/MS method for all analyses.
[c] Ions used for quantitation purposes.
[d] Argon used as collision gas at a flow rate of 0.3 mL/min
[e] Analyte has multiple resolved chromatographic peaks due to linear and branched isomers. All peaks summed for quantitation purposes.

Table 10: Transition Ions

Compound	Precursor Ion (m/z)	Quant Ion (m/z)	Precursor formula	Primary Quant Ion formula	Cone Voltage (V)	Collision (eV)	2nd Qual Ion Mass (m/z)	2nd Qual Ion formula	Collision (eV)	Quant by:	Quantitation Reference
Native PFCs											
Perfluoorobutanoic acid	213	169	[CF3(CF2)2CO2]	[CF3(CF2)2]	27	8				ID	13CF3(13CF2)2 13COOH
Perfluoropentanoic acid	263	219	[CF3(CF2)3CO2]	[CF3(CF2)3]	27	8				IS	13CF3(13CF2)6 13COOH
Perfluoro-n-hexanoic acid	313	269	[CF3(CF2)4CO2]	[CF3(CF2)4]	27	20	119	[CF3CF2]	8	ID	CF3(CF2)3(13CF2)13COOH
Perfluoro-n-heptanoic acid	363	319	[CF3(CF2)5CO2]	[CF3(CF2)5]	27	12	169	[CF3(CF2)2]	8	IS	13CF3(13CF2)6 13COOH
Perfluoro-n-octanoic acid	413	369	[CF3(CF2)6CO2]	[CF3(CF2)6]	19	12	169	[CF3(CF2)2]	12	ID	CF3(CF2)3(13CF2)3 13COOH
Perfluoro-n-nonanoic acid	463	419	[CF3(CF2)7CO2]	[CF3(CF2)7]	20	13	219	[CF3(CF2)3]	12	ID	CF3(CF2)3(13CF2)4 13COOH
Perfluoro-n-decanoic acid	513	469	[CF3(CF2)8CO2]	[CF3(CF2)8]	21	11	219	[CF3(CF2)3]	13	ID	CF3(13CF2)8 13COOH
Perfluoro-n-undecanoic acid	563	519	[CF3(CF2)9CO2]	[CF3(CF2)9]	21	15	269	[CF3(CF2)4]	12	ID	CF3(13CF2)9COOH
Perfluoro-n-dodecanoic acid	613	569	[CF3(CF2)10CO2]	[CF3(CF2)10]	22	15	319	[CF3(CF2)5]	12	ID	CF3(CF2)9(13CF2)13COOH
Perfluoro-n-tridecanoic acid	663	619	[CF3(CF2)11CO2]	[CF3(CF2)11]	20	17	319	[CF3(CF2)5]	13	IS	13CF3(13CF2)6 13COOH
Perfluoro-n-tetradecanoic acid	713	669	[CF3(CF2)12CO2]	[CF3(CF2)12]	27	21	319	[CF3(CF2)5]	11	IS	13CF3(13CF2)6 13COOH
Perfluorobutanesulfonic acid	299	80	[CF3(CF2)3SO3]	[SO3]	70	40	99	[FSO3]	35	IS	13CF3(13CF2)6 13COOH
Perfluoro-n-hexane sulfonic acid 1	399	80	[CF3(CF2)5SO3]	[SO3]	30	45	99	[FSO3]	40	ID	CF3(CF2)5S(18O)2OH
Perfluoro-n-heptane sulfonic acid	449	80	[CF3(CF2)6SO3]	[SO3]	50	39	99	[FSO3]	38	IS	13CF3(13CF2)6 13COOH
Perfluoro-n-octanesulfonic acid	499	80	[CF3(CF2)7SO3]	[SO3]	80	45	99	[FSO3]	40	ID	CF3(CF2)3(13CF2)4SO3H
Perfluorooctane sulfonamide	498	78	[CF3(CF2)7SO2N H]	[SO2N]	80	40	478	[(CF2)8SO2N]	16	IS	13CF3(13CF2)6 13COOH
N-methylperfluoro-1-octanesulfonamide	512	169	[CF3(CF2)7SO2 N(CH3)]	[CF3(CF2)2]	27	45				IS	13CF3(13CF2)6 13COOH
N-ethylperfluoro-1-octanesulfonamide	526	169	[CF3(CF2)7SO2 N(C2H5)]	[CF3(CF2)2]	27	45				IS	13CF3(13CF2)6 13COOH
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	616	59	[CF3(CF2)7SO2N(CH3)C2H4OH·CH 3CO2]	[CH3CO2]	27	45				ID	CF3(CF2)7SO2N (CD3)C2D4OH·CH3COOH
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	630	59	[CF3(CF2)7SO2N(C2H5)C2H4OH·C H3CO2]	[CH3CO2]	27	45				IS	13CF3(13CF2)6 13COOH

