Qualifications for Battelle's Accredited PFAS Lab

PFAS Method Descriptions

PFAS in Drinking Water (EPA Method 537.1)

Drinking water samples are extracted per the requirements of EPA Method 537.1 (March 2020). All samples are collected in polypropylene bottles, pre-preserved with Trizma. Samples are fortified with labeled surrogates in the original sample containers from the field. The drinking water samples are extracted using a solid phase extraction (SPE) cartridge and eluted from the SPE with methanol. Extracts are concentrated to dryness, reconstituted with 96:4 methanol/ water (V/V), and fortified with internal standard. Extracts are then transferred for liquid chromatography tandem mass spectrometry (LC-MS/MS) analysis. Samples extracts are quantified using internal standard methods. Results are reported on a ng/L basis.

PFAS in Drinking Water (EPA Method 533)

Drinking water samples are extracted per the requirements of EPA Method 533 (November 2019). All samples are collected in polypropylene bottles, pre-preserved with ammonium acetate. Samples are fortified with labeled surrogates (Isotope Dilution Analogs) in the original sample containers from the field. The drinking water samples are extracted using a weak ion exchange (WAX) SPE cartridge and eluted from the SPE with basic methanol. Extracts are concentrated to dryness, reconstituted with 80:20 methanol/ water (V/V), and fortified with internal standard (Isotope Performance Standards). Extracts are then transferred for LC-MS/MS analysis. Samples extracts are quantified using isotope dilution methods. Results are reported on a ng/L basis.

PFAS in Non-potable Water, Solid, and Tissue (EPA 1633)

Non-Potable Water: All non-potable water samples are pre-screened prior to SPE. For SPE, water samples are fortified with surrogates (extracted internal standards (EIS)) in the original sample container from the field. The water samples are extracted using a combined WAX/carbon (GCB) SPE and eluted from the SPE with basic methanol. Extracts are acidified, fortified with internal standards (non-extracted internal standards (NIS)), and transferred for LC-MS/MS analysis. Results are reported on an ng/L basis.

Solid: A well homogenized subsample of soil or sediment is weighed into a centrifuge tube and fortified with EIS. Samples are extracted serially with three separate extractions using solvent (basic methanol). The combined extract is concentrated under nitrogen in a heated block. The concentrated extract is reconstituted with water and further refined using WAX/GCB SPE and eluted from the SPE with basic methanol. Extracts are acidified, fortified with NIS, and transferred for LC-MS/MS analysis. Results are reported in ng/g on a dry weight basis.

Tissue: A well homogenized subsample of tissue sample is weighed into a centrifuge tube and fortified with EIS. Samples are extracted serially with three separate extractions using fresh solvent (basic methanol, acetonitrile, then basic methanol). The combined extract is concentrated under nitrogen in a heated block. The concentrated extract is reconstituted with water and further refined using WAX/GCB SPE and eluted from the SPE with basic methanol. Extracts are acidified, fortified with NIS, and transferred for LC-MS/MS analysis. Results are reported in ng/g on a wet weight basis.

Isotope Dilution Analysis: PFAS are measured by LC-MS/MS in the multiple reaction monitoring (MRM) mode. Except for NFDHA, PFEESA, PFMPA, PFMBA, PFBA, PFPeA, MeFOSE, and EtFOSE, two transitions are monitored, one for quantitation and the other for confirmation, and the ion ratios are monitored for all target analytes. An initial calibration consisting of target analytes, labeled analogs, and internal standards is analyzed prior to analysis of samples to demonstrate the linear range of instrument. Target PFAS are quantified using the isotope dilution method following EPA Method 1633 and QSM 5.4 Table B-24. The isotopically labeled analog of an analyte (EIS) is used for quantitation if commercially available. If a labeled analog is not commercially available, quantitation is performed using the EIS with the closest retention time and similar chemistry when available. Recoveries of the EIS analytes used for quantification are calculated against the NIS to monitor the extraction efficiency of the method analytes.

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PFAS by Isotope Dilution Analysis (Compliant With DOD QSM 5.4 Table B-15)

Non-Potable Water: All non-potable water samples are pre-screened prior to SPE. For SPE, water samples are fortified with EIS in the original sample container from the field. The water samples are extracted using WAX SPE and eluted from the SPE with basic methanol. Extracts are further refined with ENVI-Carb, fortified with NIS, acidified, and transferred for LC-MS/MS analysis. Results are reported on an ng/L basis.

