

SPONSOR REPORT

APPLICATION REPORT
ETHOS X - PFAS - EPA Method 1633



OPTIMIZING EXTRACTION OF SOLIDS FOR PFAS - EPA 1633 ANALYTES

Extraction of PFAS compounds from solids is a common challenge for many laboratories. This application report demonstrates how using the Milestone ETHOS X platform with the XTR rotor simplifies sample preparation for extracting target PFAS compounds from soils, sediments and biosolids helps environmental laboratories address efficiency, regulatory, economic and quality challenges.

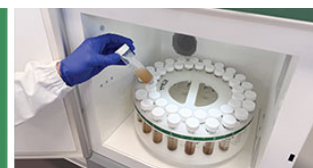
INTRODUCTION

Environmental laboratories face several types of challenges related to the determination of PFAS compounds in solid matrices such as soils, sediments and biosolids. Increasing industry competition for commercial labs puts analysis turn-around time at a premium. Labs face issues related to the availability of skilled labor, pressure on costs and budgets and limited lab space.

Sample preparation is a critical step in the entire analysis workflow as it is time-consuming, labor intensive and error prone. EPA method 1633 rev. A¹ describes extraction using a shaker table technique with multiple transfers between extractions taking a significant amount of time, labor and potential for losses due to repetitive

handling. However, EPA 1633 specifically allows improving method parameters such as extraction methodologies per section 9.1 of the method.

The use of microwave assisted extraction removes the obstacles of the shaker table approach, while providing consistent quality, lower cost and high efficiency in processing, with no regulatory obstacles and up to 70% reduction in extraction time. This Application Report describes how the Milestone ETHOS X platform provides superior efficiency and quality for the extraction of PFAS. Detailed independent laboratory data are presented showing PFAS Precision and Comparability along with Performance Evaluation data all using the Milestone ETHOS X with XTR extraction rotor.



EXPERIMENTAL

EQUIPMENT

- Milestone's ETHOS X equipped with XTR-44 extraction rotor or Milestone's ETHOS X LEAN equipped with XTR-30 extraction rotor
- 50-mL disposable PP vials
- Milestone's SFS-24 filtration system OR Centrifuge
- Nitrogen Evaporation system
- Automated SPE system
- LC/MS-MS system



Figure 1 – Milestone ETHOS X with XTR-44



Figure 2 – Milestone ETHOS X LEAN with XTR-30

SAMPLES

The study was conducted by preparing 3 different soil samples in triplicate to compare EPA 1633 shaker table vs. microwave extraction.

SAMPLE PREPARATION

In all cases, 5-gram samples (+ 0.01 g) were weighed on an as-received basis. Moisture content was determined per EPA 1633 guidance. For the blank spikes and QC samples, 5 g. of PFAS-free Ottawa sand was used. For the known soil (PT sample), 2.5 grams were utilized for extraction. Samples and QC were weighed into 50 mL disposable polypropylene centrifuge tubes and a NdFeB stir bar was introduced. An aliquot of extracted internal standard (EIS) was added to all samples/QC and allowed to sit 30 minutes per the method. 15 mL of 0.3% ammoniacal methanol was added to each sample/QC, cap tightened, and samples were placed into the Milestones ETHOS X- XTR-44 rotor. QC samples (LLOPR, OPR and MS/MSD) were spiked accordingly with PFAS targets. The samples were subjected to extraction using the program shown below:

Step	Time (min)	Power (W)	Temperature (°C)
1	5	up to 1000	65
2	10	up to 1000	65

Table 1 - Microwave Program

APPLICATION REPORT

ETHOS X – PFAS – EPA Method 1633

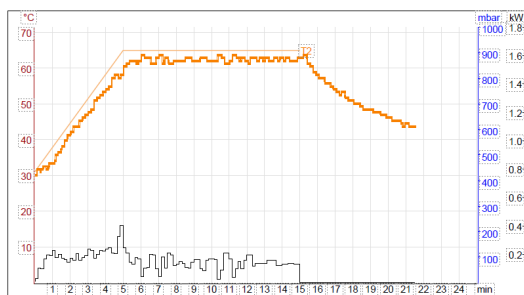
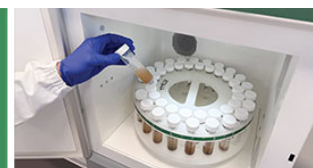


Figure 3 - Microwave run profile

EXTRACTION PROCESS AND CLEAN UP

At the end of cooling (< 5 mins), the vessels were placed into a centrifuge and centrifuged at 3000 rpm for 5-10 minutes. Alternatively, centrifugation can be eliminated by filtering through preconditioned, stacked 10um/1um on a Milestone SFS-24 vacuum filtration device

and filtered with rinsing into 50 mL PP tubes used for concentration.