Table 10: Transition Ions (continued)

Compound	Precursor Ion (m/z)	Quant Ion (m/z)	Precursor formula	Primary Quant Ion formula	Cone Voltage (V)	Collision (eV)	2nd Qual Ion Mass (m/z)	2nd Qual Ion formula	Collision (eV)	Quant by:	Quantitation Reference
Mass-labeled PFCs											
Perfluoro-n-[1,2,3,413C4]butanoic acid	217	172	[13CF3(13CF2)2 13CO2]	[13CF3(13CF2)2]	27	8				IS	13CF3(13CF2)6 13COOH
Perfluoro-n-[1,213C2]hexanoic acid	315	270	[CF3(CF2)3(13CF2)13CO2]	[CF3(CF2)3(13C F 2)]	27	8				IS	13CF3(13CF2)6 13COOH
Perfluoro-n-[1,2,3,413 C4]octanoic acid	417	372	[CF3(CF2)3 (13CF2)3 13CO2]	[CF3(CF2)3 (13CF2)3]	21	12				IS	13CF3(13CF2)6 13COOH
Perfluoro-n-[1,2,3,4,513 C5]nonanoic acid	468	423	[CF3(CF2)3 (13CF2)4 13CO2]	[CF3(CF2)3(13C F 2)4]	20	12				IS	13CF3(13CF2)6 13COOH
Perfluoro-n-[1,213C2]decanoic acid	515	470	[CF3(CF2)7(13CF2)13CO2]	[CF3(CF2)2(13C F 2)]	21	12				IS	13CF3(13CF2)6 13COOH
Perfluoro-n[1,2,3,4,5,6,7,8,9-13C9]decanoic acid	522	477	[CF3(13CF2)8 13CO2]	[CF3(13CF2)8]	20	12				IS	13CF3(13CF2)6 13COOH
Perfluoro-n-[1,213C2]undecanoic acid	565	520	[CF3(CF2)8(13CF2)13CO2]	[CF3(CF2)8(13C F 2)]	20	12				IS	13CF3(13CF2)6 13COOH
Perfluoro-n[2,3,4,5,6,7,8,9,10-13C9]undecanoic acid	572	528	[CF3(13CF2)9CO2]	[CF3(13CF2)9]	20	12				IS	13CF3(13CF2)6 13COOH
Perfluoro-n-[1,213C2]dodecanoic acid	615	570	[CF3(CF2)9 (13CF2)13CO2]	[CF3(CF2)9 (13CF2)]	22	12				IS	13CF3(13CF2)6 13COOH
Perfluoro-1-[1,218 O2]-hexanesulfonic acid	403	84	[CF3(CF2)5 S(18O)2O]	[S(18O)2O]	30	45	103	[FS(18O)2 O]	45	IS	13CF3(13CF2)6 13COOH
Perfluoro-n-[1,2,3,413 C4]-octanesulfonate	503	80	[CF3(CF2)3 (13CF2)4SO3]	[SO3]	40	45	99	[FSO3]	40	IS	13CF3(13CF2)6 13COOH
2-(Ndeuteriomethylperfluoro-1-octanesulfonamido)1,1,2,2-tetradeuterioethanol	623	59	[CF3(CF2)7SO2N (CD3)C2D4OH·C H3CO2]	[CH3CO2]	27	45				IS	13CF3(13CF2)6 13COOH
Injection Internal Standards (compound added after extraction, but prior to injection)											
Perfluoro-n[1,2,3,4,5,6,7,8-13 C8]octanoic acid	421	376	[13CF3(13CF2)6 13CO2]	[13CF3(3CF2)6]	21	12				ES	
2H-Perfluoro-[1,213C2]-2decenoic acid	459	394	[CF3(CF2)6 CF13CH13CO2]	[CF3(CF2)3 (13CF2)3]	21	11				ES	

