Note d'application

Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Accordance with EPA 1633 Part 3: Analysis of Soil and Tissue

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Abstract

US EPA Method 1633 has become the foundational method for analysis of PFAS in non-potable water matrices, soils, biosolids, and tissues in the United States. The method consists of sample preparation using weak anion exchange (WAX) solid phase extraction (SPE) with graphitized carbon black (GCB) clean up. This application note is the third in a series demonstrating a comprehensive solution for performing the EPA 1633 methodology. The focus of this note is preparation and analysis of soil and fish tissue samples utilizing a bilayer dual-phase SPE cartridge and LC-MS/MS method on an ACQUITY™ Premier BSM FTN LC System coupled to a Xevo™ TQ Absolute Tandem Quadrupole Mass Spectrometer.

Benefits

- An end-to-end workflow is presented for PFAS analysis in soil and fish tissue samples following the EPA 1633 procedure
- A bilayer dual-phase SPE cartridge containing WAX and GCB was utilized to reduce the debris and hazards of working with dispersive GCB as well as further reducing sample preparation time
- Performance criteria of EPA 1633 are met for solids and tissues demonstrating equivalency of the workflow

used

- Performance of the workflow is demonstrated by easily passing qualifications of a Waters™ ERA certified reference material

Introduction

US EPA Method 1633 was first introduced in August 2021 to become the foundational method for analysis of PFAS in non-potable water matrices, soils, biosolids, and tissues.[1] EPA 1633 was finalized in January 2024 and is multi-lab validated for each type of sample matrix included in the method.[2] The method covers 40 PFAS and utilizes isotope dilution calibration and quantitation. Required sample preparation differs slightly depending on sample type, but all sample types utilize solid phase extraction (SPE) on a weak anion exchange (WAX) cartridge in combination with graphitized carbon black (GCB) clean up. EPA 1633 was created to support sample analysis for the Clean Water Act (CWA) and Department of Defense (DoD) monitoring and remediation, but it covers such a wide range of matrices and compounds that its applicability is expected to be widespread.

This is the third in a series of application notes addressing sample preparation, analysis, and method performance of EPA 1633 using a comprehensive workflow of Waters technologies. This application note will focus on the preparation of authentic soil and tissue samples with analysis utilizing the LC-MS/MS method established in Part 1 on an ACQUITY Premier BSM FTN UPLC System coupled to a Xevo TQ Absolute Mass Spectrometer.[3] The use of a combined WAX and GCB sample extraction and cleanup workflow is demonstrated on soil and fish tissue.

Experimental

Sample Preparation

Samples discussed in this application note include soil and fish tissue. Salmon was used as the fish tissue matrix studied and was sourced at a local market. The fish tissue was homogenized using a blender before subsampling. The soil was a custom soil reference material created by the ERA that is similar to the PFAS in Soil CRM currently offered (Item number 603 <https://www.eraqc.com/pfas-in-soil-soil-

era001675?returnurl=%2fsearch%3fq%3d603%26analytessearchonly%3dfalse>). It contained the 40 EPA 1633 PFAS at a known concentration. Samples were frozen until sample analysis according to EPA 1633 guidelines and holding times.

Oasis GCB/WAX for PFAS dual-phase SPE cartridges (p/n: 186011112 < https://www.waters.com/nextgen/global/shop/sample-preparation--filtration/186011112-oasis-gcb-wax-for-pfas-analysis-6cc-vac-cartridge-50-mg-gcb-200-.html>) containing both WAX and GCB sorbents required for sample cleanup were used instead of using dispersive GCB. For soil and tissue analysis, the GCB is packed on top of the WAX sorbent to replicate the EPA 1633 where the GCB is used to clean the sample prior to WAX SPE.

Full sample preparation details are listed in Figures 1 and 2 and are adapted directly from the EPA 1633 method. Figure 1 details the two different extraction procedures used for soils/solids and tissues. Figure 2 details the SPE procedure used for all sample types. The dispersive GCB step was combined into the SPE cartridge, as described previously, providing the convenience of minimizing complications from using loose GCB material and reducing the number of steps during sample preparation without compromising the method.

Soil samples were spiked with 0.25–2 ng/g (sample concentration equivalent) of the required extracted internal standard (EIS) prior to extraction and 0.25–1.0 ng/g (sample concentration equivalent) of the required non-extracted internal standard (NIS) after extraction. Tissue samples were spiked with 0.625–5 ng/g (sample concentration equivalent) of the required extracted internal standard (EIS) prior to extraction and 0.625–2.5 ng/g (sample concentration equivalent) of the required non-extracted internal standard (NIS) after extraction. Individual concentrations vary dependent on the concentration of each component in the Wellington standard mixes. The calibration curve range for each analyte (in vial equivalent) is listed in Appendix Table 2 and was determined from the data acquired and presented in Part 1 of this application note series.[3] All standards were obtained as mixes from Wellington Laboratories.