Extracts were then concentrated using nitrogen evaporation to a volume of <10.0 mL. 40 mL PFAS-free water was then added. Extracts were then subjected to Solid Phase Extraction (SPE) resulting in a final volume of 5.0 mL. Non-Extracted internal standards (NIS) were then added to all samples/QC.

ANALYTICAL CONDITIONS

Extracts were then analyzed by EPA 1633A using an Infinity 1290 LC interfaced to an Agilent 6495 triple quadrupole Mass Spectrometer using Mass Hunter software. All preparation and analyses were performed by AEL².

RESULTS AND DISCUSSION

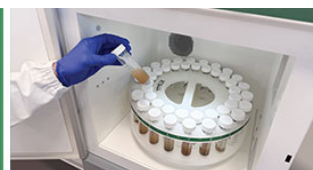
After sample preparation by Microwave extraction using the ETHOS X – XTR-44 system as described previously, the lab for this study provided all analytical data. Table 1 below summarizes the results of the shaker table extraction versus the triplicate microwave extraction data.

The data obtained by applying the same method to the reference material (PT soil) are reported in Table 2.

Table 1 Comparison of Sample Extractions using EPA 1633 Shaker Table vs. Microwave Extraction						
Data Reported to MDL (listed as < values) and J-Flagged ¹ between MDL and verified LOQ						
	Shaker Table Data	Microwave Data	Shaker Table Data	Microwave Data	Shaker Table Data	Microwave Data
	2509157004 Sample	2509157004 Sample	J2509157018 Sample	J2509157018 Sample	J2507676006 Sample	J2507676006 Sample
Target Compounds	µg/kg, dry	µg/kg, dry	µg/kg, dry	µg/kg, dry	µg/kg, dry	µg/kg, dry
11Cl- PF3OUdS	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
9Cl-PF3ONS	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
ADONA	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
4:2FTS	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
6:2FTS	<0.58	<0.51	<0.44	<0.44	<0.74	<0.74

APPLICATION REPORT

ETHOS X – PFAS – EPA Method 1633

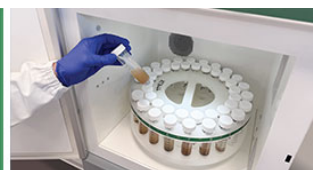


8:2FTS	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
HFPO-DA	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
NFDHA	<0.49	<1.0	<0.73	<0.73	<1.2	<1.2
PFBS	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
PFBA	0.93 J*	0.768 J*	0.89 J*	0.880 J*	<0.62	<0.62
PFDA	0.80 J*	0.982 J*	0.41 J*	0.369 J*	<0.62	<0.62
PFDS	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
PFD _o A	0.92 J*	1.03 J*	<0.36	<0.36	<0.62	<0.62
PFHpS	<0.68	<0.71	<0.51	<0.51	<0.87	<0.87
PFHpA	0.96 J*	0.747 J*	1.5	1.3	<0.62	<0.62
PFHxS	<0.49	<0.51		<0.36	9.1	11.3
PFHxA	0.67 J*	0.573 J*	1.6	1.5	1.10 J*	1.15 J*
PFMBA	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
PFMPA	<0.49	<0.51	<0.36	<0.36	<0.62	<0.62
PFNA	1.1 J*	0.815 J*	0.54 J*	0.419 J*	<0.62	<0.62
PFOS	21	20.7	38	34.5	53	57.7
PFOA	3.6	3.14	3.2	3.03	1.3	1.38
PFPeS	<0.49	<0.51	<0.36	<0.36	0.66 J*	0.799 J*
PFPeA	0.63 J*	0.596 J*	1.4	1.5	<0.62	<0.62
PFUnA	<0.49	1.15 J*	< 0.36	< 0.36	<0.62	<0.62
PFEESA	<0.49	<0.51	< 0.36	< 0.36	<0.62	<0.62

