

Extraction of 40 PFAS Compounds from Soil and Tissue Following EPA Method 1633A

Abstract

Per- and polyfluoroalkyl substances (PFAS) are a class of manmade chemicals used in various industries due to their favorable properties for goods such as nonstick cookware and firefighting foam. Their stability and widespread use have contributed to their accumulation in the environment, and the lack of remediation techniques for their removal has allowed for their bioaccumulation in humans and animals. PFAS have been shown to cause health issues in humans, such as cancer, endocrine disruption, and infertility. Thus, monitoring environmental solid samples, such as soil and tissue, is critical. EDGE PFAS™ is an automated solvent extraction system designed for the extraction of PFAS from solid or semi-solid samples. In this study, the EDGE PFAS system was used to extract 40 spiked PFAS compounds from soil and tissue following EPA Method 1633A.1 The automated extraction was completed in less than 15 minutes per sample and yielded acceptable recoveries without carryover in the system. The EDGE PFAS system is an ideal option for laboratories that want to automate their PFAS extractions of solid or semi-solid samples.

Introduction

There are currently thousands of PFAS compounds that have been used extensively across many industries. Due to their exceptional durability and bioaccumulation, they have earned the moniker of forever compounds. PFAS possess a chain of linked carbon atoms with fluorine atoms branching off of the main chain. The presence of the strong carbon-fluorine bond contributes to the stability of these compounds. Due to their ubiguity, PFAS have leached into the environment through production and waste streams, making their way into water sources. From these water sources, PFAS can rapidly spread, contaminating soil and biological tissue. Furthermore, these compounds have been found to bioaccumulate in animals and humans, and exposure in humans has been shown to cause adverse health outcomes. Thus, the assessment of the levels of PFAS in the environment is important to the health and safety of humans.

The Environmental Protection Agency (EPA) have provided EPA Method 1633A¹ for analysis of PFAS, including soil and tissue sample types. The extraction method for the solid samples detailed in this method is a long, manual process. Since the method is performance-based, the extraction can be modified, as long as quality control requirements are met. The EDGE PFAS system can be used to extract both the soil and tissue samples in less than 15 minutes, automating the solvent addition, extraction, and filtration of the extract. This allows for a rapid, efficient, and simple extraction of PFAS from these solid environmental samples.

In this work, the EDGE PFAS system was utilized to effectively extract PFAS from soil and tissue samples with acceptable recoveries. Animal tissues are difficult matrices to extract and add complexity to both the sample preparation and the analysis. With the EDGE PFAS system, one simple method can be applied to many different difficult sample types.

Materials and Methods

Reagents and Samples

A quality control study was performed at Eurofins Lancaster Laboratories and the resulting data presented herein. The control samples, solvents, solvent modifiers, spiking solutions, and cleanup materials were provided by Eurofins. The eCleanUP™, a proprietary sorbent, was provided by CEM Corporation.

EDGE Sample Preparation

Each Q-Cup® was rinsed with methanol and allowed to dry prior to use. Q-Cups were prepared with the Q-Disc® PFAS, followed by weighing 1 g of eCleanUP then 5 g of soil representative matrix or 2 g of tissue representative matrix into each Q-Cup. Each sample was spiked with native PFAS at the concentrations listed in **Table 1** (page 2), resulting in mid-spike laboratory control samples and low-spike laboratory control samples for soil and tissue. The extracted internal standards (EIS) were spiked at the concentrations listed in EPA Method 1633A. All Q-Cups, along with polypropylene centrifuge tubes, were loaded into an EDGE PFAS rack and extracted on the EDGE PFAS system using the methods listed on page 2 and 3.