Soils/Solids

- Weigh **5 g sample** into 50 mL tube
    - **Spike** with Extracted Internal Standard Mix (MPFAC-HIF-ES from Wellington)
- Add **10 mL 0.3% ammonium hydroxide in methanol**
    - Shake 30 mins, centrifuge, transfer supernatant to clean tube
- Add **15 mL 0.3% ammonium hydroxide in methanol**
- Shake 30 mins, centrifuge, transfer supernatant (combine with previous step)
- Add **5 mL 0.3% ammonium hydroxide in methanol**
    - Shake 5 mins, centrifuge, transfer supernatant (combine with previous steps)
- **Concentrate** under nitrogen to 7 mL
- **Reconstitute** up to 50 mL with water
- Check pH and adjust to approximately **pH 6**
- Proceed to **SPE** procedure

Tissues

- Weigh **2 g sample** into 15 mL tube
    - **Spike** with Extracted Internal Standard Mix (MPFAC-HIF-ES from Wellington)
- Add **10 mL 0.05 M KOH in methanol**
    - Shake gently for 16 hours, centrifuge, transfer supernatant to clean tube
- Add **10 mL acetonitrile**
    - Sonicate 30 mins, centrifuge, transfer supernatant (combine with previous step)
- Add **5 mL 0.05 M KOH in methanol**
    - Shake 5 mins, centrifuge, transfer supernatant (combine with previous steps)
- Add **1 mL water**
- **Concentrate** under nitrogen to 2.5 mL
- **Reconstitute** up to 50 mL with water
- Check pH and adjust to approximately **pH 6**
- Proceed to **SPE** procedure

Figure 1. Full method details of the extraction procedure used for soil and tissue. Adapted from EPA Method 1633.

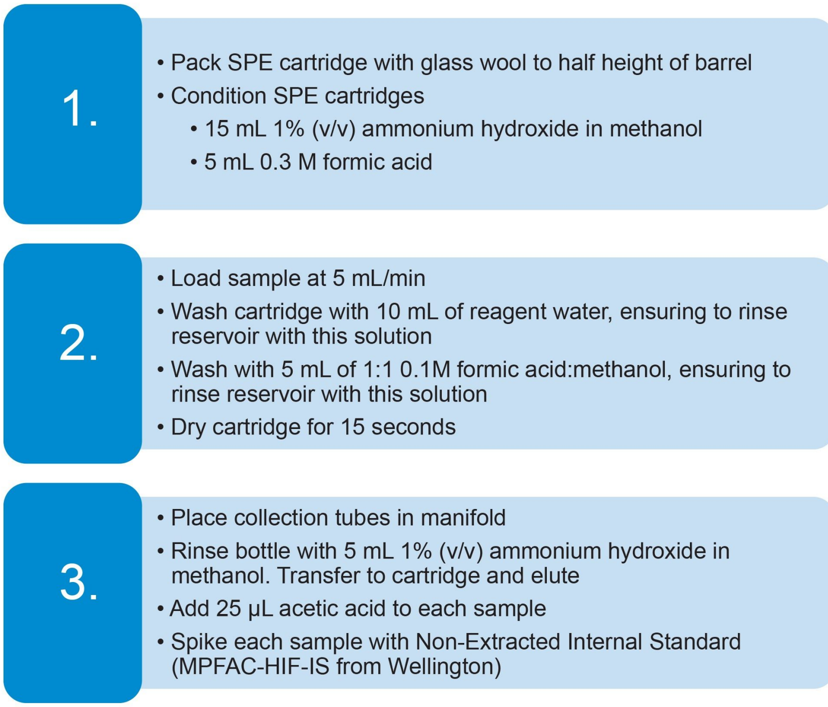

Figure 2. Full method details of the SPE procedure used for soil and tissue. Adapted from EPA Method 1633.

