Application Note

One Step Extraction, Cleanup and Concentration of Per- and Polyfluoroalkyl Substances in Biological, Environmental and Food Matrices

Per- and polyfluoroalkyl substances (PFAS) constitute a group of compounds characterized by perfluorinated or polyfluorinated carbon chain moieties, typically denoted by structures such as F(CF2)n or F(CF2)n-(C2H4)n. Due to their unique properties, these substances have found extensive application in various industrial and consumer products.

Recent developments in the United States have led to the introduction of EPA method 1633 (in 2024), which addresses the need for robust methodologies to monitor and analyze PFAS in solid matrices such as fish, meat and soil.

In this application note we describe a new simple and fast methodology for processing solid PFAS samples.

Key features are:

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- Combines Extraction, Cleanup & Concentration in a One-Step Process
- The closed-loop design eliminates PFAS cross-contamination.
- Reduces Sample Preparation Cost and Time
- Reduces PFAS food & environmental testing from a day to a few hours
- Revolutionizing PFAS Analysis — From a Full Day to Just a Few Hours
- It's modular & expandable from 1 to 8 samples.
- Integrated nitrogen and heat evaporator for direct, in-line concentration.
- Automated pressure and flow control with real-time monitoring.
- Interchangeable WAX/Graphite cartridges for PFAS extraction.
- Compact modular architecture—adaptable for food, water, and soil matrices.

Instrumentation

- FMS Pressurized Liquid Extraction (PLE®) System. The system is modular in nature and can be extended to a total of 8 modules for a total of 8 samples processed in parallel.
- Agilent 6475 TripleQuad LC/MS

Consumables

- FMS WAX/graphite cartridges
- Ultrapure DI water
- Methanol pesticide grade
- Relevant PFAS spiking standards

Method

- **<u>One-Step Extraction, Cleanup & Concentration System</u>:**
- FMS's patent-pending One-Step System revolutionizes PFAS sample preparation by merging three separate steps into a single, automated process. It eliminates contamination, accelerates analysis, and reduces cost, allowing laboratories to process more samples in less time.
- The integrated system minimizes handling and solvent use while maintaining high reproducibility—ideal for environmental, biological, and food matrices.
- **<u>Contamination-Free Design</u>:**
- PFAS are found at ppt–ppq levels, making contamination control essential. Even minor exposure from tubing, fittings, glassware, or operators can compromise results. FMS eliminates these risks through:
- · Fully enclosed flow paths to prevent exposure.
- · PTFE-free solvent and gas contact surfaces.
- · Nitrogen purge and heating elements to remove solvent carryover.
- This ensures reliable, contamination-free performance that meets the strictest QA/QC standards.
- **<u>How it Works</u>:**
- The Pressurized Liquid Extraction (PLE) system automates PFAS extraction from soil, tissue, fish, and other solids. It applies pressure and temperature when needed to
- accelerate solvent extraction, recovering PFAS efficiently while using less solvent. The PLE
- connects directly to a WAX/Graphite cartridge for cleanup and transfers extracts directly to the
- evaporator, concentrating to the final volume—all within a few hours.

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Analysis

■ Take aliquot from final 5 mL extract (Method 1633 does not require volume reduction of final extract)
■ Agilent 1290 Infinity II LC System
■ Agilent 6475 Triple quad LC/MS
■ Agilent Zorbax Eclipse Plus C18 column 3.0 x 50 mm, 1.8 um
■ Column temperature 40 °C
■ Injection 5.0 uL
■ Mobile phase 5 mM ammonium acetate in 95% water, 5% acetonitrile (A) and methanol (B)
■ Gradient
  0 min 98% A 2% B
  0.2 min 98% A 2% B
  10 min 5% a 95% B
■ Stop time 12.2 min
■ Dynamic MRM negative electrospray
■ T (gas) = 230 °C
■ T (sheath) = 355 °C

Pressurized Liquid Extraction controller and module.