Table 1. Spiked Concentrations of Native PFAS Compounds (ng/g)

Compound	Soil Mid	Soil Low	Tissue Mid Snike	Tissue Low
	Бріке		эріке	
	9.98	1.59	25.0	4.02
	4.99	0.795	12.5	2.01
Perfluorohexanoic acid (PFHxA)	2.50	0.398	6.25	1.01
Perfluoroheptanoic acid (PFHpA)	2.50	0.398	6.25	1.01
Perfluorooctanoic acid (PFOA)	2.50	0.398	6.25	1.01
Perfluorononanoic acid (PFNA)	2.50	0.398	6.25	1.01
Perfluorodecanoic acid (PFDA)	2.50	0.398	6.25	1.01
Perfluoroundecanoic acid (PFUnA)	2.50	0.398	6.25	1.01
Perfluorododecanoic acid (PFDoA)	2.50	0.398	6.25	1.01
Perfluorotridecanoic acid (PFTrDA)	2.50	0.398	6.25	1.01
Perfluorotetradecanoic acid (PFTeDA)	2.50	0.398	6.25	1.01
Perfluorobutanesulfonic acid (PFBS)	2.22	0.353	5.55	0.920
Perfluoropentanesulfonic acid (PFPeS)	2.35	0.374	5.88	0.945
Perfluorohexanesulfonic acid (PFHxS)	2.28	0.363	5.70	0.917
Perfluoroheptanesulfonic acid (PFHpS)	2.38	0.379	5.96	0.959
Perfluorooctanesulfonic acid (PFOS)	2.32	0.37	5.81	0.935
Perfluorononanesulfonic acid (PFNS)	2.40	0.383	6.01	0.967
Perfluorododecanesulfonic acid (PFDoS)	2.42	0.386	6.06	0.975
1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2 FTS)	9.36	1.49	23.5	3.77
1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2 FTS)	9.50	1.51	23.8	3.83
1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2 FTS)	9.58	1.53	24.0	3.86
Perfluorooctanesulfonamide (PFOSA)	2.50	0.398	6.25	1.01
N-methylperfluorooctane sulfonamide (NMeFOSA)	2.50	0.398	6.25	1.01
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2.50	0.398	6.25	1.01
N-ethylperfluorooctane sulfonamide (NEtFOSA)	2.50	0.398	6.25	1.01
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2.50	0.398	6.25	1.01
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	25.0	3.98	62.5	10.1
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	25.0	3.98	62.5	10.1
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	9.98	1.59	25.0	4.02
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	9.44	1.50	23.7	3.80
Perfluoro-4-methoxybutanoic acid (PFMBA)	4.99	0.795	12.5	2.01

Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	4.99	0.795	12.5	2.01
9-Chlorohexadecafluoro-3-oxanonane-1- sulfonic acid (9CI-PF3ONS)	9.32	1.49	23.4	3.75
11-Chloroeicosafluoro-3-oxaundecane-1- sulfonic acid (11Cl-PF30UdS)	9.42	1.50	23.6	3.79
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	4.45	0.709	11.2	1.79
3-Perfluoropropylpropanoic acid (3:3 FTCA)	12.5	1.99	31.3	5.03
3-Perfluoropentylpropanoic acid (5:3 FTCA)	62.4	9.94	156	25.1
3-Perfluoroheptylpropanoic acid (7:3 FTCA)	62.4	9.94	156	25.1
Perfluorodecanesulfonic acid (PFDS)	2.41	0.383	6.03	0.969
Perfluoro-3-methoxypropanoic acid (PFMPA)	4.99	0.795	12.5	2.01

EDGE PFAS Method for PFAS from Soil

Q-Disc: Q-Disc PFAS

Cycle 1

Extraction Solvent: 0.3% ammonium hydroxide in methanol Top Add: 10 mL Temperature: 65 °C Hold Time: 03:00 (mm:ss)

Cycle 2

Extraction Solvent: 0.3% ammonium hydroxide in methanol Top Add: 15 mL Temperature: 65 °C Hold Time: 03:00 (mm:ss) Rinse Solvent: 0.3% ammonium hydroxide in methanol Rinse: 5 mL

Wash 1

Wash Solvent: Isopropanol Wash Volume: 10 mL Temperature: ---

Hold Time: --:-- (mm:ss)

Wash 2

Wash Solvent: 0.3% ammonium hydroxide in methanol Wash Volume: 10 mL Temperature: 65 °C Hold Time: 00:30 (mm:ss)