LC Conditions

LC system:	ACQUITY Premier BSM with FTN
Vials:	700 µL Polypropylene Screw Cap Vials (p/n: 186005219)
Analytical column:	ACQUITY Premier BEH™ C_{18} 2.1 x 50 mm, 1.7 µm (p/n: 186009452)
Isolator column:	Atlantis Premier BEH C18 AX 2.1 x 50 mm, 5.0 µm (p/n: 186009407)

Column temperature:	35 °C
Sample temperature:	10 °C
PFAS kit:	PFAS Install Kit with OASIS™ WAX 150 mg (p/n: 176004548)
Injection volume:	2 µL
Flow rate:	0.3 mL/min
Mobile phase A:	2 mM ammonium acetate in water
Mobile phase B:	2 mM ammonium acetate in acetonitrile

Gradient Table

Time (min)	%A	%B	Curve
0	95	5	initial
0.5	75	25	6
3	50	50	6
6.5	15	85	6
7	5	95	6
8.5	5	95	6
9	95	5	6
11	95	5	6

MS Conditions

MS system:	Xevo TQ Absolute Mass Spectrometer
Ionization mode:	ESI-

Capillary voltage:	0.5 kV
Source temperature:	100 °C
Desolvation temperature:	350 °C
Desolvation flow:	900 L/hr
Cone flow:	150 L/hr
MRM method:	See Appendix for Full MRM Method details

Data Management

Software:	waters_connect™ for Quantitation

Results and Discussion

Recovery in Soil and Tissue Samples

EPA 1633 is a performance-based method that allows modifications as long as the performance criteria outlined in the method are all met. The only modification presented in this work was to use a bilayer dual-phase SPE cartridge that combines the otherwise dispersive GCB clean up step into the WAX SPE cartridge. GCB is difficult to work with and accurately measure, therefore utilizing a bilayer cartridge eliminates the untidy dispersive GCB step. More importantly, combining the GCB cleanup step into the SPE extraction saves valuable time in the laboratory during the sample preparation process. Additionally, less preparation steps allow for fewer opportunities for introduction of unintended PFAS sample contamination. For this work, a cartridge with the GCB stacked on top of the WAX was utilized to replicate the workflow of EPA 1633 where the GCB clean up step occurs before loading the sample onto the WAX cartridge.

One of the important performance criteria that must be established in order to prove equivalence of this approach is the target analyte (natives) and extracted internal standard (EIS) recovery acceptance limits (See Table 7 within that document).[1] The individual recovery performance of the bilayer dual-phase SPE cartridge for soil and fish tissue are listed for each EIS in Table 1. The data reported in Table 1 is the average recovery and %RSD for 3 replicate extractions of each matrix type. The mean recovery of all EIS among the soil and tissue samples extracted was 81% and 85%, respectively, with mean RSDs of 2.8% and 9.2% for soil and tissue, respectively.

Figure 3 directly compares the average recovery for the EIS in each sample type with the allowable recoveries in EPA 1633 Table 7. All PFAS analyzed in this study in both soil and fish tissue, were easily within the recovery acceptance limits for each compound, and in all cases were significantly above the minimum recovery level, demonstrating that the bilayer GCB/WAX SPE cartridge has equivalent performance as using dispersive GCB and is fit-for-purpose.

Compound	Soil		Fish tissue	
	Average recovery (%)	%RSD	Average recovery (%)	%RSD
$^{13}C_4$-PFBA	93.9	1.6	79.8	13.0
$^{13}C_5$-PFPeA	91.4	2.3	80.5	13.4
$^{13}C_5$-PFHxA	89.1	2.7	79.5	14.8
$^{13}C_4$-PFHpA	93.1	1.8	89.2	12.4
$^{13}C_8$-PFOA	92.4	1.1	81.9	14.7
$^{13}C_9$-PFNA	92.3	2.5	77.0	9.3
$^{13}C_6$-PFDA	88.7	0.5	77.8	5.7
$^{13}C_7$-PFUnDA	90.3	1.6	87.1	6.8
^{13}C-PFDoDA	83.0	2.1	87.4	5.3
$^{13}C_2$-PFTreDA	64.6	14.8	95.2	8.2
$^{13}C_3$-PFBS	89.1	11.4	81.3	16.1
$^{13}C_3$-PFHxS	88.8	5.6	108.8	12.7
$^{13}C_8$-PFOS	88.0	2.1	80.3	8.4
$^{13}C_2$-4:2 FTS	89.0	5.7	82.5	19.2
$^{13}C_2$-6:2 FTS	84.9	2.0	129.6	13.7
$^{13}C_2$-8:2 FTS	85.2	0.9	133.3	14.4
$^{13}C_8$-FOSA	93.7	4.3	92.9	4.9
$^{13}C_3$-GenX	89.3	4.5	81.0	12.7
D_5-N-EtFOSAA	88.2	1.0	141.5	4.8
D_3-N-MeFOSAA	88.7	1.3	152.7	7.6
d_3-NMeFOSA	58.5	4.8	70.3	6.7
d_5-NEtFOSA	51.9	2.1	26.7	6.6
d_7-NMeFOSE	57.6	10.6	66.5	5.3
d_9-NEtFOSE	52.2	10.3	63.2	5.0

Table 1. Average recovery of the extracted internal standards (EIS) using the bilayer dual-phase SPE cartridge for soil and fish tissue (n=3).

