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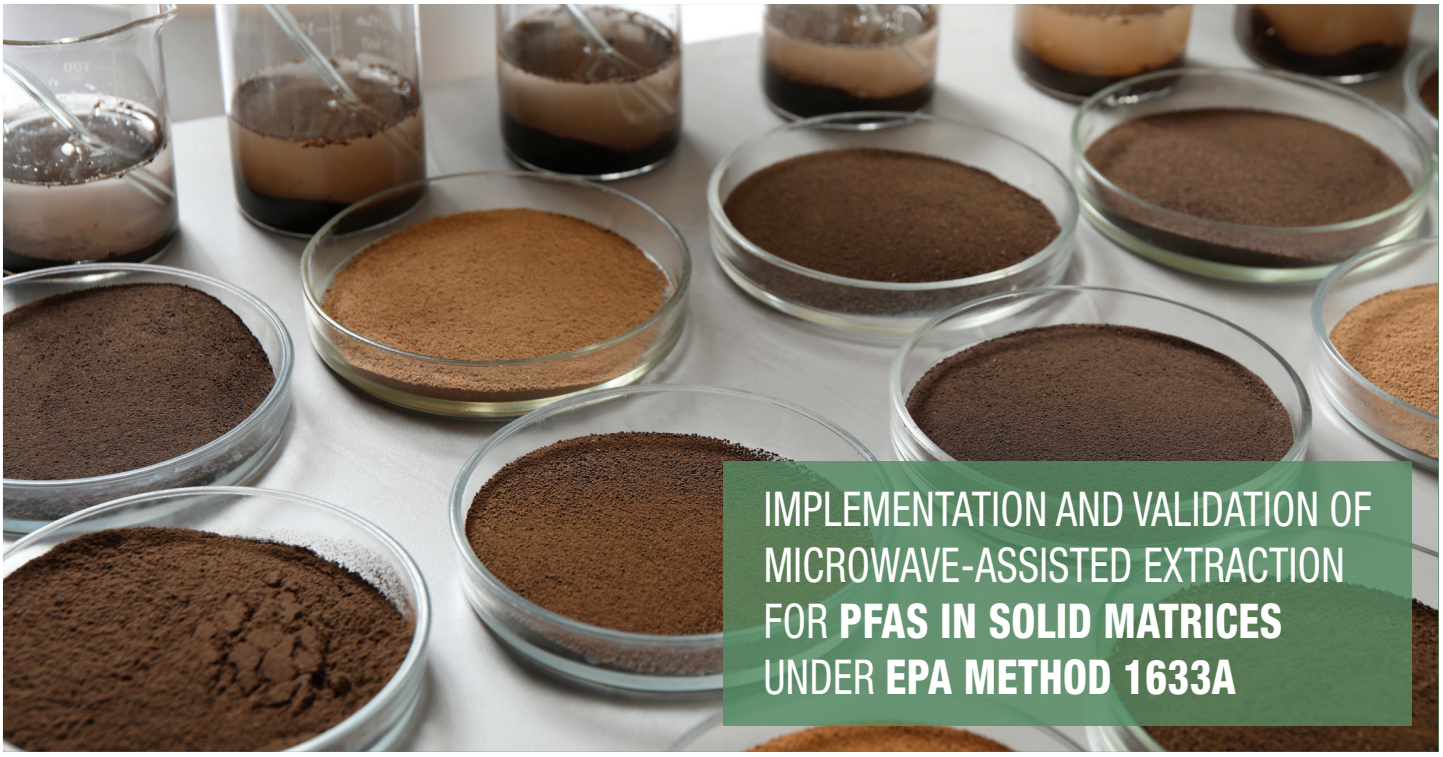


IMPLEMENTATION AND VALIDATION OF MICROWAVE-ASSISTED EXTRACTION FOR **PFAS IN SOLID MATRICES** UNDER **EPA METHOD 1633A**

ABSTRACT

This white paper presents the successful implementation of a microwave-assisted extraction (MAE) system for PFAS sample preparation in solid matrices by Advanced Environmental Laboratories, Inc. (AEL). The study compares MAE with the conventional shaker table extraction specified in EPA Method 1633A, demonstrating how microwave extraction significantly reduces total extraction time, minimizes manual handling, and improves recoveries for challenging PFAS compounds. Operating at 65 °C with active stirring for 15 minutes, the MAE approach enables the simultaneous processing of up to 44 samples per batch. This represents a faster, more consistent, and higher-throughput alternative to the 3-hour shaker table procedure, while maintaining full compliance with Method 1633A quality assurance requirements.

