Simplified SPE for PFAS Analysis of Non-Potable Waters

Faster, More Reliable Sample Preparation for EPA Method 1633 PFAS Analysis

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Abstract

PFAS analysis in nondrinking water matrices presents unique challenges to sample preparation. Trace-level analysis of PFAS in surface water and wastewater requires both low background contamination and the ability to handle challenging matrices. Resprep PFAS cartridges allow fast, reliable SPE sample preparation of non-potable waters, such as those covered in EPA Method 1633 for PFAS analysis. The inclusion of both WAX and carbon layers in a single cartridge allows for simultaneous extraction and cleanup. In addition, an optional filter aid prevents the cartridges from clogging when preparing samples that contain high amounts of suspended solids.

Introduction

While methods for PFAS analysis in drinking water have been widely used for some time, the absence of methods for non-potable waters using solid-phase extraction (SPE) has led many labs to modify drinking water methods when analyzing surface water and wastewater. To address this, EPA Method 1633 [1], which was finalized in January of 2024, provides a sample preparation approach that includes an SPE extraction using a weak anion exchange (WAX) cartridge manually packed with glass wool and a dispersive carbon cleanup. The manual packing of glass wool and separate carbon cleanup step add extra time, complexity, and risk of introducing contaminants to the sample preparation process, and manually packed wool can increase variation among technicians. Labs interested in streamlining their sample preparation workflows can utilize Resprep PFAS cartridges, which are dual-bed SPE cartridges that contain both WAX and carbon sorbent beds that allow the SPE extraction and carbon cleanup to be accomplished in a single cartridge. An optional filter aid eliminates the need for manual addition of glass wool to handle samples containing solids, and it provides much better performance in preventing clogs.

Related Products

- *Force C18 column 1.8 µm, 50 mm x 2.1 mm (cat.# 9634252)*
- *PFAS delay column (cat.# 27854)*
- *Resprep PFAS cartridge (cat.# 28930)*
- *Resprep PFAS cartridge with filter aid (cat.# 28931)*
- *Resprep QR-12 vacuum manifold (cat.# 28298-VM)*
- *Quick-replace liners (cat.# 28310-VM)*
- *Resprep sample delivery system (cat.# 26250)*