	Shaker Table Data	Microwave Data	Shaker Table Data	Microwave Data	Shaker Table Data	Microwave Data
	250915700 4 Sample	250915700 4 Sample	J250915701 8 Sample	J250915701 8 Sample	J250767600 6 Sample	J250767600 6 Sample
Target Compound s	µg/kg, dry	µg/kg, dry	µg/kg, dry	µg/kg, dry	µg/kg, dry	µg/kg, dry
PFNS	<0.78	<0.81	<0.58	<0.58	<0.99	<0.99
PFOSA	<0.49	<0.51	< 0.36	< 0.36	<0.62	<0.62
PFTeDA	<0.49	0.542 J*	< 0.36	< 0.36	<0.62	<0.62
PFT _r DA	<0.49	0.666 J*	< 0.36	< 0.36	<0.62	<0.62
NEtFOSAA	<0.49	<0.51	< 0.36	< 0.36	<0.62	<0.62
NMeFOSAA	<0.49	<0.51	< 0.36	< 0.36	<0.62	<0.62
PFD _o S	<0.97	<1.0	1.1 J*	<0.73	<1.2	<1.2
NMeFOA	<0.49	<0.51	< 0.36	< 0.36	<0.62	<0.62
NEtFOA	<0.49	<0.51	< 0.36	< 0.36	<0.62	<0.62
NMeFOSE	<4.9	<5.1	<3.6	<3.6	<6.2	<6.2
NEtFOSE	<4.9	<5.1	<3.6	<3.6	<6.2	<6.2
3:3FTCA	<1.9	<2.0	>1.5	>1.5	<2.5	<2.5
5:3FTCA	<9.7	<10.0	<7.3	<7.3	<12	<12
7:3FTCA	<9.7	<10.0	<7.3	<7.3	<12	<12

APPLICATION REPORT

ETHOS X – PFAS – EPA Method 1633



"J" indicates the results greater than the MDL and less than the Reporting limit

Table 2 EPA 1633 Microwave Extraction Data on reference soil (PT sample)

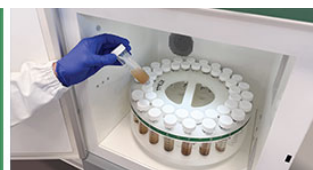
PFAS Compound-EPA 1633 40 compound list	<i>Certified Value</i>	EPA 1633A Microwave	Acceptance Range ¹
	µg/kg	µg/kg	µg/kg
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	<2.50	<2.5	ND ²
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	17.9	20.4	8.95 - 26.8
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	24.9	30.4	12.4 - 37.3
N-Ethylperfluorooctanesulfonamide (NEtFOSA)	30.4	34.5	15.2 - 45.5
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	36.0	40.7	18.0 - 53.9
N-Ethylperfluorooctanesulfonamido ethanol (NEtFOSE)	32.8	34.8	16.4 - 49.1
Hexafluoropropylene oxide dimer acid (HFPO-DA)	37.6	39.9	18.8 - 56.2
N-Methylperfluorooctanesulfonamide (NMeFOSA)	32.8	45.4	16.4 - 49.1
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	32.0	33.1	16.0 - 47.9

Table 2 EPA 1633 Microwave Extraction Data on reference soil (PT sample)

PFAS Compound-EPA 1633 40 compound list	<i>Certified Value</i>	EPA 1633A Microwave	Acceptance Range ¹
	µg/kg	µg/kg	µg/kg
N-Methylperfluorooctanesulfonamidoethanol (NMeFOSE)	33.6	39.6	16.8 - 50.3
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	18.0	21.9	9.00 - 26.9
1H, 1H, 2H, 2H-Perfluorodecanesulfonic acid (8:2 FTS)	47.9	54.6	24.0 - 71.7
1H, 1H, 2H, 2H-Perfluorohexanesulfonic acid (4:2 FTS)	33.6	37.2	16.8 - 50.3
1H, 1H, 2H, 2H-Perfluorooctanesulfonic acid (6:2 FTS)	47.4	52.5	23.7 - 70.9
2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA)	48.0	33.8	24.0 - 71.8
2H, 2H, 3H, 3H-perfluorohexanoic acid (3:3 FTCA)	10.4	7.1	5.20 - 15.6
2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA)	<2.50	<2.5	ND
Perfluorobutanesulfonic acid (PFBS)	16.3	19.7	8.15 - 24.4
Perfluorobutanoic acid (PFBA)	21.6	24.3	10.8 - 32.3
Perfluorodecane sulfonic acid (PFDS)	9.45	10.4	4.72 - 14.1
Perfluorodecanoic acid (PFDA)	12.4	14.7	6.20 - 18.6
Perfluorododecanesulfonic acid (PFDoS)	19.4	19.0	9.70 - 29.0
Perfluorododecanoic acid (PFDoA)	<2.50	<2.5	ND

