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Note d'application

Simplifying the Analysis of PFAS in Aqueous Samples for EPA Method 1633 by Reducing Sample Volume

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Abstract

US EPA Method 1633 is a multi-lab validated method for the analysis of 40 per- and polyfluoroalkyl substances (PFAS) in a variety of environmental matrices. The method was validated using 500 mL aqueous sample volumes leading to a lengthy sample preparation process. Sample preparation time was reduced by half, to approximately one hour per batch, using a 50 mL sample volume. Although a significantly smaller sample volume was used, high sensitivity was still achieved and all the recovery requirements for EPA 1633 were met. A variety of complex environmental water samples were prepared using this reduced sample volume and analysis was performed using the Waters™ PFAS workflow for LC-MS/MS.

Benefits

- Reduced sample volume extraction of environmental water samples while maintaining performance criteria of EPA 1633 and the ability to detect trace levels of PFAS in samples, allowing flexibility for the lab
- Performance of the reduced volume workflow is demonstrated by easily passing qualifications of a Waters

ERA™ certified reference material, demonstrating equivalent results to other standard sample volumes

- Sample preparation time reduced in half from 250 mL automated time of two hours to about an hour per batch, thereby minimizing overall time to process samples
- Field collection of lower sample volumes benefits overall sample transportation and storage costs
- Easier sample preparation using solid phase extraction (SPE) automation and lower chance of SPE cartridge clogging when loading smaller volumes of challenging aqueous matrices

Introduction

US EPA Method 1633A is a multi-lab validated method for the analysis of non-potable water matrices, soils, biosolids, and tissues.[1] The method covers 40 PFAS and, for aqueous matrices, utilizes a sample preparation incorporating SPE on a weak anion exchange (WAX) cartridge followed by graphitized carbon black (GCB) clean-up. The method was multi-laboratory validated using 500 mL sample sizes, which leads to a lengthy sample preparation process that can be difficult to complete with challenging sample types. In previous work, a 250 mL aqueous sample size was introduced,[2] followed by the full automation of the SPE.[3] By utilizing the fully automated SPE system and 250 mL sample sizes, sample preparation time was reduced to approximately 2 hours for a batch of 8 samples. In both studies, the Xevo™ TQ Absolute Mass Spectrometer was used for the analysis of samples after sample preparation and was able to reach limits of quantification (LOQs) 10x lower than the LOQs reported in the EPA 1633A method. This allows for further reduction in sample volume, offering additional advantages to routine laboratories. These benefits include reduced costs with collection and transportation of field samples, along with less burden some sample storage requirements. Additionally, a smaller sample size results in faster SPE sample preparation, allowing for higher sample throughput and faster turnaround time for customers. Finally, a reduction in sample volume also enables easier sample handling and fewer problems with SPE cartridge clogging when working with challenging samples. The data presented also indicates that a smaller sample size can increase overall recovery and, therefore, higher sensitivity is possible for previously problematic PFAS in the more challenging aqueous sample types. The entire sample preparation of 50 mL aqueous samples was completed using a fully automated SPE workflow in approximately one hour and was then analyzed using an 11-minute method on an ACQUITY™ Premier System with Binary Solvent Management and Flow Through Needle coupled with a Xevo TQ Absolute Mass Spectrometer.

Experimental

Sample Preparation

Samples discussed in this application note include surface water that was collected locally, as well as influent and effluent wastewater that were kindly provided by a municipal wastewater treatment facility in the Midwest United States. Influent wastewater samples were sampled only after the primary settling phase, prior to any further treatment. Effluent wastewater samples were fully treated samples ready for discharge. These samples are from the same sources studied in a previous application note that performed EPA 1633 extractions using manual and automated techniques with 250 mL of sample.[2] All water samples were collected using grab sampling directly into 50 mL polypropylene centrifuge tubes and were not sub-sampled. Samples were frozen until sample analysis according to EPA 1633 guidelines and holding times. Sample bottles were weighed prior to sample preparation (full) and after sample preparation (empty) to determine the exact volume collected in each bottle. In addition to authentic samples, the Waters ERA PFAS in Wastewater (Item No. 404 < https://www.eraqc.com/pfas-in-wastewater-wp-era001663?returnurl=%2fpfas-products%2f>) certified reference material (CRM) was processed with the samples. The preparation instructions for the CRM were reduced 10x to create a 50 mL sample instead of the recommended 500 mL sample.