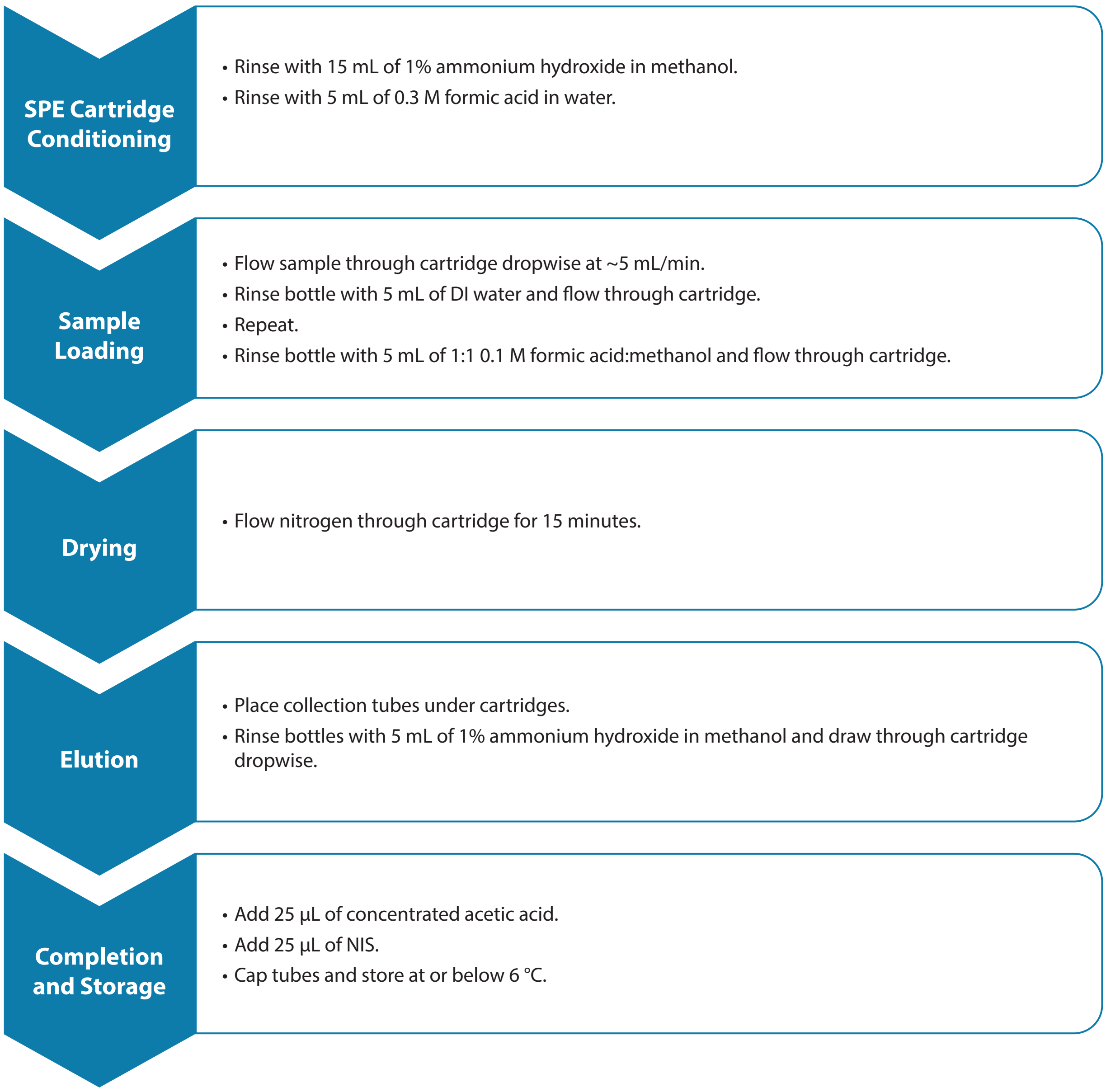
Experimental

Sample Preparation

Samples for precision, accuracy, and method detection limit (MDL) studies were prepared in polypropylene bottles using 500 mL deionized water spiked with 25 µL of extracted internal standards (EIS, Wellington Laboratories p/n MPFAC-HIF-ES) as per EPA Method 1633, Section 11.2.4. Four samples for precision and recovery analysis were spiked with 200 µL of native PFAS standards (Wellington Laboratories p/n EPA-1633STK), giving pre-extraction concentrations of 100-2500 ng/L. Seven MDL samples were spiked with 20 µL of a 20:1 dilution of the native standard, giving pre-extraction concentrations of 0.5-12.5 ng/L. Seven blank replicates were prepared for the MDL study as well. The MDL samples were prepared and analyzed over three days.

Resprep PFAS SPE cartridges (6 mL), which contained 150 mg of 30 µm WAX and 50 mg of CarboPrep Plus carbon (cat.# 28930), were placed on a Thermo AutoTrace PFAS system. After preparation of the samples and setup of the SPE system, the samples were extracted following the instructions in EPA Method 1633, Section 11.4, which is summarized in Figure 1. After extraction, the extracts were spiked with 25 µL of non-extracted internal standards (NIS, Wellington Laboratories p/n MPFAC-HIF-IS).

Figure 1: Sample Preparation Procedure for EPA Method 1633 PFAS Analysis



SPE Cartridge Conditioning

- Rinse with 15 mL of 1% ammonium hydroxide in methanol.
- Rinse with 5 mL of 0.3 M formic acid in water.

Sample Loading

- Flow sample through cartridge dropwise at ~5 mL/min.
- Rinse bottle with 5 mL of DI water and flow through cartridge.
- Repeat.
- Rinse bottle with 5 mL of 1:1 0.1 M formic acid:methanol and flow through cartridge.

Drying

- Flow nitrogen through cartridge for 15 minutes.

Elution

- Place collection tubes under cartridges.
- Rinse bottles with 5 mL of 1% ammonium hydroxide in methanol and draw through cartridge dropwise.

Completion and Storage

- Add 25 µL of concentrated acetic acid.
- Add 25 µL of NIS.
- Cap tubes and store at or below 6 °C.

Samples to test filter aid efficiency were prepared using ASTM substitute wastewater [2] diluted by 2.5x to be ~100 mg/L suspended solids. Resprep PFAS SPE cartridges (cat.# 28931), which contained 2000 mg of filter aid, 150 mg of 30 µm WAX, and 50 mg of CarboPrep Plus carbon, were placed on a Resprep QR-12 vacuum manifold (cat.# 28298-VM) that was fitted with quick-replace liners (cat.# 28310-VM). Resprep sample delivery system lines (cat.# 26250) were used to transfer the samples to the SPE cartridges. Five hundred milliliters of the substitute wastewater was loaded onto the cartridge. The volume of sample that passed through without clogging was compared to cartridges containing only WAX (cat.# 28292) and WAX cartridges manually packed with glass wool (cat.# 24324) to half the height of the SPE cartridge as described in Method 1633, Section 12.1.1.

Analytical System

After extraction, the samples were analyzed by LC-MS/MS under the conditions shown below. The use of a PFAS delay column is important to prevent any PFAS contamination upstream of the injector from coeluting with the samples. Thorough blank checking of the analytical system was performed and showed no detectable PFAS contamination.