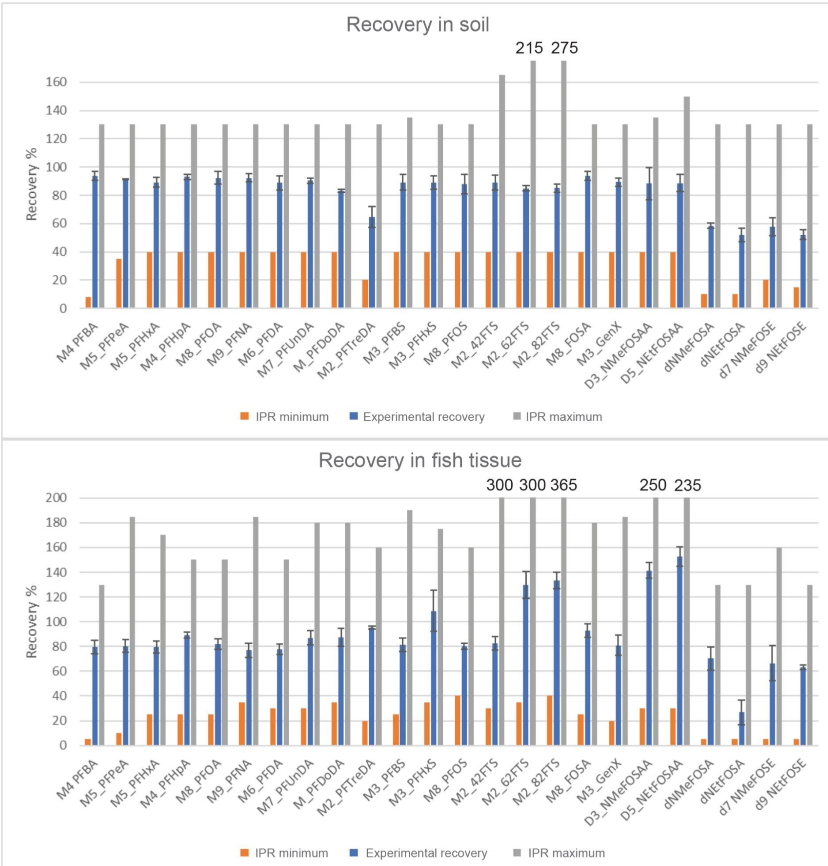

Figure 3. Average recovery of the extracted internal standards (EIS) in soil (top) and fish tissue (bottom). Experimental values (blue) are compared to the minimum (orange) and maximum (gray) percent recoveries allowed in the EPA 1633 method. n=3 replicates for each sample matrix. Bars are labelled that go off the scale of the graph.