The Oasis™ WAX/GCB Cartridge, a bilayer dual-phase SPE cartridge containing both WAX and GCB sorbents was used for sample preparation of all samples. The addition of GCB into the SPE cartridge allows for the full sample extraction and sample clean-up required by EPA 1633 to be automated, rather than having to perform the GCB clean-up step using a dispersive technique.

Sample extraction was performed using the PromoChrom (Richmond, BC, Canada) SPE-03 Gen 4 Automated SPE System. The method used to control the automated SPE system is detailed in Table 1 and was directly adapted from EPA 1633. A sample volume of 50 mL was extracted. High-capacity inline filters and anti-clogging tips for the PromoChrom MOD-004 caps were used on the sample inlet lines to filter out particulates before introduction to the SPE system. For the wastewater samples, which contained a substantial amount of suspended solids and particulates, glass wool was also packed to half-height of the SPE cartridge to prevent cartridge clogging.

All samples were spiked with 25 ng/L (sample concentration equivalent) of the required extracted internal standard (EIS) prior to extraction and 25 ng/L (sample concentration equivalent) of the required non-extracted internal standard (NIS) after extraction. The calibration curve range for each analyte is listed in Appendix Table 2.

All standards were obtained as mixes from Wellington Laboratories (Guelph, Ontario, Canada).

Action	Inlet 1	Inlet 2 (ratio)	Flow	Volume
Elute W2	Solvent 5	–	8	15
Elute W1	Solvent 3	–	8	5
Add sample W1	Sample	–	5	275
Rinse	Solvent 2	Air (20%)	45	2.5
Add sample W1	Sample	–	5	5
Rinse	Solvent 2	Air (20%)	45	5
Add sample W1	Sample	–	5	5
Rinse	Solvent 2	Air (20%)	45	5
Add sample W1	Sample	–	5	5
Shake	–	Time based	–	20 s
Rinse	Solvent 4	Air (20%)	45	1.3
Add sample W1	Sample	–	5	3
Rinse	Solvent 4	Air (20%)	45	5
Add sample W2	Sample	–	5	5
Shake	–	Time based	–	20 s
Air Purge W2	Air	–	5	3
Add sample W2	Sample	–	5	5
Blow N2	-	Time based	–	3 min
Rinse	Solvent 5	Air (20%)	45	1.3
Collect 1	Sample	–	1	3
Rinse	Solvent 5	Air (20%)	45	5
Air purge R	Air	–	20	5
Shake	–	Time based	–	15 s
Clean	Sample	–	30	5
Clean	Sample	–	30	5
Collect 1	Sample	–	1	15

Table 1. PromoChrom SPE-03 method conditions for EPA 1633 aqueous sample preparation of 50 mL samples. Solvent 2: water, Solvent 3: 0.3 M formic acid, Solvent 4: 1:1 0.1 M formic acid:methanol, Solvent 5: 1% ammonium hydroxide in methanol.

Method Conditions

LC Conditions

LC system:	ACQUITY Premier System with Binary Solvent Management and Flow Through Needle
Vials:	700 µL Polypropylene Screw Cap Vials (p/n: 186005219)
Analytical column:	ACQUITY Premier BEH™ C_{18} Column 2.1 x 50 mm, 1.7 µm (p/n: 186009452)
Isolator column:	Atlantis™ Premier BEH C_{18} AX Column 2.1 x 50 mm, 5.0 µm (p/n: 186009407)
Column temperature:	35 °C
Sample temperature:	10 °C
PFAS kit:	PFAS Install Kit (p/n: 176004548)
Injection volume:	10 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 Table 1 for full MRM method details