CERTIFICATE OF ACCREDITATION

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

This is to certify that

Alpha Analytical Inc.
320 Forbes Blvd.
Mansfield, MA 02048

has been assessed by ANAB
and meets the requirements of

ISO/IEC 17025:2005 and DoD-ELAP

while demonstrating technical competence in the field of

TESTING

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

L2474
Certificate Number

ANAB Approval

Certificate Valid: 12/06/2017-05/30/2019
Version No. 003 Issued: 12/06/2017

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

ANSI-ASQ National Accreditation Board

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

Alpha Analytical, Inc.
320 Forbes Blvd
Mansfield, MA 02048
James Todaro
508-898-9220

TESTING

Valid to: **May 30, 2019** Certificate Number: **L2474**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#1-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#2
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#3-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#4/#10-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#9
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#7
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#6
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#5
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#8
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#19-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#14
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#30
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#18
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#11
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#17
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#12
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#27
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#13
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#24

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#16
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#32
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#15-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#34
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#23
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#54-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#29-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#50-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#26
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#25
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#53
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#-31
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#28
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#33
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#21/#20
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#51
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#45
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#22
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#73/#46
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#69
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#43
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#36
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#52
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#48
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#49
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#104-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#47
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#65/#75/#62
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#39
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#38
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#44
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#59
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#42
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#71
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#35
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#41

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#72
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#96
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#103
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#68/#64
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#40
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#37-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#100
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#94
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#57
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#67/#58
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#102
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#61
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#98
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#76
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#93
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#63
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#121
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#95/#88
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#74
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#155-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#70
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#66
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#91
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#80
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#55
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#92
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#89/#84
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#101/#90
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#56
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#113
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#99
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#150
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#60
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#152
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#119
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#83/#125/#112

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#86/#109
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#97
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#116
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#87/#111
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#145
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#148
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#79
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#154-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#78
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#136
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#117
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#115
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#85
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#120
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#110
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#81
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#151
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#135
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#82
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#144
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#147/#149
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#77-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#143/#139
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#124
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#108
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#107/#123
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#140
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#188-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#134
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#106
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#133
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#142
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#118
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#131
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#184
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#165

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#146
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#161
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#122
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#168
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#114
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#153
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#132
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#179
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#141
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#176
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#105
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#137
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#127
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#186
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#130/#164
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#178
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#138
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#163/#160
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#129/#158
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#182/#175
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#187
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#183
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#166
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#159
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#126-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#185
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#162
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#174
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#128
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#167
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#202-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#181
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#177
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#204/#200-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#171
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#173

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#172
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#192
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#156
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#157
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#180
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#193
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#197
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#191
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#199
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#198
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#201
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#170
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#190
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#196
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#203
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#169-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#208-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#207
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#189-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#195
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#194
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#205-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#206-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl10-BZ#209-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Monochlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Dichlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Trichlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Tetrachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Pentachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Hexachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Heptachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Octachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Nonachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Decachlorobiphenyl
GC/MS-SIM	EPA 8270D-SIM Isotope Dilution	1,4-Dioxane
GC/MS-SIM	EPA 522	1,4-Dioxane