Instrument Conditions for EPA Method 1633 PFAS Analysis

System: Waters ACQUITY Premier LC/Xevo TQ Absolute Triple Quadrupole MS
Columns:
- PFAS delay column (cat.# 27854)
- Analytical column: Force C18, 1.8 µm x 50 mm x 2.1 mm (cat.# 9634252)

Injection volume: 3 µL
Mobile phase A: Water, 5 mM ammonium acetate
Mobile phase B: Methanol
Flow rate: 0.4 mL/min
Temperature: 40 °C
Gradient:

Time (min)	%B
0.00	20
6	95
6.6	95
6.61	20
7.5	20

Ion source: Electrospray
Ion mode: ESI-
Mode: MRM

The analytical system was calibrated using the concentrations shown in Table 4 of Method 1633 for PFAS analysis. Curve fits were chosen that minimized % relative standard error (%RSE).

Method Detection Limits (MDL)

The method detection limit was calculated from the analysis of seven blank replicates and seven low-level spikes, as outlined in EPA's Definition and Procedure for the Determination of the Method Detection Limit, Revision 2 [3]. The standard deviations of the spike and blank results were multiplied by the Student's *t*-value of 3.143 to determine the MDL, and the higher of the results between the spikes and blanks was selected as the MDL.

Accuracy and Precision

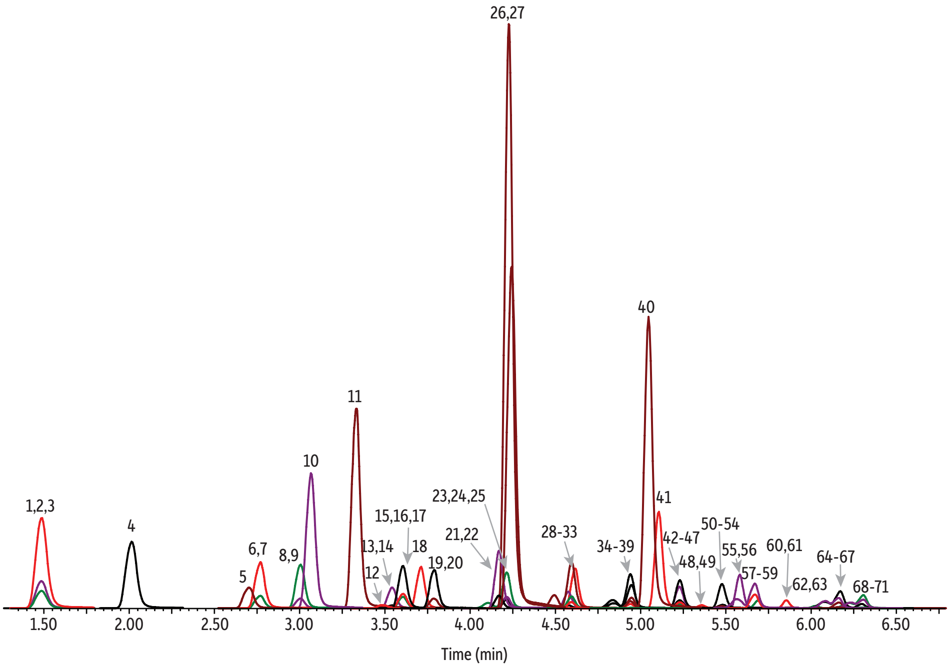
Accuracy and precision were determined by analyzing four replicate spikes. The accuracy of the spikes was calculated and compared to the recovery limits in Table 5 of EPA Method 1633. The relative standard deviations of the spike results were also determined and compared to the limits in Table 5. The recovery of the isotope dilution standards was calculated from the spike replicates and compared to the recovery limits from Table 6 of EPA Method 1633 for PFAS analysis.

Results & Discussion

Method Performance Verification Experiment

Good chromatographic results were obtained for all compounds, as shown in Figure 2. The MDL, blank levels, accuracy, and precision results for native PFAS analytes are shown in Table I. The calculated MDLs were below the reporting limits shown in Table 9 in EPA Method 1633, and the accuracy of the spikes ranged from 77 to 125% of the spiked value, meeting the requirements in Table 5 of Method 1633. The spikes showed good precision as well, with the results being ≤25% RSD. Similarly, the recoveries for the isotope dilution standards were also within the limits listed in Table 6 of the method. The results are shown in Table II.