Data Management

Software:	waters_connect™ for Quantitation Software

Results and Discussion

Sensitivity and Method Detection Limits

The sample size was the only part of the sample preparation process that was changed from the automated workflow that was previously being used.[3] Equivalency to the EPA 1633 quality control guidelines has been demonstrated using a 250 mL sample. In this work, the sample volume was reduced to just 50 mL of sample, resulting in a 5x reduction of enrichment factor from the SPE extraction. To adjust for the difference in enrichment factor between the two sample sizes, the injection volume was increased from 2 µL to 10 µL for the analysis of the 50 mL samples. Figure 1 demonstrates that this adjustment produced response equivalent to that of the the 250 mL extract, while reducing sample preparation time to about an hour per batch of 8 samples, or approximately half the time required for 250 mL samples.

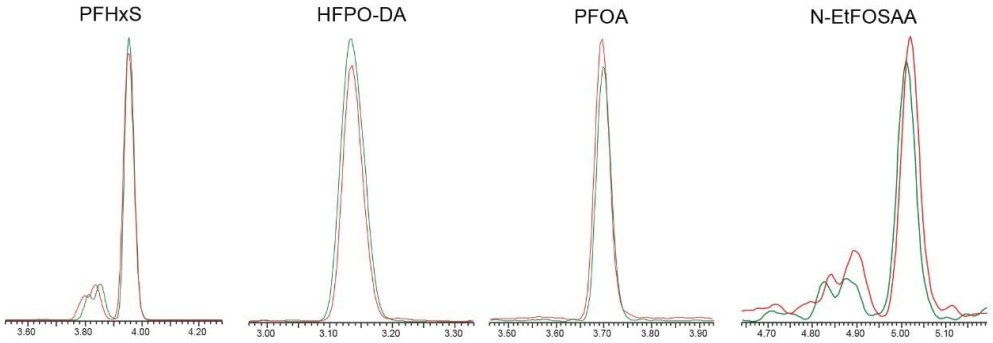

Figure 1. Chromatogram overlay of four example PFAS extracted using both 250 mL (green) and 50 mL (orange) sample sizes.

A method detection limit study was performed following the guidance of the Code of Federal Regulations Guidelines Establishing Test Procedures for the Analysis of Pollutants (40 CFR Part 136, Appendix B).[4] For this study, 8 replicate 50 mL water samples spiked near the expected detection limit were extracted and analyzed

over a 3-day period. MDLs were calculated based on the guidance document procedure, including calculating an MDL blank (MDL_b) for PFBA, PFPeA, PFOA, and PFNA, which were detected in the method blank samples.

Using these calculations, the MDLs for all 40 PFAS compounds were determined and are listed in Table 2. A comparison is also made to the pooled MDLs provided in EPA 1633A for aqueous samples which were derived from the method's multi-laboratory validation study using a 500 mL sample extract. Except for the four PFAS that required an MDL_b to be calculated due to solvent contamination, all compounds had a lower MDL using the 50 mL extraction technique followed by analysis on the Xevo TQ Absolute Mass Spectrometer than those reported in EPA 1633A, meaning a lower concentration is detectable. When compared, on average, the MDLs were approximately 3 times lower with a range of 1.5 – 6.2 times lower MDLs depending on compound.

Compound	50 mL sample MDL ng/L	EPA 1633 MDL ng/L
PFBA*	1	0.79
PFPeA*	0.53	0.54
PFHxA	0.14	0.46
PFHpA	0.24	0.37
PFOA*	0.41	0.54
PFNA*	0.85	0.45
PFDA	0.13	0.52
PFUnDA	0.23	0.45
PFDoDA	0.15	0.4
PFTriDA	0.28	0.46
PFTreDA	0.18	0.49
PFBS	0.09	0.37
PFPeS	0.18	0.5
PFHxS	0.16	0.54
PFHpS	0.15	0.5
PFOS	0.11	0.63
PFNS	0.18	0.47
PFDS	0.23	0.6
PFDoDS	0.13	0.6
HFPO-DA	0.43	0.51