Beyond time savings, microwave extraction has proven effective across a wider range of solid matrices, including soils, biosolids, and tissues, where the shaker table method often fails to achieve robust extraction performance.

INTRODUCTION

Per- and polyfluoroalkyl substances (PFAS) are a large family of synthetic compounds widely used since the 1940s for their resistance to heat, water, and chemicals. These same properties have made PFAS valuable in industrial and consumer applications, such as coatings, textiles, and packaging, but also highly persistent in the environment. As a result, PFAS have become a major focus of global regulatory action.

In response, the U.S. Environmental Protection Agency (EPA) released Method 1633A in December 2024, establishing a standardized procedure for the determination of PFAS in soils, biosolids, tissues, and aqueous samples. While robust, the method specifies a three-hour extraction using ammoniacal methanol, a process involving multiple manual transfers that limit throughput and reproducibility. Environmental laboratories analyzing PFAS in solid matrices face growing workloads and increasingly stringent data quality objectives.

The extraction prescribed by EPA Method 1633A often becomes a bottleneck, its long duration and manual handling steps limiting throughput and reproducibility.

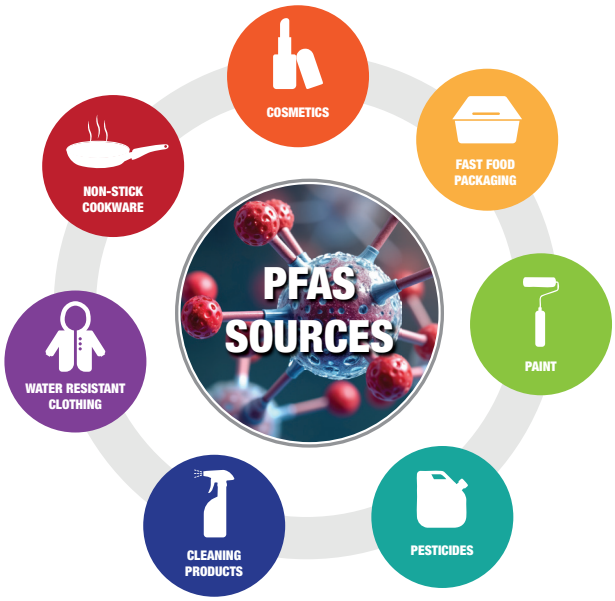
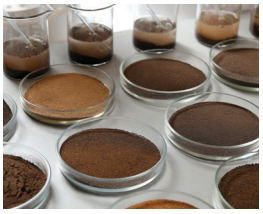


Figure 1. PFAS in products

IMPLEMENTATION AND VALIDATION OF MICROWAVE-ASSISTED EXTRACTION FOR **PFAS IN SOLID MATRICES** UNDER **EPA METHOD 1633A**



To overcome these challenges, Advanced Environmental Laboratories (AEL) validated a microwave-assisted extraction (MAE) method developed by Milestone, designed to accelerate PFAS sample preparation for soils, biosolids, and tissues while maintaining full compliance with EPA Method 1633A performance requirements.

The following sections summarize the validation study and comparative results, demonstrating how MAE delivers faster, more consistent, and fully compliant PFAS extractions from solid matrices.

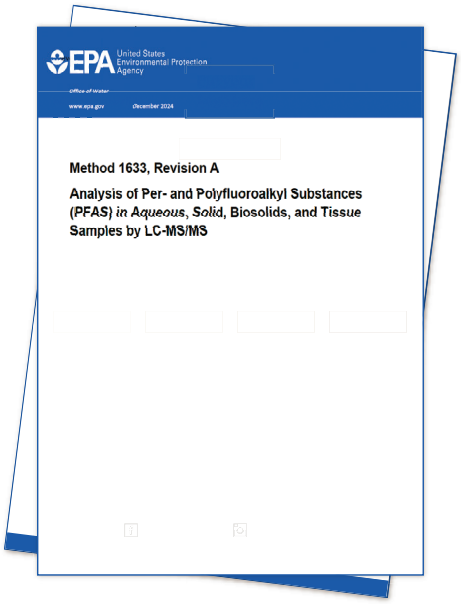


Figure 2. EPA Method 1633A

TECHNICAL BACKGROUND

EPA Method 1633A – Shaker Table Extraction

EPA Method 1633A describes the extraction of PFAS from solid matrices using a shaker table and 0.3% ammoniacal methanol. A 2.5 g (biosolids) or 5 g (soil/tissue) sample is weighed into a 50 mL polypropylene centrifuge tube, spiked with isotopically labeled standards, and allowed to equilibrate for 30 minutes. The sample is then extracted three consecutive times on the shaker table at 200 rpm, each cycle lasting 30 minutes, followed by centrifugation and decanting of the supernatant after every extraction.