Figure 2: Recovery Standard for EPA Method 1633 PFAS Analysis



LC_EV0599

Column	Force C18 (cat.# 9634252)
Dimensions:	50 mm x 2.1 mm ID
Particle Size:	1.8 µm
Pore Size:	100 Å
Temp.:	40 °C
Standard/Sample	
Inj. Vol.:	3 µL
Mobile Phase	
A:	Water, 5 mM ammonium acetate
B:	Methanol

Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	80	20
6	0.4	5	95
6.6	0.4	5	95
6.61	0.4	80	20
7.5	0.4	80	20

Max Pressure:	~400 bar
Detector	Waters Xevo TQ-S
Ion Mode:	ESI-
Mode:	MRM
Instrument	Waters ACQUITY Premier
Sample Preparation	Resprep PFAS cartridges (cat.# 28930) and a Thermo AutoTrace PFAS instrument were used for the following sample preparation procedure. 1. Spike 500 mL DI water with 200 µL of Wellington EPA-1633STK and 25 µL of Wellington MPFAC-HIF-ES. 2. Rinse cartridge with 15 mL of 1% ammonium hydroxide in methanol. 3. Rinse with 5 mL of 0.3 M formic acid in water. 4. Load sample onto SPE cartridge dropwise at 5 mL/min. 5. Rinse bottle with 5 mL DI water, load on to SPE cartridge dropwise at 5 mL/min, repeat. 6. Rinse bottle with 5 mL of 1:1 0.1 M formic acid:methanol and load onto SPE cartridge dropwise at 5 mL/min. 7. Flow nitrogen through cartridge for 15 minutes. 8. Rinse bottles with 5 mL of 1% ammonium hydroxide in methanol, elute into collection vessel. 9. Add 25 µL of concentrated acetic acid and 25 µL of Wellington MPFAC-HIF-IS. 10. Transfer aliquot to autosampler vial (cat. # 23243) and cap (cat. # 23244).
Notes	A PFAS delay column (cat.# 27854) was installed before the injector.

Figure 2 continued on next page.