Non-Potable Water		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	EPA 537	N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)
SPE/LC/MS/MS	EPA 537	N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)
SPE/LC/MS/MS	EPA 537	Perfluorobutanesulfonic acid (PFBS)
SPE/LC/MS/MS	EPA 537	Perfluorodecanoic acid (PFDA)
SPE/LC/MS/MS	EPA 537	Perfluorododecanoic acid (PFDoA)
SPE/LC/MS/MS	EPA 537	Perfluoroheptanoic acid (PFHpA)
SPE/LC/MS/MS	EPA 537	Perfluorohexanesulfonic acid (PFHxS)
SPE/LC/MS/MS	EPA 537	Perfluorohexanoic acid (PFHxA)
SPE/LC/MS/MS	EPA 537	Perfluorononanoic acid (PFNA)
SPE/LC/MS/MS	EPA 537	Perfluorooctanesulfonic acid (PFOS)
SPE/LC/MS/MS	EPA 537	Perfluorooctanoic acid (PFOA)
SPE/LC/MS/MS	EPA 537	Perfluorotetradecanoic acid (PFTA)
SPE/LC/MS/MS	EPA 537	Perfluorotridecanoic acid (PFTrDA)
SPE/LC/MS/MS	EPA 537	Perfluoroundecanoic acid (PFUnA)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	N-ethyl perfluorooctanesulfonamidoacetic acid N-EtFOSAA (cas# 2991-50-6)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	N-methyl perfluorooctanesulfonamidoacetic acid N-MeFOSAA (cas# 2355-31-9)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorobutanesulfonic acid (PFBS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorodecanoic acid PFDA (cas# 335-76-2)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorododecanoic acid PFDoA (cas# 307-55-1)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoroheptanoic acid PFHpA (cas# 375-85-9)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorohexanesulfonic acid (PFHxS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorohexanoic acid PFHxA (cas# 307-24-4)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorononanoic acid PFNA (cas# 375-95-1)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorooctanesulfonic acid (PFOS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorooctanoic acid PFOA (cas# 335-67-1)

Non-Potable Water		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorotridecanoic acid PFTrDA (cas# 72629-94-8)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoroundecanoic acid PFUnA (cas# 2058-94-8)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-n-tetradecanoic acid PFTeDA (cas# 376-06-7)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-n-pentanoic acid (PFPeA)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-n-butanoic acid PFBA (cas# 375-22-4)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-1-decanesulfonate (PFDS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-1-nonanesulfonate (PFNS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-1-heptanesulfonate (PFHpS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluorohexanesulfonate (PFHxS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-1-pentanesulfonate (PFPeS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-1-butanesulfonate (PFBS)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	Perfluoro-1-octanesulfonamide (FOSA)
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	1H,1H,2H,2H-perfluorodecane sulfonate (8:2) 8:2FTS
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	1H,1H,2H,2H-perfluorooctane sulfonate (6:2) 6:2FTS
SPE/LC/MS/MS	EPA 537 (Mod) Isotope Dilution	1H,1H,2H,2H-perfluorohexane sulfonate (4:2) 4:2FTS
Preparation	**Method**	**Type**
Extraction	EPA 3510C	Separatory Funnel
Cleanup	EPA 3630C	Silica Gel Cleanup
Cleanup	EPA 3660B	Sulfur Removal Cleanup
Cleanup	EPA 3665A	Sulfuric Acid Cleanup
Cleanup	EPA 3610 / EPA 3611	Alumina Column Cleanup

Drinking Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM Isotope Dilution	1,4-Dioxane
GC/MS-SIM	EPA 522	1,4-Dioxane
SPE/LC/MS/MS	EPA 537	N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)
SPE/LC/MS/MS	EPA 537	N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)
SPE/LC/MS/MS	EPA 537	Perfluorobutanesulfonic acid (PFBS)
SPE/LC/MS/MS	EPA 537	Perfluorodecanoic acid (PFDA)
SPE/LC/MS/MS	EPA 537	Perfluorododecanoic acid (PFDoA)
SPE/LC/MS/MS	EPA 537	Perfluoroheptanoic acid (PFHpA)
SPE/LC/MS/MS	EPA 537	Perfluorohexanesulfonic acid (PFHxS)
SPE/LC/MS/MS	EPA 537	Perfluorohexanoic acid (PFHxA)
SPE/LC/MS/MS	EPA 537	Perfluorononanoic acid (PFNA)
SPE/LC/MS/MS	EPA 537	Perfluorooctanesulfonic acid (PFOS)
SPE/LC/MS/MS	EPA 537	Perfluorooctanoic acid (PFOA)
SPE/LC/MS/MS	EPA 537	Perfluorotetradecanoic acid (PFTA)
SPE/LC/MS/MS	EPA 537	Perfluorotridecanoic acid (PFTrDA)
SPE/LC/MS/MS	EPA 537	Perfluoroundecanoic acid (PFUnA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#1-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#2
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#3-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#4/#10-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#9
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#7
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#6
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#5
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#8
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#19-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#14
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#30
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#18