Compound	50 mL sample MDL ng/L	EPA 1633 MDL ng/L
ADONA	0.34	0.5
NFDHA	0.42	0.75
9ClPF3ONS	0.33	1.38
11ClPF3OUdS	0.28	1.67
4:2 FTS	0.87	1.69
6:2 FTS	0.43	2.45
8:2 FTS	0.8	2.5
PFOSA	0.17	0.32
NMeFOSA	0.25	0.43
NEtFOSA	0.18	0.45
NMeFOSAA	0.16	0.68
NEtFOSAA	0.18	0.59
NMeFOSE	1.79	3.81
NEtFOSE	1.24	4.84
3:3 FTCA	0.86	2.47
5:3 FTCA	1.63	9.59
7:3 FTCA	2.33	8.71
PFMPA	0.26	1.46
PFMBA	0.19	1.41
PFEESA	0.19	1.17

*Table 2. Calculated method detection limits (MDLs) for the 50 mL extracted water samples (n=8) compared to the pooled MDLs provided in EPA 1633A. * indicates the MDL is calculated using the method blank technique.*

Recovery in Water Samples

The average percent recovery of the EIS extracted from surface water, influent wastewater, and effluent wastewater samples (all containing suspended solids) are shown in Figure 2A-C, comparing 250 mL and 50 mL extraction volumes for each. The minimum recovery limits required by EPA 1633 are indicated by the black lines in Figure 2.[1] Generally, both extraction volumes produced comparable recoveries and were well above the required minimum recovery values. In the wastewater samples, the recovery using the 50 mL sample volume was greater for some of the longer chain carboxylates (PFDoDA and PFTreDA) and sulfonamides (FOSAs, FOSAAs, and FOSEs). These groups of PFAS either exclusively use (FOSAs) or more heavily rely on long chain carboxylates, the reversed phase mechanism of the WAX sorbent. With less organic material from the matrix being loaded onto the SPE cartridge with the smaller sample volume, this may be demonstrating there is less competition for reversed phase sites, resulting in the increased recovery of these types of PFAS. The mean recovery of all EIS from the 50 mL samples across the different aqueous sample types was 77% with a mean RSD of 7.7%. In comparison, the mean recovery and %RSD in the 250 mL samples were nearly identical, with values of 76% and 8.8%, respectively.

Additionally, the targeted native PFAS analytes were spiked into the surface water sample to determine the native recoveries. Figure 2D shows the recovery of three replicates of this spiked sample, with the minimum and maximum recovery guidelines represented by the black bars. The recovery ranged from 88 – 123%, with a mean recovery of 99% and mean RSD of 7.1%. Similar to the EIS, the native recoveries were all within the required range indicating that the targeted PFAS can be reliably recovered from the smaller sample volume.