APPLICATION REPORT

ETHOS X – PFAS – EPA Method 1633



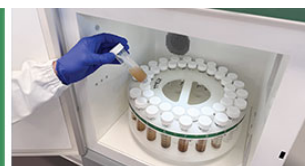
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	12.1	14.4	6.05 - 18.1
Perfluoroheptane sulfonic acid (PFHpS)	13.0	14.9	6.50 - 19.4
Perfluoroheptanoic acid (PFHpA)	39.2	45.5	19.6 - 58.6
Perfluorohexanesulfonic acid (PFHxS)	24.0	26.6	12.0 - 35.9
Perfluorohexanoic acid (PFHxA)	12.8	15.5	6.40 - 19.1
Perfluoro-4-methoxybutanoic acid (PFMBA)	25.6	33.1	12.8 - 38.3
Perfluoro-3-methoxypropanoic acid (PFMPA)	38.4	45.8	19.2 - 57.4
Perfluorononane sulfonic acid (PFNS)	16.2	18.0	8.10 - 24.2
Perfluorononanoic acid (PFNA)	26.4	34.0	13.2 - 39.5
Perfluorooctanesulfonamide (PFOSA)	14.4	20.2	7.20 - 21.5
Perfluorooctanesulfonic acid (PFOS)	21.5	21.6	10.8 - 32.2
Perfluorooctanoic acid (PFOA)	16.8	24.9	8.40 - 25.1
Perfluoropentane sulfonic acid (PFPeS)	45.1	53.7	22.6 - 67.5
Perfluoropentanoic acid (PFPeA)	<2.50	<2.5	ND
Perfluorotetradecanoic acid (PFTDA)	21.6	22.8	10.8 - 32.3

Table 2 EPA 1633 Microwave Extraction Data on reference oil (PT sample)

PFAS Compound-EPA 1633 40 compound list	<i>Certified Value</i>	EPA 1633A Microwave	Acceptance Range ¹	
	µg/kg	µg/kg	µg/kg	
Perfluorotridecanoic acid (PFTrDA)	40.0	49.7	20.0 - 59.8	
Perfluoroundecanoic acid (PFUnA)	8.40	10.1	4.20 - 12.6	
Ranges were determined by Pt provider – ERA- based upon shaker table extraction, not microwave ND = not detected down to RL-<PTRL				

APPLICATION REPORT

ETHOS X – PFAS – EPA Method 1633



CONCLUSION

The Milestone ETHOS X platform with XTR-rotor and integrated stirring successfully processed solid matrices with excellent comparability, precision, and PT performance. All data were well within method requirement, and the studies clearly demonstrated the superior performance of the ETHOS X platform for EPA 1633A target analytes.

Compared with shaker table extraction, the ETHOS X configuration offers laboratories a reliable path to improve the sample preparation steps for PFAS analysis saving significant time and error prone handling steps.

A summary of the key benefits the Milestone ETHOS X – XTR solution brings to the lab include:

- Increased Capacity and ability to execute rush soil work
- Less labor and elapsed time - doing more with less- 15 mins. vs. hours
- Ability to improve and better predict analysis turn-around times
- Improved data quality, consistency and confidence in the results
- More robust extraction given varying matrices received by the lab
- Easier workflow implementation and on-boarding of new staff members.

REFERENCES

- 1- EPA 1633 Revision A Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS
<https://www.epa.gov/system/files/documents/2024-12/method-1633a-december-5-2024-508-compliant.pdf>
- 2- Advanced Environmental Laboratories, Inc (AEL), Jacksonville, FL an independent laboratory approved for EPA 1633 PFAS under NELAC/TNI and DOD QSM programs.

ABOUT MILESTONE

At Milestone we help chemists by providing the most innovative technology for metals analysis, direct mercury analysis and the application of microwave technology to extraction, ashing and synthesis. Since 1988 Milestone has helped chemists in their work to enhance food, pharmaceutical and consumer product safety, and to improve our world by controlling pollutants in the environment.