Solid and Tissue: A well homogenized subsample of soil, sediment, or tissue sample is weighed into a centrifuge tube and fortified with EIS. Samples are extracted serially with two separate extractions using fresh solvent (basic methanol). The combined extract is further refined using an ENVI-Carb to remove possible matrix interferences. The refined extracts are reconstituted with water and further refined using WAX SPE and eluted from the SPE with basic methanol. Extracts are acidified, fortified with NIS, and transferred for LC-MS/MS analysis. Results are reported in ng/g on a dry weight basis for soil and sediment and ng/g on a wet weight basis for tissues.

Isotope Dilution Analysis: Analysis follows the procedures outlined under EPA 1633 and QSM 5.4 Table B-24, except following guidance in QSM 5.4 Table B-15.

Total Oxidizable Precursor (TOP) Assay

TOP Assay is performed on a sub-sample of a non-potable water sample or on an unfortified extract of a solid or tissue sample, following methods like those described by Houtz and Sedlak 2012. Samples are to convert oxidizable precursors into perfluoroalkyl acids. All samples are analyzed before the oxidation following the extraction and analysis methods described above and again post oxidation to compare the changes in concentrations of perfluoroalkyl acids.

Laboratory Accreditations

Battelle's Analytical laboratory, located in Norwell Massachusetts, holds national accreditation through the National Environmental Laboratory Accreditation Program (NELAP), Department of Defense (DoD) Quality Systems Manual (QSM) version 5.4 accreditation through the Environmental Laboratory Accreditation Program (ELAP), as well as several state level accreditations specifically for PFAS in various matrices, as available. The table below includes the analytical methods and analytes covered under our NELAP and DoD NELAP accreditations. Pending accreditations include AFFF by Method 1633 for DoD, and 537.1, 533, and 1633 for the Department of Energy.

Analyte	CAS No.	EPA 537.1[1]	EPA 533[1]	EPA 1633[2]	QSM 5.4 Table B-15[2]	Analyte	CAS No.	EPA 537.1[1]	EPA 533[1]	EPA 1633[2]	QSM 5.4 Table B-15[2]
NFDHA	151772-58-6		X	X	X	NEtFOSE	1691-99-2			X	X
PFEESA	113507-82-7		X	X	X	PFOSA	754-91-6			X	X
PFMPA	377-73-1		X	X	X	PFBS	375-73-5	X	X	X	X
PFMBA	863090-89-5		X	X	X	PFPeS	2706-91-4		X	X	X
PFBA	375-22-4		X	X	X	PFHxS	355-46-4	X	X	X	X
PFPeA	2706-90-3		X	X	X	PFHpS	375-92-8		X	X	X
PFHxA	307-24-4	X	X	X	X	PFOS	1763-23-1	X	X	X	X
PFHpA	375-85-9	X	X	X	X	PFNS	68259-12-1			X	X
PFOA	335-67-1	X	X	X	X	PFDS	335-77-3			X	X
PFNA	375-95-1	X	X	X	X	PFDoS	79780-39-5			X	X
PFDA	335-76-2	X	X	X	X	4:2FTS	757124-72-4		X	X	X
PFUnA	2058-94-8	X	X	X	X	6:2FTS	27619-97-2		X	X	X
PFDoA	307-55-1	X	X	X	X	8:2FTS	39108-34-4		X	X	X
PFTrDA	72629-94-8	X		X	X	10:2FTS	120226-60-0				X
PFTeDA	376-06-7	X		X	X	3:3 FTCA	356-02-5			X	X
PFHxDA	67905-19-5				X	5:3 FTCA	914637-49-3			X	X
PFODA	16517-11-6				X	7:3 FTCA	812-70-4			X	X
NMeFOSAA	2355-31-9	X		X	X	HFPO-DA	13252-13-6	X	X	X	X
NEtFOSAA	2991-50-6	X		X	X	Adona	919005-14-4	X	X	X	X
NMeFOSA	31506-32-8			X	X	9Cl-PF3ONS	756426-58-1	X	X	X	X
NEtFOSA	4151-50-2			X	X	11Cl-PF3OUdS	763051-92-9	X	X	X	X
NMeFOSE	24448-09-7			X	X						

Sources
[1]Drinking water only
[2]Non-potable water, solid, and tissue

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