Figure 2 *(continued)*

Peaks	tR (min)	Conc. (ng/L)	Precursor 1	Product 1	Product 2	Precursor 2	Product 1
1. Perfluoro-*n*-[1,2,3,4- 13C3]butanoic acid (13C3-PFBA)	1.485	5	216	172	-	-	-
2. Perfluoro-*n*-[1,2,3,4- 13C4]butanoic acid (13C4-PFBA)	1.487	10	217	172	-	-	-
3. Perfluorobutanoic acid (PFBA)	1.489	10	213	169	-	-	-
4. Perfluoro-3-methoxypropanoic acid (PFMPA)	2.007	5	229	85	-	-	-
5. 3-Perfluoropropyl propanoic acid (3:3FTCA)	2.7	12.5	241	177	117	-	-
6. Perfluoro-*n*-[1,2,3,4,5- 13C5]pentanoic acid (13C5-PFPeA)	2.765	5	268	223	-	-	-
7. Perfluoropentanoic acid (PFPeA)	2.768	5	263	219	69	-	-
8. Perfluorobutane sulfonate (PFBS)	3.006	2.5	299	80	99	-	-
9. Sodium perfluoro-1-[2,3,4- 13C3]butanesulfonate (13C3-PFBS)	3.007	2.5	302	80	99	-	-
10. Perfluoro-4-methoxybutanoic acid (PFMBA)	3.071	5	279	85	-	-	-
11. Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	3.333	5	315	135	83	-	-
12. Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	3.493	5	295	201	85	-	-
13. 1*H*, 1*H*,2*H*,2*H*-perfluorohexane sulfonate (4:2 FTS)	3.545	10	327	307	81	-	-
14. Sodium 1*H*,1*H*,2*H*,2*H*-perfluoro-1-[1,2- 13C2]hexane sulfonate (13C2-4:2FTS)	3.55	5	329	81	309	-	-
15. Perfluorohexanoic acid (PFHxA)	3.608	2.5	313	269	119	-	-
16. Perfluoro-*n*-[1,2,3,4,6- 13C2]hexanoic acid (13C2-PFHxA)	3.609	2.5	315	270	119	-	-
17. Perfluoro-*n*-[1,2,3,4,6- 13C5]hexanoic acid (13C5-PFHxA)	3.61	2.5	318	273	120	-	-
18. Perfluoropentane sulfonate (PFPeS)	3.718	2.5	349	80	99	-	-
19. 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy13C3-propanoic acid (13C3-HFPO-DA)	3.79	10	287	169	185	-	-
20. Hexafluoropropylene oxide (HFPO-DA)	3.795	10	285	169	185	-	-
21. Perfluoroheptanoic acid (PFHpA)	4.174	2.5	363	319	169	-	-
22. Perfluoro-*n*-[1,2,3,4- 13C4]heptanoic acid (13C4-PFHpA)	4.174	2.5	367	322	-	-	-
23. Perfluoro-1-hexane[18O2]sulfonic acid (18O2-PFHxS)	4.218	2.5	403	84	-	-	-
24. Sodium perfluoro-1-[1,2,3- 13C3]hexanesulfonate (13C3-PFHxS)	4.218	2.5	402	80	99	-	-
25. Perfluorohexane sulfonate (PFHxS)	4.22	2.5	399	80	99	-	-
26. 4,8-Dioxa-3*H*-perfluorononanoic acid (ADONA)	4.232	10	377	251	85	-	-
27. 2*H*,2*H*,3*H*,3*H*-Perfluorooctanoic acid (5:3FTCA)	4.247	62.5	341	237	217	-	-
28. 1*H*,1*H*,2*H*,2*H*-perfluorooctane sulfonate (6:2 FTS)	4.58	10	427	407	81	-	-
29. Sodium 1*H*,1*H*,2*H*,2*H*-perfluoro-1-[1,2-13C2]-octane sulfonate (13C2-6:2FTS)	4.581	5	429	81	409	-	-
30. Perfluoro-*n*-[13C8]octanoic acid (13C8-PFOA)	4.598	2.5	421	376	-	-	-
31. Perfluorooctanoic acid (PFOA)	4.601	2.5	413	369	169	-	-
32. Perfluoro-*n*-[13C4]octanoic acid (13C4-PFOA)	4.601	2.5	417	172	-	-	-
33. Perfluoroheptane sulfonate (PFHpS)	4.621	2.5	449	80	99	-	-
34. Perfluorononanoic acid (PFNA)	4.944	2.5	463	419	219	-	-
35. Perfluoro-*n*-[13C5]nonanoic acid (13C5-PFNA)	4.944	1.25	468	423	-	-	-
36. Perfluoro-*n*-[13C9]nonanoic acid (13C9-PFNA)	4.944	1.25	472	427	-	-	-
37. Perfluorooctane sulfonate (PFOS)	4.949	2.5	499	80	99	-	-
38. Sodium perfluoro-[13C4]octanesulfonate (13C4-PFOS)	4.95	2.5	503	80	99	-	-
39. Sodium perfluoro-[13C8]octanesulfonate (13C8-PFOS)	4.95	2.5	507	80	99	-	-
40. 3-Perfluoroheptyl propanoic acid (7:3FTCA)	5.046	62.5	441	317	337	-	-
41. 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	5.114	10	531	351	-	533	353
42. Perfluorodecanoic acid (PFDA)	5.237	2.5	513	469	219	-	-
43. Perfluoro-*n*-[1,2,3,4,5,6- 13C2]decanoic acid (13C2-PFDA)	5.237	1.25	515	470	-	-	-
44. Perfluoro-*n*-[1,2,3,4,5,6- 13C6]decanoic acid (13C6-PFDA)	5.237	1.25	519	474	-	-	-
45. 1*H*,1*H*,2*H*,2*H*-perfluorodecane sulfonate (8:2 FTS)	5.238	10	527	507	81	-	-
46. Sodium 1*H*,1*H*,2*H*,2*H*-perfluoro-1-[1,2- 13C2]-decane sulfonate (13C2-8:2FTS)	5.238	5	529	81	509	-	-
47. Perfluorononanesulfonic acid (PFNS)	5.239	2.5	549	80	99	-	-
48. *N*-methyl-d 3-perfluoro-1-octanesulfonamidoacetic acid (D3-NMeFOSAA)	5.361	5	573	419	-	-	-
49. *N*-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	5.366	2.5	570	419	483	-	-
50. Perfluorodecanesulfonic acid (PFDS)	5.475	2.5	599	80	99	-	-
51. Perfluoroundecanoic acid (PFUnA)	5.486	2.5	563	519	269	-	-
52. Perfluoro-*n*-[1,2,3,4,5,6,7- 13C7]undecanoic acid (13C7-PFUnA)	5.486	1.25	570	525	-	-	-
53. *N*-ethyl-d 5-perfluoro-1-octanesulfonamidoacetic acid (D5-NEtFOSAA)	5.487	5	589	419	-	-	-
54. *N*-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	5.487	2.5	584	419	526	-	-
55. 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	5.59	10	631	451	-	633	453
56. Perfluorooctanesulfonamide (PFOSA)	5.669	2.5	498	78	478	-	-
57. Perfluoro-1-[13C8]octanesulfonamide (13C8-PFOSA)	5.669	2.5	506	78	-	-	-
58. Perfluorododecanoic acid (PFDoA)	5.691	2.5	613	569	319	-	-
59. Perfluoro-*n*-[1,2- 13C2]dodecanoic acid (13C2-PFDoA)	5.692	1.25	615	570	-	-	-
60. Perfluorododecanesulfonic acid (PFDoS)	5.861	2.5	699	80	99	-	-
61. Perfluorotridecanoic acid (PFTrDA)	5.876	2.5	663	619	169	-	-
62. Perfluorotetradecanoic acid (PFTeDA)	6.031	2.5	713	669	169	-	-
63. Perfluoro-*n*-[1,2- 13C2]tetradecanoic acid (13C2-PFTeDA)	6.031	1.25	715	670	-	-	-
64. *N*-methyl perfluorooctanesulfonamide (NMeFOSA)	6.158	2.5	512	219	169	-	-
65. *N*-methyl-D 3-perfluoro-1-octanesulfonamide (D3-NMeFOSA)	6.159	2.5	515	219	-	-	-
66. *N*-methyl perfluorooctanesulfonamidoethanol (NMeFOSE)	6.166	25	616	59	-	-	-
67. *N*-methyl-D 7-perfluorooctanesulfonamidoethanol (D7-NMeFOSE)	6.167	25	623	59	-	-	-
68. *N*-ethyl-D 5-perfluoro-1-octanesulfonamide (D5-NEtFOSA)	6.295	2.5	531	219	-	-	-
69. *N*-ethyl perfluorooctanesulfonamide (NEtFOSA)	6.296	2.5	526	219	169	-	-
70. *N*-ethyl perfluorooctanesulfonamidoethanol (NEtFOSE)	6.298	25	630	59	-	-	-
71. *N*-ethyl-D 9-perfluorooctanesulfonamidoethanol (D9-NEtFOSE)	6.298	25	639	59	-	-	-