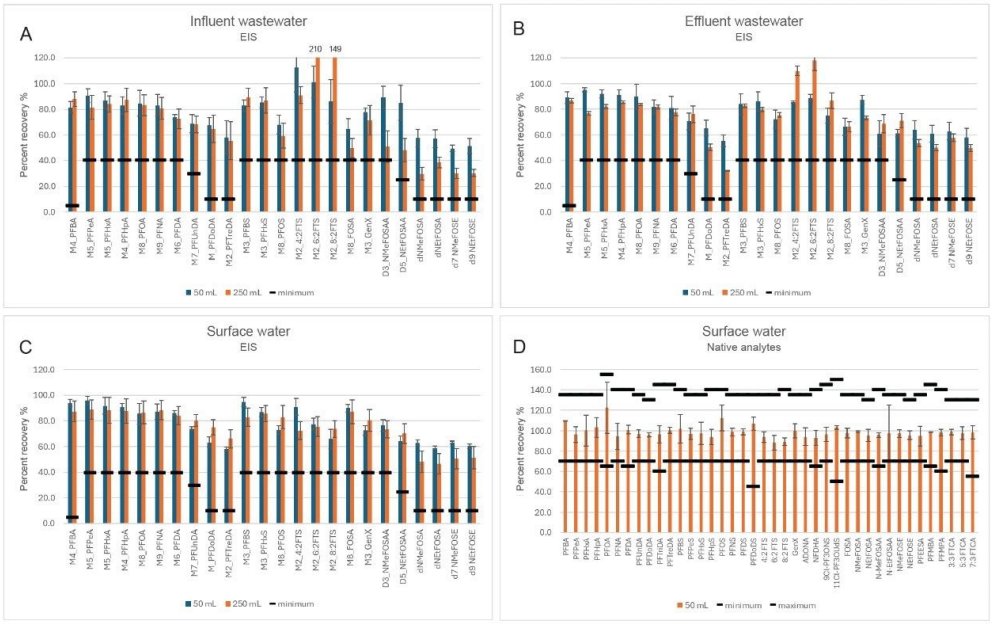

Figure 2. Extracted internal standard (EIS) recovery for influent wastewater (A), effluent wastewater (B), and surface water (C) comparing results from both 50 mL and 250 mL sample sizes. Recovery of native analytes spiked into surface water 50 mL sample (D). n=3 replicates for all samples.

Analysis of a Certified Reference Material and Authentic Water Samples

To assess accuracy of utilizing a smaller sample size, a CRM from Waters ERA was processed with the authentic samples. The PFAS in the Wastewater CRM is certified for all EPA 1633 analytes, giving a representative reference material for method performance without having to spike unknown matrix samples, which can become complicated without a sample free from PFAS. The CRM was prepared in both 250 and 50 mL volumes to compare the smaller sample size with a more standard one.

Figure 3 shows the average quantitative results for 3 replicate extractions and analyses of the Wastewater CRM at both sample sizes, compared to the certified values and minimum/maximum certified value range. All 40 target PFAS in EPA 1633 were quantified within the allowable minimum and maximum concentration range for both sample sizes. The mean trueness for the 50 mL CRM extraction was 104%, compared to 103% for the 250 mL CRM extraction. These results demonstrate that the automated workflow in combination with the sample analysis is highly accurate and repeatable, regardless of sample size.

Different types of authentic environmental water samples were extracted using the 50 mL sample size to quantify the levels of PFAS in these authentic samples. PFAS were detected in all the samples tested at different concentration levels with the details of each sample listed in Table 3, including the total PFAS concentration in each sample type. These results demonstrate that even with the smaller sample volumes, trace levels of PFAS can be detected in different types of challenging water samples.