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	SOIL	FISH	MEAT	EPA 1633
	% Recoveries	% Recoveries	% Recoveries	% Criteria
13C2-4-2 FTSA	85	99	87	40-200
13C2-6-2 FTS	76	109	74	40-200
13C2-8-2 FTSA	92	103	76	40-300
13C2-PFDoDA	108	82	67	10-130
13C2-PFTDA	95	88	72	10-130
13C3-HFPO-DA	93	65	69	40-130
13C3-PFBS	83	74	77	40-135
13C3-PFHxS	80	77	65	40-130
13C4-PFBA	93	82	94	5-130
13C4-PFHpA	91	69	72	40-130
13C5-PFHxA	91	65	60	40-130
13C5-PFPeA	92	66	69	40-130
13C6-PFDA	105	80	67	40-130
13C7-PFUnA	101	87	61	30-130
13C8-PFOA	100	72	67	50-200
13C8-PFOS	93	83	68	50-200
13C8-PFOSA	92	79	61	40-130
13C9-PFNA	94	61	66	40-130
2H3-N-MeFOSA	86	71	61	10-130
2H3-N-MeFOSAA	93	1	88	40-170
2H5-N-EtFOSA	88	72	79	10-130
2H5-N-EtFOSAA	98	106	74	25-135
2H7-MeFOSE	90	89	64	10-130
2H9-EtFOSE	88	70	60	10-130

Table 1. **Recoveries (%) and acceptance windows (%) for 24 surrogate PFAS in soil, fish and meat.**

Compound Method Name	Average for Soil per 5 g Final Conc. (ng/ml)	Average for Meat per 2 g beef Final Conc. (ng/ml)	Average for Fish per 2 g salmon Final Conc. (ng/ml)
11Cl-PF3OUdS	0.0013	0.0029	0.0031
3-3 FTCA	0.0141	0.0380	0.0160
4-2 FTS	0.0069	0.0082	0.0274
5-3 FTCA	0.0125	0.0000	0.0000
6-2 FTS	1.5011	5.0822	5.3701
7-3 FTCA	0.0408	0.1347	0.0522
8-2 FTS	0.1852	0.0000	0.2230
9Cl-PF3ONS	0.0051	0.0000	0.0000
ADONA	0.0066	0.0017	0.0088
EtFOSE	0.0472	0.7434	1.7347
HFPO-DA	0.0447	0.1148	0.1094
MeFOSE	0.0389	0.0206	0.0179
N-EtFOSA	0.1135	0.0863	0.0504
N-EtFOSAA	0.7694	0.0000	0.0000
NFDHA	0.0000	0.0591	0.0000
N-MeFOSA	0.0586	0.0000	0.0000
N-MeFOSAA	0.2191	0.0000	0.0000
PFBA	0.3984	0.0204	0.0346
PFBS	1.3575	0.0607	0.0491
PFDA	2.5426	0.0000	0.0000
PFDoA	1.4669	0.0000	0.0000
PFDoS	0.0000	0.0000	0.0000
PFDS	0.1033	0.0000	0.0000
PFEESA	0.0138	0.0331	0.0173
PFHpA	1.4543	0.0000	0.0000
PFHpS	0.2433	0.0000	0.0295
PFHxA	2.0593	0.2019	0.2147
PFHxS	0.4410	0.0000	0.1669
PFMBA	0.0083	0.0022	0.0028
PFMPA	0.1968	0.0402	0.0119
PFNA	1.8462	0.0000	0.0000
PFNS	0.0242	0.0000	0.0287
PFOA	4.8033	0.0000	0.0000
PFOS	13.9061	1.2793	2.1991
PFOSA	0.1028	0.0224	0.0303
PFPeA	0.5680	0.0280	0.0125
PFPeS	0.0359	0.0000	0.0000
PFTDA	0.5201	0.0000	0.0000
PFTrDA	0.6367	0.0000	0.0000
PFUnA	1.6558	0.0000	0.0000

Table 2. Native PFAS with PLE for method 1633 in Solid matrices (in ng/L)

Discussion and Conclusions

Benefits:

- Contamination-Free Workflow for accurate, reproducible results.
- Faster Turnaround: Reduces prep and analysis from a full day to hours.
- Lower Cost: Minimizes solvent, labor, and consumables.
- Regulatory Ready: Meets EPA and global PFAS method standards.
- Flexible Design: Scales from small labs to high-throughput facilities.

Applications:

- PFAS extraction from food, water, soil, wastewater, and biological samples.
- Environmental and public health testing.
- Industrial contamination and compliance monitoring.

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