Table I: Results from MDL, Blank, Precision, and Accuracy Experiments for Native PFAS

Compound	Abbreviation	MDL (ng/L)	Blank (ng/L)*	Accuracy (%)	(%RSD)
Perfluorobutanoic acid	PFBA	0.34	ND	112	5
Perfluoro-3-methoxypropanoic acid	PFMPA	0.20	ND	109	6
3-Perfluoropropyl propanoic acid	3:3FTCA	0.31	ND	95	6
Perfluoropentanoic acid	PFPeA	0.26	ND	111	6
Perfluorobutane sulfonate	PFBS	0.19	ND	101	5
Perfluoro-4-methoxybutanoic acid	PFMBA	0.18	ND	111	6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	0.15	ND	94	5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	0.46	ND	121	5
1*H*,1*H*,2*H*,2*H*-perfluorohexane sulfonate	4:2 FTS	0.36	ND	104	5
2*H*,2*H*,3*H*,3*H*-Perfluorooctanoic acid	5:3FTCA	1.48	ND	102	6
Perfluorohexanoic acid	PFHxA	0.08	ND	113	5
Perfluoropentane sulfonate	PFPeS	0.07	ND	122	4
Hexafluoropropylene oxide dimer acid	HFPO-DA	0.40	ND	114	12
Perfluoroheptanoic acid	PFHpA	0.08	ND	109	5
Perfluorohexane sulfonate	PFHxS	0.07	ND	104	7
4,8-Dioxa-3*H*-perfluorononanoic acid	ADONA	0.39	ND	99	9
1*H*,1*H*,2*H*,2*H*-perfluorooctane sulfonate	6:2 FTS	0.37	ND	107	4
3-Perfluoroheptyl propanoic acid	7:3FTCA	2.32	ND	100	6
Perfluoroheptane sulfonate	PFHpS	0.76	ND	121	8
Perfluorooctanoic acid	PFOA	0.32	ND	104	2
Perfluorooctane sulfonate	PFOS	0.20	ND	112	7
Perfluorooctanesulfonamide	PFOSA	0.21	ND	81	11
Perfluorononanoic acid	PFNA	0.08	ND	114	5
N-methyl perfluorooctanesulfonamide	NMeFOSA	0.07	ND	104	13
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	0.61	ND	85	11
Perfluorononanesulfonic acid	PFNS	0.25	ND	79	7
Perfluorodecanoic acid	PFDA	0.18	ND	117	8
N-ethyl perfluorooctanesulfonamide	NEtFOSA	0.14	ND	112	15
1*H*,1*H*,2*H*,2*H*-perfluorodecane sulfonate	8:2 FTS	0.91	ND	113	11
Perfluoroundecanoic acid	PFUnA	0.26	ND	125	12
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	0.15	ND	95	12
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	0.23	ND	93	12
Perfluorodecanesulfonic acid	PFDS	0.25	ND	94	23
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	0.56	ND	77	25
Perfluorododecanoic acid	PFDoA	0.27	ND	118	15
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	1.11	ND	106	9
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1.10	ND	111	9
Perfluorotridecanoic acid	PFTrDA	0.33	ND	108	15
Perfluorododecanesulfonic acid	PFDoS	0.13	ND	89	12
Perfluorotetradecanoic acid	PFTeDA	0.32	ND	102	23

*ND = Not detected above MDL.