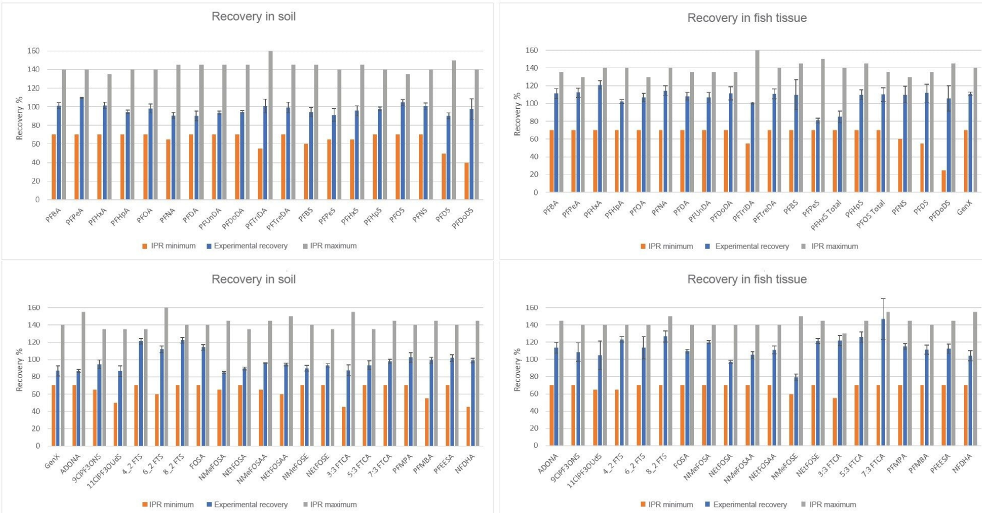

Figure 4. Average recovery of the target PFAS analytes in soil (left) and fish tissue (right). Experimental values (blue) are compared to the minimum (orange) and maximum (gray) percent recoveries allowed in the EPA 1633 method. n=3 replicates for each sample matrix. Bars are labelled that go off the scale of the graph.

Analysis of a Certified Reference Material for Soil

Accuracy of analysis is important for quantitating PFAS in customer samples. A custom certified reference material (CRM) from Waters ERA was processed to demonstrate workflow accuracy. The reference material analyzed contained all 40 EPA 1633 PFAS compounds in a representative soil matrix to evaluate method performance in a known sample free from PFAS. Figure 5 shows the average quantitative results for three replicate extraction and analyses of the soil CRM. The dotted and dashed red lines indicate ±20% of the designated concentration of the CRM (solid blue line) and the solid gray line represents the average experimental quantitated value determined during sample analysis. All 40 target PFAS in EPA 1633 were quantified within ±20% of the concentration range with a mean trueness of 97% and trueness range of 85–120%. This demonstrates confidence in accuracy of the sample preparation, analysis, and data processing workflow using Waters solutions.

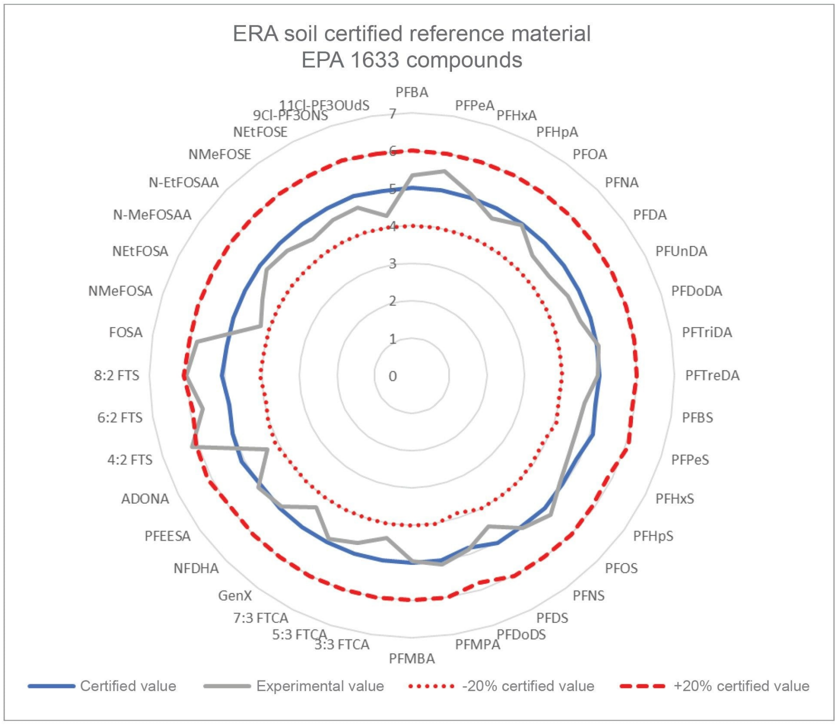

Figure 5. Quantified values of all 40 EPA 1633 target analytes in a custom Waters ERA PFAS in Soil CRM. Red lines represent ±20% of the certified value range of the CRM. The blue line represents the certified value. The solid gray line represents the average experimental quantitated value (n=3).

Conclusion

Sample preparation and analysis was performed for soil and fish tissue samples using EPA 1633 procedures. Oasis GCB/WAX bilayer SPE cartridges containing both WAX and GCB were utilized for the sample extraction and clean up in place of performing the extraction and clean up in two separate steps with dispersive GCB. This cartridge provides a better user experience and reduces time spent in sample preparation. All recoveries were within the acceptance criteria ranges with the mean EIS recovery of 81% and 85% for soil and fish tissue respectively. Mean RSDs were 2.8% and 9.2% for soil and tissue, respectively. This demonstrates the equivalence of the bilayer dual-phase SPE cartridge as a suitable single step replacement for the multi-step extraction and clean up presented in EPA 1633. Additionally, a Waters ERA soil reference material processed and analyzed using the same method was easily within the certified reference value range, giving high confidence in method accuracy. The data presented demonstrates that the Oasis GCB/WAX for PFAS SPE cartridge in combination with the LC-MS/MS system easily fulfills all requirements for analysis of solids and tissues for EPA 1633.