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#11
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#17
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#12
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#27
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#13
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#24
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#16
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#32
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#15-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#34
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#23
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#54-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#29-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#50-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#26
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#25
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#53
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#-31
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#28
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#33
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#21/#20
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#51
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#45
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#22
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#73/#46
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#69
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#43
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#36
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#52
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#48
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#49
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#104-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#47
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#65/#75/#62
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#39
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#38

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#44
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#59
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#42
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#71
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#35
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#41
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#72
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#96
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#103
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#68/#64
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#40
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#37-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#100
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#94
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#57
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#67/#58
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#102
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#61
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#98
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#76
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#93
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#63
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#121
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#95/#88
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#74
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#155-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#70
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#66
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#91
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#80
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#55
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#92
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#89/#84
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#101/#90
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#56
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#113

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#99
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#150
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#60
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#152
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#119
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#83/#125/#112
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#86/#109
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#97
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#116
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#87/#111
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#145
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#148
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#79
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#154-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#78
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#136
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#117
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#115
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#85
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#120
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#110
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#81
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#151
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#135
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#82
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#144
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#147/#149
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#77-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#143/#139
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#124
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#108
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#107/#123
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#140
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#188-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#134
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#106

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#133
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#142
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#118
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#131
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#184
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#165
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#146
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#161
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#122
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#168
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#114
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#153
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#132
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#179
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#141
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#176
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#105
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#137
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#127
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#186
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#130/#164
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#178
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#138
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#163/#160
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#129/#158
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#182/#175
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#187
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#183
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#166
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#159
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#126-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#185
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#162
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#174
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#128
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#167

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#202-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#181
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#177
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#204/#200-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#171
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#173
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#172
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#192
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#156
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#157
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#180
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#193
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#197
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#191
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#199
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#198
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#201
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#170
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#190
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#196
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#203
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#169-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#208-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#207
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#189-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#195
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#194
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#205-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#206-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl10-BZ#209-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Monochlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Dichlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Trichlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Tetrachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Pentachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Hexachlorobiphenyls

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Heptachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Octachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Nonachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Decachlorobiphenyl
Gravimetric	SM 2540G	Percent Total Solids
Preparation	**Method**	**Type**
Extraction	EPA 3570	Microscale Extraction (MSE)
Waste Dilution	EPA 3580A	Waste Dilution
Cleanup	EPA 3630C	Silica Gel Cleanup
Cleanup	EPA 3660B	Sulfur Removal Cleanup
Cleanup	EPA 3665A	Sulfuric Acid Cleanup
Cleanup	EPA 3610 / EPA 3611	Alumina Column Cleanup

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#1-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#2
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl1-BZ#3-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#4/#10-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#9
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#7
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#6
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#5
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#8
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#19-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#14
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#30
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#18
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#11
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#17
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#12
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#27
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#13
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#24
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#16

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#32
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl2-BZ#15-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#34
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#23
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#54-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#29-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#50-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#26
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#25
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#53
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#-31
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#28
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#33
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#21/#20
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#51
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#45
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#22
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#73/#46
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#69
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#43
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#36
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#52
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#48
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#49
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#104-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#47
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#65/#75/#62
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#39
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#38
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#44
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#59
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#42
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#71
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#35
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#41
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#72

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#96
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#103
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#68/#64
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#40
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl3-BZ#37-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#100
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#94
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#57
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#67/#58
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#102
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#61
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#98
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#76
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#93
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#63
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#121
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#95/#88
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#74
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#155-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#70
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#66
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#91
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#80
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#55
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#92
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#89/#84
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#101/#90
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#56
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#113
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#99
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#150
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#60
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#152
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#119
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#83/#125/#112
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#86/#109

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#97
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#116
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#87/#111
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#145
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#148
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#79
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#154-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#78
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#136
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#117
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#115
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#85
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#120
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#110
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#81
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#151
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#135
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#82
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#144
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#147/#149
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl4-BZ#77-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#143/#139
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#124
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#108
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#107/#123
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#140
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#188-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#134
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#106
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#133
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#142
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#118
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#131
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#184
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#165
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#146