Wash 3

Wash Solvent: 0.3% ammonium hydroxide in methanol Wash Volume: 10 mL Temperature: ---Hold Time: --:- (mm:ss)



EDGE PFAS Method for PFAS from Tissue

Q-Disc: Q-Disc PFAS

Cycle 1

Extraction Solvent: 0.05M KOH in methanol Top Add: 10 mL Temperature: 65 °C Hold Time: 03:00 (mm:ss)

Cycle 2

Extraction Solvent: Acetonitrile Top Add: 10 mL Temperature: 65 °C Hold Time: 03:00 (mm:ss) Rinse Solvent: 0.05M KOH in methanol Rinse: 5 ml

Wash 1

Wash Solvent: Isopropanol Wash Volume: 10 mL Temperature: --Hold Time: --:-- (mm:ss)

Wash 2

Wash Solvent: 0.05M KOH in methanol Wash Volume: 10 mL Temperature: 65 °C Hold Time: 00:30 (mm:ss)

Wash 3

Wash Solvent: 0.05M KOH in methanol Wash Volume: 10 mL Temperature: --Hold Time: --:-- (mm:ss)

Post-Extraction Cleanup and Analysis

All samples were subject to concentration or dilution, pH adjustment, and cleanup according to EPA 1633A, performed by Eurofins Lancaster Laboratories. Analysis was also completed by Eurofins Lancaster Laboratories, following 1633A methodology.

Results

Representative samples for both the soil and tissue were extracted with simple and rapid automated extraction methods; the same parameters were used for both sample types, with exception of the extraction solvent and volume. The extraction solvents were chosen based on EPA Method 1633A. The addition of eCleanUP to the soil samples facilitates the extraction of high moisture samples. For both sample types, extraction took less than 15 minutes per sample, including solvent addition, extraction, and filtration. The same clean up and analysis procedures were applied to all samples. Acceptable percent recovery limits were 40-150% for the native compounds and 20-150% for the extracted internal compounds. Acceptable percent recoveries were achieved for the native compounds and the extracted internal standards in both sample types, as seen in Table 2 (page 4) and Table 3 (page 5). Furthermore, no PFAS were detected above the limit of quantification in solvent blanks that were run between the samples. Using traditional extraction techniques, three long cycles are generally required to efficiently extract both soil and tissue samples, with tissue samples taking longer than 16 hours. Utilizing the EDGE PFAS system, three cycles were completed in less than 15 minutes to achieve acceptable recoveries for both soil and tissue QC samples.

Conclusion

PFAS are an ongoing issue for environmental contamination and, as the scope of required testing increases, the more we learn. Their migration throughout the ecosystem has led to PFAS contamination being discovered nearly in all corners of the globe and in all manner of living beings. As analysis methods increase in sensitivity, simpler and quicker extraction methods are also needed to contend with the increasing sample throughput required. In this study, we have shown the use of the EDGE PFAS system to extract spiked soil and tissue samples. Acceptable recoveries were achieved with a rapid, simple, and efficient automated extraction method.



Table 2. % Recovery Values for Both a Middle and Low Range Spike of 40 Native PFAS in Soil and Tissue