References

1. US Environmental Protection Agency. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. January 2024.
2. US Environmental Protection Agency. Clean Water Act Analytical Methods: CWA Analytical Methods for Per- and Polyfluorinated Alkyl Substances (PFAS). https://www.epa.gov/cwa-methods/cwa-analytical-methods-and-polyfluorinated-alkyl-substances-pfas#documents <https://www.epa.gov/cwa-methods/cwa-analytical-methods-and-polyfluorinated-alkyl-substances-pfas#documents> Accessed 31 Jan, 2024.
3. K Organtini, K Rosnack, P Hancock. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Accordance with EPA 1633 Part 1: Establishing and Assessing the Method. Waters Application Note 720008117. 2023

Appendix

Compound	Parent	Fragment	CV	CE	Soft transmission	Internal standard	Type of internal standard
PFBA	213.0	169	10	10	No	$^{13}C_3$-PFBA	–
PFPeA	262.9	219	10	5	No	$^{13}C_5$-PFPeA	–
PFHxA	312.9	269	5	10	No	$^{13}C_5$-PFHxA	–
		119	5	20			
PFHpA	362.9	319	15	10	No	$^{13}C_4$-PFHpA	–
		169	15	15			
PFOA	412.9	369	10	10	No	$^{13}C_8$-PFOA	–
		169	10	15			
PFNA	462.9	419	10	10	No	$^{13}C_9$-PFNA	–
		219	10	15			
PFDA	512.9	468.9	15	9	No	$^{13}C_6$-PFDA	–
		219	15	15			
PFUnDA	562.9	518.9	25	10	No	$^{13}C_7$-PFUnDA	–
		269	25	20			
PFDoDA	612.9	568.9	30	10	No	^{13}C-PFDoDA	–
		169	30	25			
PFTriDA	662.9	618.9	5	10	No	^{13}C-PFDoDA + $^{13}C_2$-PFTreDA	–
		169	5	30			
PFTreDA	712.9	668.9	10	25	No	$^{13}C_2$-PFTreDA	–
		169	10	15			
PFBS	298.9	80.1	15	30	No	$^{13}C_3$-PFBS	–
		99.1	15	30			
PFPeS	348.9	79.9	10	30	No	$^{13}C_3$-PFHxS	–
		98.9	10	30			
PFHxS	398.9	80.1	10	35	No	$^{13}C_3$-PFHxS	–
		99.1	10	30			
PFHpS	448.9	80.1	15	35	No	$^{13}C_8$-PFOS	–
		99.1	15	35			
PFOS	498.9	80.1	15	40	No	$^{13}C_8$-PFOS	–
		99.1	15	40			
PFNS	548.9	80.1	20	40	No	$^{13}C_8$-PFOS	–
		99.1	20	40			
PFDS	598.9	80.1	46	46	No	$^{13}C_8$-PFOS	–
		99.1	46	46			
PFDoDS	699.1	80	40	55	No	$^{13}C_8$-PFOS	–
		99	40	55			
GenX (HFPO-DA)	285.0	169	5	7	Yes	$^{13}C_3$-HFPO-DA	–
		119	5	35			
ADONA	376.9	251	10	10	No	$^{13}C_3$-HFPO-DA	–
		85	10	25			
9Cl-PF3ONS	530.9	350.9	15	25	No	$^{13}C_3$-HFPO-DA	–
		82.9	15	25			
11Cl-PF3OUdS	630.9	450.9	30	30	No	$^{13}C_3$-HFPO-DA	–
		82.9	30	30			
4:2 FTS	326.9	306.9	15	15	No	$^{13}C_2$-4:2 FTS	–
		80.9	15	35			
6:2 FTS	426.9	407	10	20	No	$^{13}C_2$-6:2 FTS	–
		80.1	12	32			