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#161
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#122
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#168
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#114
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#153
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#132
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#179
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#141
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#176
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#105
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#137
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#127
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#186
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#130/#164
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#178
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#138
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#163/#160
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#129/#158
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#182/#175
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#187
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#183
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#166
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#159
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl5-BZ#126-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#185
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#162
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#174
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#128
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#167
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#202-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#181
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#177
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#204/#200-Cal
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#171
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#173
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#172

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#192
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#156
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#157
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#180
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#193
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#197
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#191
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#199
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#198
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#201
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#170
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#190
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#196
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#203
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl6-BZ#169-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#208-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#207
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl7-BZ#189-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#195
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#194
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl8-BZ#205-RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl9-BZ#206-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Cl10-BZ#209-Cal/RTW
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Monochlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Dichlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Trichlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Tetrachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Pentachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Hexachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Heptachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Octachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Nonachlorobiphenyls
GC/MS-SIM	EPA 8270D-SIM / EPA 680	Decachlorobiphenyl
Preparation	**Method**	**Type**
Extraction	EPA 3570	Microscale Extraction (MSE)
Extraction	Alpha SOP ID 2264	Tissue Extraction
Waste Dilution	EPA 3580A	Waste Dilution

Biological Tissue		
Technology	**Method**	**Analyte**
Cleanup	EPA 3630C	Silica Gel Cleanup
Cleanup	EPA 3660B	Sulfur Removal Cleanup
Cleanup	EPA 3665A	Sulfuric Acid Cleanup
Cleanup	EPA 3610 / EPA 3611	Alumina Column Cleanup