Compound	Soil Mid Spike	Soil Low Spike	Tissue Mid Spike	Tissue Low Spike
	Recovery	Recovery	Recovery	Recovery
Perfluorobutanoic acid (PFBA)	105	107	64	110
Perfluoropentanoic acid (PFPeA)	106	107	63	105
Perfluorohexanoic acid (PFHxA)	110	118	70	113
Perfluoroheptanoic acid (PFHpA)	125	103	66	103
Perfluorooctanoic acid (PFOA)	130	127	75	134
Perfluorononanoic acid (PFNA)	106	119	63	118
Perfluorodecanoic acid (PFDA)	116	108	71	120
Perfluoroundecanoic acid (PFUnA)	113	104	69	105
Perfluorododecanoic acid (PFDoA)	82	125	47	118
Perfluorotridecanoic acid (PFTrDA)	110	117	73	119
Perfluorotetradecanoic acid (PFTeDA)	128	120	83	112
Perfluorobutanesulfonic acid (PFBS)	114	110	71	103
Perfluoropentanesulfonic acid (PFPeS)	113	111	60	98
Perfluorohexanesulfonic acid (PFHxS)	103	93	68	107
Perfluoroheptanesulfonic acid (PFHpS)	100	114	52	100
Perfluorooctanesulfonic acid (PFOS)	105	144	63	139
Perfluorononanesulfonic acid (PFNS)	120	109	73	108
Perfluorododecanesulfonic acid (PFDoS)	98	104	58	92
1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2 FTS)	122	102	49	87
1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2 FTS)	117	110	62	107
1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2 FTS)	132	113	72	103
Perfluorooctanesulfonamide (PFOSA)	106	107	62	199
N-methylperfluorooctane sulfonamide (NMeFOSA)	115	110	65	103
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	109	113	71	105
N-ethylperfluorooctane sulfonamide (NEtFOSA)	135	123	78	117
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	110	106	71	112
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	115	113	80	110
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	100	103	84	115
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	105	107	61	116
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	108	105	79	125
Perfluoro-4-methoxybutanoic acid (PFMBA)	85	99	69	104
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	130	141	61	99
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CI-PF30NS)	89	96	86	134
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF30UdS)	76	87	73	111
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	123	117	77	109
3-Perfluoropropylpropanoic acid (3:3 FTCA)	89	88	62	96
3-Perfluoropentylpropanoic acid (5:3 FTCA)	91	92	59	88
3-Perfluoroheptylpropanoic acid (7:3 FTCA)	95	78	63	98
Perfluorodecanesulfonic acid (PFDS)	104	102	70	94
Perfluoro-3-methoxypropanoic acid (PFMPA)	112	102	72	102



Table 3. % Recovery Values for the Extracted InternalStandards in Soil and Tissue

Compound	Soil Mid Spike Recovery	Soil Low Spike Recovery	Tissue Mid Spike Recovery	Tissue Low Spike Recovery
$13C_4$ PFBA	85.8	86.3	59.9	53.9
$13C_{5}$ PFPeA	84.3	87.0	56.5	52.2
$13C_{5}$ PFHxA	77.4	81.9	56.0	51.5
$13C_4$ PFHpA	78.7	88.3	63.4	59.1
12C8 PFOA	79.0	76.9	64.0	55.4
13C9 PFNA	81.0	79.3	58.9	54.6
$13C_6$ PFDA	83.6	84.7	64.2	55.1
12C7 PFUnA	81.2	83.3	71.5	63.0
$13C_2$ PFTeDA	66.0	71.2	59.0	57.2
$13C_{3}$ PFBS	90.5	88.6	56.5	56.0
$13C_{3}$ PFHxS	86.5	95.3	63.2	57.6
13C8 PFOS	74.7	85.7	66.2	66.2
13C8 FOSA	76.4	84.0	61.2	63.6
d3-NMeFOSAA	91.5	95.0	63.7	68.0
d2-NEtFOSAA	106	111	70.5	73.0
13C ₂ 4:2 FTS	84.0	93.6	86.1	78.5
13C ₂ 6:2 FTS	83.5	99.0	63.4	55.2
13C ₂ 8:2 FTS	68.4	77.3	57.4	61.6
$13C_{3}$ HFPO-DA	92.8	90.0	61.5	57.9
D7-NMeFOSE	74.9	83.0	47.1	54.0
D9-NEtFOSE	76.9	82.7	46.2	50.2
d5-NEtPFOSA	56.4	64.5	59.7	59.6
d3-NMePFOSA	66.6	75.7	69.3	71.7
13C ₂ PFDoA	67.5	70.8	62.7	54.9

Reference

¹ United States Environmental Protection Agency. Method 1633A Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/ MS, Revision A, December, 2024. <u>https://www.epa.gov/</u> <u>system/files/documents/2024-12/method-1633a-december-</u> <u>5-2024-508-compliant.pdf</u> (accessed May 28, 2025).

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