Compound	Parent	Fragment	CV	CE	Soft transmission	Internal standard	Type of internal standard
8:2 FTS	526.9	506.8	15	25	No	$^{13}C_2$-8:2 FTS	–
		80.9	15	37			
FOSA	497.9	78	40	30	No	$^{13}C_8$-FOSA	–
N-MeFOSA	511.9	168.9	40	30	No	d_3NMeFOSA	–
		218.9	40	25			
N-EtFOSA	525.9	168.9	5	25	No	d_5NEtFOSA	–
		218.9	5	25			
N-MeFOSAA	569.9	418.9	35	25	No	d_3-N-MeFOSAA	–
		219.1	35	20			
N-EtFOSAA	584.0	418.9	15	20	No	d_5-N-EtFOSAA	–
		525.9	15	20			
N-MeFOSE	616.0	59	15	15	No	d_7-NMeFOSE	–
N-EtFOSE	630.0	59	15	15	No	d_9-NEtFOSE	–
3:3 FTCA	241.0	116.9	5	40	No	$^{13}C_5$-PFPeA	–
		176.9	5	10			
5:3 FTCA	340.9	216.9	5	25	No	$^{13}C_5$-PFHxA	–
		237	5	10			
7:3 FTCA	440.9	316.9	10	22	No	$^{13}C_5$-PFHxA	–
		337	10	17			
PFMPA	228.9	84.9	23	10	No	$^{13}C_5$-PFPeA	
PFMBA	278.9	84.9	10	10	No	$^{13}C_5$-PFHxA	
PFEESA	314.9	82.9	15	20	No	$^{13}C_5$-PFHxA	–
		134.9	15	20			
NFDHA	295.0	84.9	5	10	No	$^{13}C_5$-PFHxA	–
		200.9	5	10			
$^{13}C_4$-PFBA	216.8	171.9	10	10	No	$^{13}C_3$-PFBA	Extracted IS
$^{13}C_5$-PFPeA	267.9	223	10	5	No	$^{13}C_2$-PFHxA	Extracted IS
$^{13}C_5$-PFHxA	317.9	272.9	10	5	No	$^{13}C_2$-PFHxA	Extracted IS
		119.9	10	20			
$^{13}C_4$-PFHpA	366.9	321.9	15	10	No	$^{13}C_2$-PFHxA	Extracted IS
		169	15	15			
$^{13}C_8$-PFOA	420.9	375.9	5	15	No	$^{13}C_4$-PFOA	Extracted IS
		172	5	10			
$^{13}C_9$-PFNA	471.9	426.9	10	10	No	$^{13}C_5$-PFNA	Extracted IS
		223	10	15			
$^{13}C_6$-PFDA	519	473.9	5	10	No	$^{13}C_2$-PFDA	Extracted IS
		219	5	15			
$^{13}C_7$-PFUnDA	569.9	524.9	5	10	No	$^{13}C_2$-PFDA	Extracted IS
		274	5	15			
^{13}C-PFDoDA	614.9	569.9	10	10	No	$^{13}C_2$-PFDA	Extracted IS
		169	10	25			
$^{13}C_2$-PFTreDA	714.9	169	25	35	No	$^{13}C_2$-PFDA	Extracted IS
		669.9	25	10			
$^{13}C_3$-PFBS	301.9	80.1	10	30	No	18O2-PFHxS	Extracted IS
		99.1	10	25			
$^{13}C_3$-PFHxS	401.9	80.1	10	40	No	18O2-PFHxS	Extracted IS
		99.1	10	35			
$^{13}C_8$-PFOS	506.9	80.1	15	40	No	$^{13}C_4$-PFOS	Extracted IS
		99.1	15	40			

Compound	Parent	Fragment	CV	CE	Soft transmission	Internal standard	Type of internal standard
$^{13}C_3$-GenX	287	169	5	12	Yes	$^{13}C_2$-PFHxA	Extracted IS
		119	5	12			
$^{13}C_2$-4:2 FTS	328.9	308.9	40	15	No	$^{18}O_2$-PFHxS	Extracted IS
		81	40	25			
$^{13}C_2$-6:2 FTS	428.9	409	10	20	No	$^{18}O_2$-PFHxS	Extracted IS
		80.9	10	27			
$^{13}C_2$-8:2 FTS	528.9	508.9	10	20	No	$^{18}O_2$-PFHxS	Extracted IS
		81	10	35			
$^{13}C_8$-FOSA	505.9	78.1	35	25	No	$^{13}C_4$-PFOS	Extracted IS
d_3NMeFOSA	514.9	168.9	40	30	No	$^{13}C_4$-PFOS	Extracted IS
d_5NEtFOSA	531	168.9	5	25	No	$^{13}C_4$-PFOS	Extracted IS
D_5-N-EtFOSAA	589	418.9	30	20	No	$^{13}C_4$-PFOS	Extracted IS
		506.9	30	15			
D_3-N-MeFOSAA	572.9	418.9	35	20	No	$^{13}C_4$-PFOS	Extracted IS
		482.7	35	15			
d_7-NMeFOSE	623	58.9	15	15	No	$^{13}C_4$-PFOS	Extracted IS
d_9-NEtFOSE	639	58.9	15	15	No	$^{13}C_4$-PFOS	Extracted IS
$^{13}C_3$-PFBA	216	172	10	10	No	–	Non-extracted IS
$^{13}C_2$-PFHxA	314.9	119.9	10	20	No	–	Non-extracted IS
		270	10	5			
$^{13}C_4$-PFOA	417	172	10	20	No	–	Non-extracted IS
$^{13}C_5$-PFNA	468	423	10	10	No	–	Non-extracted IS
$^{13}C_2$-PFDA	515	470	20	10	No	–	Non-extracted IS
$^{18}O_2$-PFHxS	403	83.9	10	40	No	–	Non-extracted IS
$^{13}C_4$-PFOS	503	80.2	15	40	No	–	Non-extracted IS
		99.1	15	40			