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	1,1,1,2-tetrachloroethane
GC/MS	EPA TO-15	1,1,1-trichloroethane
GC/MS	EPA TO-15	1,1,2,2-tetrachloroethane
GC/MS	EPA TO-15	1,1,2-trichloroethane
GC/MS	EPA TO-15	1,1-dichloroethane
GC/MS	EPA TO-15	1,1-dichloroethene
GC/MS	EPA TO-15	1,1-dichloropropene
GC/MS	EPA TO-15	1,2,3-trichlorobenzene
GC/MS	EPA TO-15	1,2,3-trichloropropane
GC/MS	EPA TO-15	1,2,4-trichlorobenzene
GC/MS	EPA TO-15	1,2,4-trimethylbenzene
GC/MS	EPA TO-15	1,2-dibromo-3-chloropropane
GC/MS	EPA TO-15	1,2-dibromoethane
GC/MS	EPA TO-15	1,2-dichlorobenzene
GC/MS	EPA TO-15	1,2-dichloroethane
GC/MS	EPA TO-15	1,2-dichloropropane
GC/MS	EPA TO-15	1,3,5-trimethylbenzene
GC/MS	EPA TO-15	1,3-butadiene
GC/MS	EPA TO-15	1,3-dichlorobenzene
GC/MS	EPA TO-15	1,3-dichloropropane
GC/MS	EPA TO-15	1,4-dichlorobenzene
GC/MS	EPA TO-15	1,4-dioxane
GC/MS	EPA TO-15	2,2,4-trimethylpentane
GC/MS	EPA TO-15	2,2-dichloropropane
GC/MS	EPA TO-15	2-butanone
GC/MS	EPA TO-15	2-chlorotoluene
GC/MS	EPA TO-15	2-hexanone
GC/MS	EPA TO-15	3-chloropropene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	4-chlorotoluene
GC/MS	EPA TO-15	4-ethyl toluene
GC/MS	EPA TO-15	4-methyl-2-pentanone (MIBK)
GC/MS	EPA TO-15	acetone
GC/MS	EPA TO-15	acetonitrile
GC/MS	EPA TO-15	acrolein
GC/MS	EPA TO-15	acrylonitrile
GC/MS	EPA TO-15	benzene
GC/MS	EPA TO-15	benzyl chloride
GC/MS	EPA TO-15	bromobenzene
GC/MS	EPA TO-15	bromodichloromethane
GC/MS	EPA TO-15	bromoform
GC/MS	EPA TO-15	bromomethane
GC/MS	EPA TO-15	carbon disulfide
GC/MS	EPA TO-15	carbon tetrachloride
GC/MS	EPA TO-15	chlorobenzene
GC/MS	EPA TO-15	chlorodifluoromethane
GC/MS	EPA TO-15	chloroethane
GC/MS	EPA TO-15	chloroform
GC/MS	EPA TO-15	chloromethane
GC/MS	EPA TO-15	cis-1,2-dichloroethene
GC/MS	EPA TO-15	cis-1,3-dichloropropene
GC/MS	EPA TO-15	cyclohexane
GC/MS	EPA TO-15	dibromochloromethane
GC/MS	EPA TO-15	dibromomethane
GC/MS	EPA TO-15	dichlorodifluoromethane
GC/MS	EPA TO-15	dichlorofluoromethane
GC/MS	EPA TO-15	diisopropyl ether
GC/MS	EPA TO-15	ethanol
GC/MS	EPA TO-15	ethyl acetate
GC/MS	EPA TO-15	ethyl ether
GC/MS	EPA TO-15	ethylbenzene
GC/MS	EPA TO-15	Freon 113
GC/MS	EPA TO-15	Freon-114
GC/MS	EPA TO-15	n-heptane
GC/MS	EPA TO-15	hexachlorobutadiene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	hexane
GC/MS	EPA TO-15	isopropyl alcohol
GC/MS	EPA TO-15	isopropylbenzene
GC/MS	EPA TO-15	m+p-xylene
GC/MS	EPA TO-15	methanol
GC/MS	EPA TO-15	methylene chloride
GC/MS	EPA TO-15	methyl methacrylate
GC/MS	EPA TO-15	MTBE
GC/MS	EPA TO-15	naphthalene
GC/MS	EPA TO-15	n-butylbenzene
GC/MS	EPA TO-15	n-propylbenzene
GC/MS	EPA TO-15	octane
GC/MS	EPA TO-15	o-xylene
GC/MS	EPA TO-15	n-pentane
GC/MS	EPA TO-15	p-isopropyltoluene
GC/MS	EPA TO-15	propane
GC/MS	EPA TO-15	propylene
GC/MS	EPA TO-15	sec-butylbenzene
GC/MS	EPA TO-15	styrene
GC/MS	EPA TO-15	tert-amyl methyl ether
GC/MS	EPA TO-15	tert-butlybenzene
GC/MS	EPA TO-15	tert-butyl ethyl ether
GC/MS	EPA TO-15	tetrachloroethene
GC/MS	EPA TO-15	tetrahydrofuran
GC/MS	EPA TO-15	toluene
GC/MS	EPA TO-15	trans-1,2-dichloroethene
GC/MS	EPA TO-15	trans-1,3-dichloropropene
GC/MS	EPA TO-15	trichloroethene
GC/MS	EPA TO-15	trichlorofluoromethane
GC/MS	EPA TO-15	vinyl acetate
GC/MS	EPA TO-15	vinyl bromide
GC/MS	EPA TO-15	vinyl chloride
GC/MS	EPA TO-15	decane
GC/MS	EPA TO-15	undecane
GC/MS	EPA TO-15	butane
GC/MS	EPA TO-15	nonane

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	tert butyl alcohol
GC/MS	EPA TO-15	dodecane
GC/MS	EPA TO-15	butyl acetate
GC/MS	EPA TO-15	3-methylthiophene
GC/MS	EPA TO-15	2-ethylthiophene
GC/MS	EPA TO-15	2-methylthiophene
GC/MS	EPA TO-15	thiophene
GC/MS	EPA TO-15	benzothiophene
GC/MS	EPA TO-15	1,2,3-trimethylbenzene
GC/MS	EPA TO-15	indene
GC/MS	EPA TO-15	1,2,4,5-tetramethylbenzene
GC/MS	EPA TO-15	indan
GC/MS	EPA TO-15	1-methylnaphthalene
GC/MS	EPA TO-15	2-methylnaphthalene
GC/MS	EPA TO-15	acetaldehyde

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2474

Vice President

ATTACHMENT 4

RESPONSES TO COMMENTS

Army Responses to EPA Comments on the
Work Plan for PFAS Sampling of Selected Community and Private Wells
Former Fort Devens Army Installation, Devens, MA
13 June 2018

The following U.S. Army responses pertain to the U.S. Environmental Protection Agency (EPA) comments, dated 24 May 2018, on the Work Plan for PFAS Sampling of Selected Community and Private Wells at the *Former Fort Devens Army Installation, Devens, Massachusetts*, dated 16 May 2018.