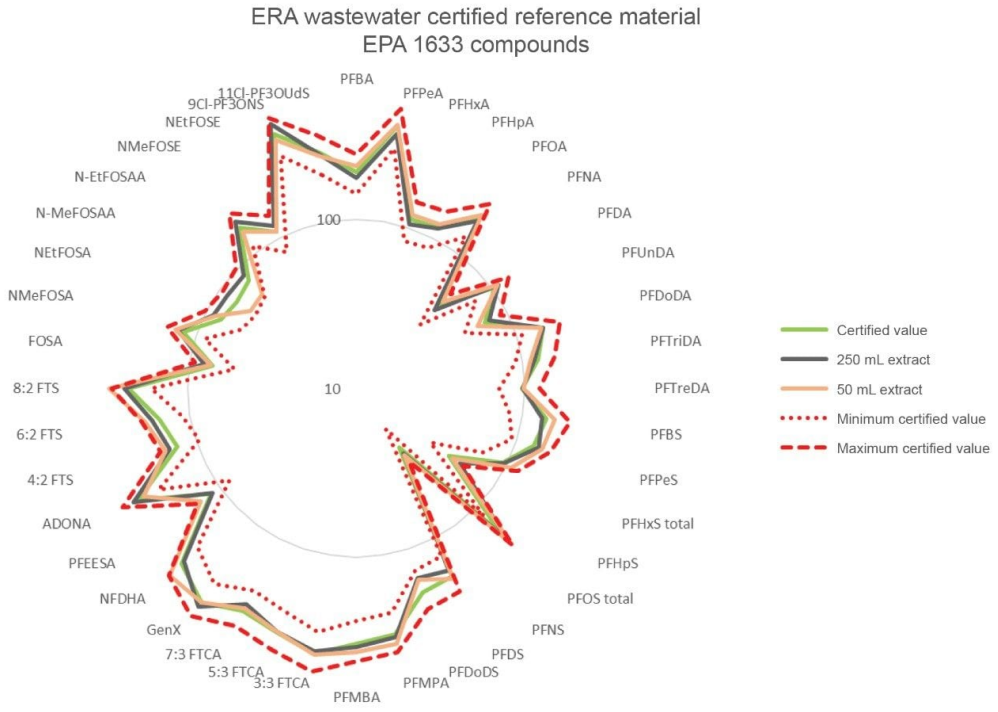

Figure 3. Quantitative results for the extraction and analysis of a wastewater CRM comparing the results for 250 mL (black) and 50 mL (orange) sample sizes with the CRM values (green) and the minimum and maximum certification range (dotted and dashed red).

Compound	Surface water (ng/L)	Influent wastewater (ng/L)	Effluent wastewater (ng/L)
PFBA	11.4	18.1	17.6
PFPeA	2.0	12.4	19.1
PFHxA	1.7	20.7	57.3
PFHpA	2.0	4.6	4.6
PFOA	8.8	11.3	14.3
PFNA	2.1	1.8	0.9
PFDA	1.9	N.D.	0.2
PFBS	3.4	41.7	43.9
PFHxS Total	N.D.	7.7	7.2
PFOS Total	7.3	15.8	7.5
6_2 FTS	N.D.	5.7	5.7
5:3 FTCA	N.D.	47.8	N.D.
7:3 FTCA	N.D.	5.7	N.D.
Total PFAS	40.6	193.3	178.3

Table 3. Concentrations of PFAS detected in each water sample reported in ng/L.

Conclusion

The sample size for aqueous samples was successfully reduced to only 50 mL while maintaining the sensitivity and overall method performance as observed when using 250 mL sample sizes. This was demonstrated using a method detection limit study, recovery of both extracted internal standards and native analytes, and a certified

reference material. Extracted internal standard recoveries in three different water sample types were well above the required minimum recovery values. Native PFAS analyte recoveries were also well within the required recovery range in a surface water sample. Additionally, calculated concentration values for a wastewater reference material were determined to be accurate when compared to the provided certified range, reinforcing confidence in method accuracy. Using the reduced sample size, authentic water samples were collected, extracted, and analyzed with PFAS detected and quantified in each sample. The benefits of using a smaller sample size are realized from sample collection and transportation in the field, laboratory storage, reduced sample preparation time, and in some cases, better recovery and overall performance.

References

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3. K Organtini, K Rosnack, O Burt, I Wan. Automating the Sample Preparation Workflow for Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples Following EPA Method 1633. Waters Application Note 720008825 <https://www.waters.com/nextgen/global/library/application-notes/2025/automating-the-sample-preparation-workflow-for-per-and-polyfluoroalkyl-substances-pfas-in-aqueous-samples-following-epa-method-1633.html> . 2025.

4. Code of Federal Regulations. Guidelines Establishing Test Procedures for the Analysis of Pollutants. *40 CFR Part 136, Appendix B.*

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	$^{13}C_6$-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	$^{13}C_8$-PFOS	–
		82.9	15	25			
11Cl-PF3OUdS	630.9	450.9	30	30	No	$^{13}C_8$-PFOS	–
		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			
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	d7-NMeFOSE	–
N-EtFOSE	630.0	59	15	15	No	d9-NEtFOSE	–
3:3 FTCA	241.0	116.9	5	40	No	$^{13}C_5$-PFPeA	–
		176.9	5	10			

Compound	Parent	Fragment	CV	CE	Soft transmission	Internal standard	Type of internal standard
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
$^{13}C_6$-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}C4-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	$^{18}O_2$-PFHxS	Extracted IS
		99.1	10	25			
$^{13}C_3$-PFHxS	401.9	80.1	10	40	No	$^{18}O_2$-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			
$^{13}C_3$-GenX	287	169	5	12	Yes	$^{13}C_3$-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 Mass Spectrometer.

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
7:3 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)
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 Mass Spectrometer.

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ACQUITY Premier System <https://www.waters.com/nextgen/global/products/chromatography/chromatography-systems/acquity-premier-system.html>

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