Appendix Table 1. MS Method conditions used for PFAS analysis of EPA 1633 compounds in water samples on the Xevo TQ Absolute MS.

Compound	Cal 1 (ng/mL)	Cal 2 (ng/mL)	Cal 3 (ng/mL)	Cal 4 (ng/mL)	Cal 5 (ng/mL)	Cal 6 (ng/mL)	Cal 7 (ng/mL)	Cal 8 (ng/mL)
PFBA	0.02	0.04	0.20	0.40	1.00	2.00	4.0	10.0
PFPeA	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
PFHxA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFHpA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFOA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFNA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFDA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFUnDA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFDoDA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFTriDA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFTreDA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFBS	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFPeS	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFHxS	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFHpS	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFOS	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFNS	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFDS	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
PFDoDS	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
GenX	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
ADONA	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
9ClPF3ONS	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
11ClPF3OUdS	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
4_2 FTS	0.02	0.04	0.20	0.40	1.00	2.00	4.0	10.0
6_2 FTS	0.02	0.04	0.20	0.40	1.00	2.00	4.0	10.0
8_2 FTS	0.02	0.04	0.20	0.40	1.00	2.00	4.0	10.0
FOSA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
NMeFOSA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
NEtFOSA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
NMeFOSAA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
NEtFOSAA	0.005	0.01	0.05	0.10	0.25	0.50	1.0	2.5
NMeFOSE	0.05	0.10	0.50	1.00	2.50	5.00	10.0	25.0
NEtFOSE	0.05	0.10	0.50	1.00	2.50	5.00	10.0	25.0
3:3 FTCA	0.02	0.04	0.20	0.40	1.00	2.00	4.0	10.0
5:3 FTCA	0.10	0.20	1.00	2.00	5.00	10.0	20.0	50.0

Compound	Cal 1 (ng/mL)	Cal 2 (ng/mL)	Cal 3 (ng/mL)	Cal 4 (ng/mL)	Cal 5 (ng/mL)	Cal 6 (ng/mL)	Cal 7 (ng/mL)	Cal 8 (ng/mL)
7:3 FTCA	0.10	0.20	1.00	2.00	5.00	10.0	20.0	50.0
PFMPA	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
PFMBA	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
PFEESA	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
NFDHA	0.01	0.02	0.10	0.20	0.50	1.00	2.0	5.0
M4 PFBA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
M5_PFPeA	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
M5_PFHxA	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M4_PFHpA	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M8_PFOA	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M9_PFNA	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
M6_PFDA	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
M7_PFUnDA	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
M_PFDoDA	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
M2_PFTreDA	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
M3_PFBS	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M3_PFHxS	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M8_PFOS	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M2_42FTS	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
M2_62FTS	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
M2_82FTS	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
M8_FOSA	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
M3_GenX	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
D3_NMeFOSAA	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
D5_NEtFOSAA	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
dNMeFOSA	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
dNEtFOSA	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
d7 NMeFOSE	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d9 NEtFOSE	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
M3 PFBA_NIS	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
M2 PFHxA_NIS	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M4 PFOA_NIS	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M5 PFNA_NIS	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
M2 PFDA_NIS	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
18O2 PFHxS_NIS	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
M4 PFOS_NIS	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50

Appendix Table 2. Calibration curve range used for PFAS analysis of EPA 1633 compounds in water samples on the Xevo TQ Absolute MS.

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