E-mailed Comments

Comment 1, Page 3 – There is a disconnect between some of the information given here and what is provided in the lab SOP/Method 537. Method 537 states that polypropylene containers shall be used along with polypropylene caps not HDPE. The SOP also states that 3 containers should be collected per sample. Please revise accordingly.

> **Response** – The third paragraph under the Field Activities section has been reworded to read as follows:
>
> > *"Samples will be collected in three 250 milliliter (mL) polypropylene bottles fitted with polypropylene screw caps."*

Comment 2, Page 3 – Please include the holding time and storage temperature in this section.

> **Response** – The following text has been added to the third paragraph under the Field Activities section:
>
> > *"Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Aqueous samples must be extracted within 14 days and extracts should be stored at room temperature and analyzed within 28 days after extraction."*

Comment 3, Page 3 – Please clarify that at least one field reagent blank will be submitted with each sample set.

> **Response** – Field reagent blanks (FRB) will be submitted to be representative of field conditions. The following text has been added to the fourth paragraph under the Field Activities section:
>
> > *"One FRB will be collected during the community well field sampling activity and one FRB will be collected during the residential well sampling activity. Additional FRB samples may be collected if warranted by site conditions."*

Comment 4 – Is this plan written as an addendum to the Long-Term Monitoring and Maintenance Program QAPP from 2016? For instance, there are no chain of custody/sample

documentation procedures provided in this plan, but there are in the Long-Term Monitoring QAPP. Will those same COC procedures be used? If so, please state this in the plan.

Response – The chain-of-custody and sample documentation procedures will follow the 2016 LTM QAPP. The following text has been added to the fourth paragraph under the Field Activities section:

> *"Samples will be recorded on chains-of-custody, packed in coolers, and shipped on ice to the analytical laboratory in accordance with the 2016 Long Term Monitoring and Maintenance Program (LTMMP) QAPP. The sample register, listing field sample locations and associated quality control samples, is provided in Table 1."*

<u>Verbal Comments from the 24 May 2018 Meeting</u>

Comment 5 – EPA disagrees with the Work Plan's description of the regional hydrogeology because more site-specific data are needed to understand the overall groundwater flow patterns in the area.

Response – The last sentence of the first paragraph in the Regional Hydrogeology section has been revised to read as follows:

> *"Based on the current review of site topography, overburden soil thickness, overburden soil characteristics, modeled groundwater flow contours, groundwater hydraulic gradients, and aquifer transmissivity, it is unlikely that potential PFAS impacts to these wells would be a result of groundwater originating from the Former Fort Devens; however, site-specific hydrogeology and its relation to the current understanding of the regional hydrogeology will be further investigated during a Remedial Investigation commencing in 2018."*

Comment 6 – More details are needed regarding the construction of the community/private wells and their depth in the aquifer.

Response – The following text has been added to the Field Activities section:

> *"Information regarding the well construction (e.g., type, depth) will be collected from available town and state databases and/or interviews of the well owners."*

Comment 7 – The Work Plan should include additional private wells located across the Nashua River, west of the airfield. Specifically, the Work Plan should include private wells along Kittredge Road and Lawton Road in Shirley, MA as well as the hydroponics lettuce facility.

Response – The Work Plan has been modified to include four additional private wells in the Town of Shirley, west of the airfield, as well as the hydroponics farm on Walker Road.

Note: The Town Forest well located in Groton was removed from the Work Plan because it was previously sampled for PFAS in September 2016 (and was non-detect for PFOS/PFOA).

Comment 8 – The Work Plan should include the one identified private well located north of the Appleworks well along Ayer Road.

Response – The requested well has been added (354 Ayer Road, Harvard, MA).

Comment 9 – The Army should revise and resubmit the Work Plan to include the additional wells along with any of the available well construction information, although the work plan and sampling should not be delayed to find the well construction information if it is not currently available.

Response – The following text has been added to the Field Activities section:

> *"Information regarding the well construction (e.g., type, depth) will be collected from available town and state databases and/or interviews of the well owners."*