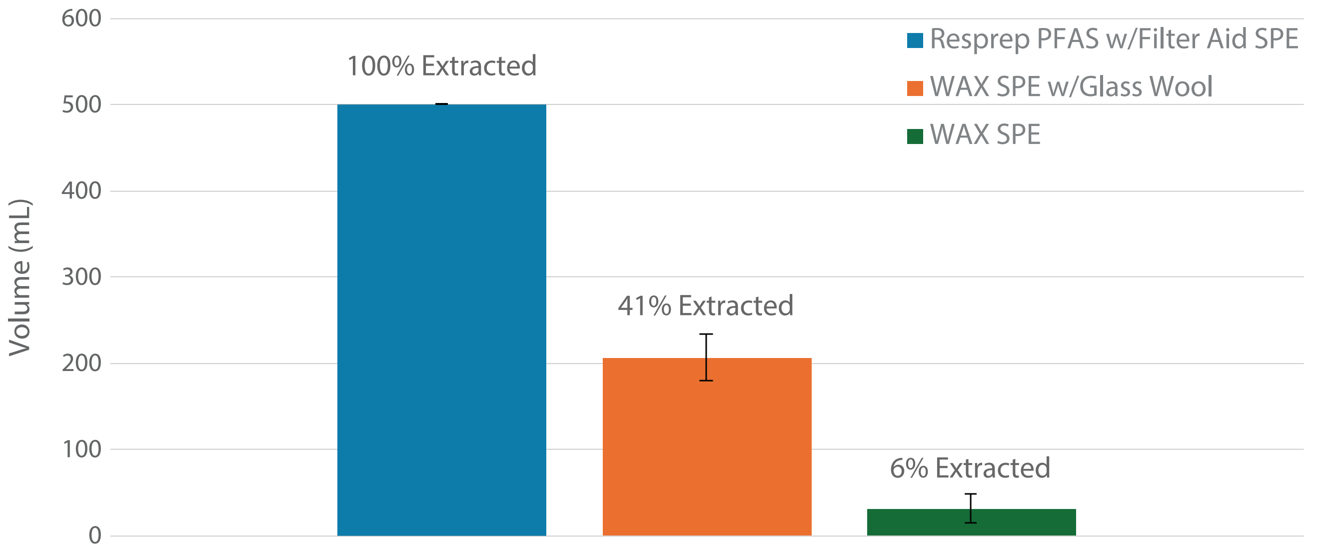
Table II: Results from Precision and Accuracy Experiments for Isotope Dilution Standards

Compound	Abbreviation	Accuracy (%)	%RSD
Perfluoro-*n*-[1,2,3,4- 13C4]butanoic acid	$^{13}C_4$-PFBA	88	4
Perfluoro-*n*-[1,2,3,4,5- 13C5]pentanoic acid	$^{13}C_5$-PFPeA	88	5
Sodium perfluoro-1-[2,3,4- 13C3]butanesulfonate	$^{13}C_3$-PFBS	97	3
Sodium 1*H*,1*H*,2*H*,2*H*-perfluoro-1-[1,2- 13C2]hexane sulfonate	^{13}C2-4:2FTS	95	8
Perfluoro-*n*-[1,2,3,4,6- 13C5]hexanoic acid	$^{13}C_5$-PFHxA	122	2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy13C3-propanoic acid	$^{13}C_3$-HFPO-DA	92	4
Perfluoro-*n*-[1,2,3,4- 13C4]heptanoic acid	$^{13}C_4$-PFHpA	107	4
Sodium perfluoro-1-[1,2,3- 13C3]hexanesulfonate	$^{13}C_3$-PFHxS	102	4
Sodium 1*H*,1*H*,2*H*,2*H*-perfluoro-1-[1,2-13C2]-octane sulfonate	$^{13}C_2$-6:2FTS	83	5
Perfluoro-*n*-[13C8]octanoic acid	$^{13}C_8$-PFOA	106	5
Sodium perfluoro-[13C8]octanesulfonate	$^{13}C_8$-PFOS	77	7
Perfluoro-1-[13C8]octanesulfonamide	$^{13}C_8$-PFOSA	94	11
N-methyl-d 3-perfluoro-1-octanesulfonamidoacetic acid	D_3-NMeFOSAA	85	9
N-ethyl-d 5-perfluoro-1-octanesulfonamidoacetic acid	D_5-NEtFOSAA	87	11
Perfluoro-*n*-[13C9]nonanoic acid	$^{13}C_9$-PFNA	83	7
Perfluoro-*n*-[1,2,3,4,5,6- 13C6]decanoic acid	$^{13}C_6$-PFDA	80	7
Sodium 1*H*,1*H*,2*H*,2*H*-perfluoro-1-[1,2- 13C2]-decane sulfonate	$^{13}C_2$-8:2FTS	84	5
N-methyl-D 7-perfluorooctanesulfonamidoethanol	D_7-NMeFOSE	108	13
N-ethyl-D 9-perfluorooctanesulfonamidoethanol	D_9-NEtFOSE	88	17
N-methyl-D 3-perfluoro-1-octanesulfonamide	D_3-NMeFOSA	84	8
N-ethyl-D 5-perfluoro-1-octanesulfonamide	D_5-NEtFOSA	111	10
Perfluoro-*n*-[1,2,3,4,5,6,7- 13C7]undecanoic acid	$^{13}C_7$-PFUnA	78	13
Perfluoro-*n*-[1,2- 13C2]dodecanoic acid	$^{13}C_2$-PFDoA	45	24
Perfluoro-*n*-[1,2- 13C2]tetradecanoic acid	$^{13}C_2$-PFTeDA	36	21

Filtration and Sample Flow Experiment

Resprep PFAS SPE cartridges are available with and without a factory-packed filter aid that allows faster, more consistent sample preparation compared to using manually packed glass wool. As demonstrated in Figure 3, the filter aid provides another significant benefit: it prevents clogging so that the full sample can be processed, saving the time, effort, and cost of a second extraction. In this comparison, only the Resprep PFAS cartridge with filter aid allowed for extraction of the full 500 mL of the substitute wastewater matrix containing 100 mg/L suspended solids. This is a great improvement over the use of glass wool, which could extract less than half of the sample, and WAX alone, which could extract less than a tenth of the sample.

Figure 3: Results from Filtration and Sample Flow Experiment



The average volume of 500 mL wastewater sample extracted before clogging. Error bars are ±SD of three replicates.

Conclusion

Nondrinking water matrices present unique challenges to labs using SPE sample preparation. Resprep PFAS cartridges allows labs to handle difficult water matrices more efficiently while meeting or exceeding the requirements of EPA Method 1633 for PFAS analysis. Visit www.restek.com/PFAS for additional products, methods, and technical resources.

References

1. U.S. Environmental Protection Agency, Method 1633, Analysis of per- and polyfluoroalkyl substances (PFAS) in aqueous, solid, biosolids, and tissue samples by LC-MS/MS, January 2024. https://www.epa.gov/system/files/documents/2024-01/method-1633-final-for-web-posting.pdf
2. ASTM International, D5905-98(2018), Standard practice for the preparation of substitute wastewater, December 2018. https://www.astm.org/d5905-98r18.html
3. U.S. Environmental Protection Agency, Definition and procedure for the determination of the method detection limit, Revision 2, December 2016. https://www.epa.gov/sites/default/files/2016-12/documents/mdl-procedure_rev2_12-13-2016.pdf

Force C18 LC Column

- A traditional end-capped C18 ideal for general-purpose use in reversed-phase chromatography.
- Wide pH range (2–8) provides excellent data quality for many applications, matrices, and compounds.
- High carbon load (20%) offers high hydrophobic retention.

Catalog No.	Product Name	Units
9634252	Force C18 LC Column, 1.8 µm, 50 mm x 2.1 mm	ea.

PFAS Delay Column

- Traps system-related PFAS, preventing interference and ensuring accurate trace-level analysis of PFAS in samples.
- Universal compatibility: works with
    - any HPLC or UHPLC up to 15,000 psi (1034 bar);
    - both FPP and SPP analytical columns; and
    - all stationary phases.
- Highly retentive of system-related PFAS; no breakthrough even with extended equilibration times.
- Easy installation with standard fittings.

Catalog No.	Product Name	Units
27854	PFAS Delay Column, 5 µm, 50 x 2.1 mm HPLC Column	ea.

Resprep PFAS Cartridges

- Optional factory-packed filter aid prevents clogging, so samples are processed completely, in less time, and more consistently than with manually packed glass wool.
- Streamlined sample prep: WAX and CarboPrep Plus carbon sorbents are factory packed in a single dual-bed SPE cartridge for a faster, simpler workflow.
- Ultra-clean polymeric and carbon sorbents—every WAX lot is tested for Method 1633 PFAS to ensure method detection limits can be met.
- Compatible with both vacuum manifolds and automated systems.

WAX on top for aqueous samples.

582051

Carbon on top for solid samples.

Catalog No.	Product Name	Sorbent Phase	Volume (mL)	Units
28930	Resprep PFAS Cartridge	WAX 150 mg/GCB 50 mg	6	30-pk.
28931	Resprep PFAS Cartridge with Filter Aid	Filter aid 2000 mg/ WAX 150 mg/GCB 50 mg	6	30-pk.
582051*	Resprep PFAS Cartridge	GCB 50 mg/WAX 150 mg	6	30-pk.

*Please contact Restek Customer Service to order custom catalog no. 582051.

Resprep QR-12 Vacuum Manifold

Catalog No.	Product Name	Units
28298-VM	Resprep QR-12 Vacuum Manifold (Includes Cover with Flow Control Valves & Gasket (cat.# 28316-VM); Collection Rack (cat.# 28318-VM); Plate for 16 mm Test Tubes (cat.# 28319-VM); 100-pk. Quick Replace Liners, PTFE (cat.# 28310-VM); 12-pk. Liner Guide (cat.# 28312-VM); 12-pk. Test Tubes (cat.# 28315-VM))	kit

Quick Replace Liners

Catalog No.	Product Name	Units
28310-VM	Quick Replace Liners, PTFE, for Resprep QR Vacuum Manifolds	100-pk.

Resprep Sample Delivery System

Catalog No.	Product Name	Units
26250	Resprep Sample Delivery System	6-pk.

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