This process involves approximately 30 mL of solvent and takes around three hours per batch of 20 samples plus QC. Besides being time-consuming, it requires multiple manual transfers, increasing the risk of contamination or analyte loss. Furthermore, the limited mechanical agitation of a 50 mL conical tube often results in incomplete solvent penetration, particularly for cohesive matrices such as clays, biosolids, or tissues. As a result, recoveries may vary widely depending on matrix type, making this method unsuitable for high-throughput or complex sample workflows.

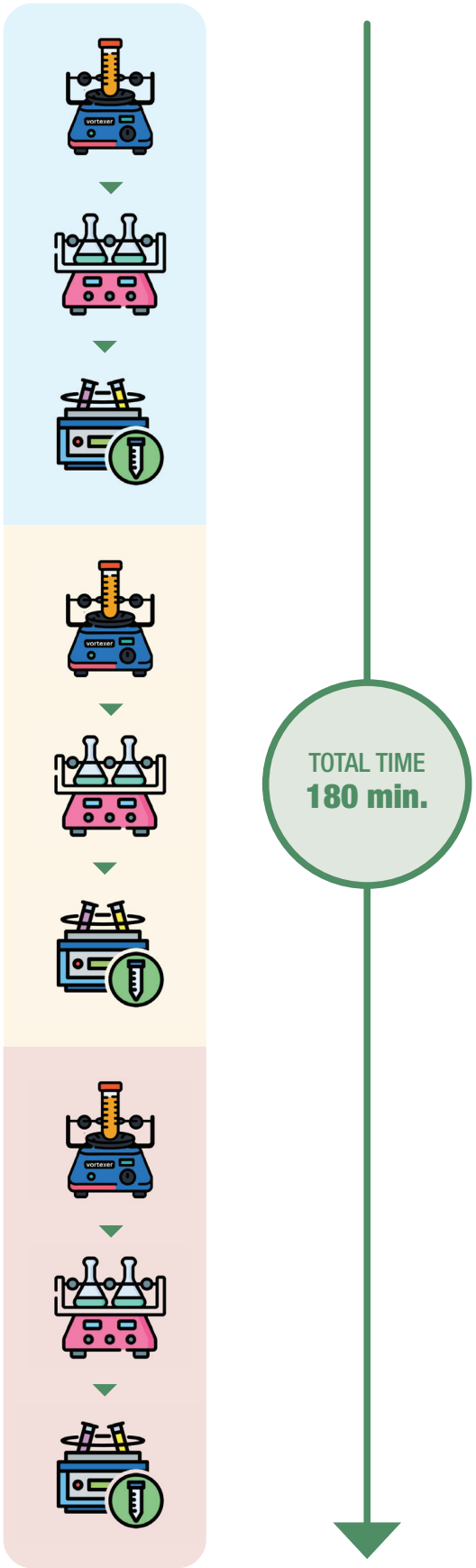
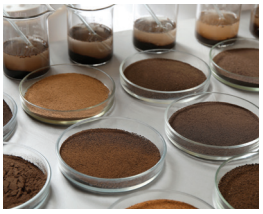


Figure 3. 2 EPA 1633A Extraction Steps

IMPLEMENTATION AND VALIDATION OF MICROWAVE-ASSISTED EXTRACTION FOR **PFAS IN SOLID MATRICES** UNDER **EPA METHOD 1633A**



EPA Method 1633A – Shaker Table Extraction

The Milestone ETHOS X system, equipped with the XTR-44 rotor, offers a controlled microwave-assisted extraction process that overcomes these limitations. Samples are heated to 65 °C in closed 50 mL polypropylene tubes containing 0.3% ammoniacal methanol, while active stirring promotes homogeneous solvent contact and efficient desorption of PFAS from the solid matrix.

This combination of temperature and agitation significantly enhances solvent diffusivity and extraction kinetics, enabling complete extraction within 15 minutes, followed by a brief 5-minute cooling step. After cooling, samples are centrifuged and decanted as required by Method 1633A.

The system can process up to 44 samples and QC per batch, resulting in approximately 75% time savings compared to the shaker table method. The microwave-assisted approach also improves reproducibility and robustness, particularly for difficult matrices such as biosolids or organic-rich soils, where conventional shaking often underperforms.

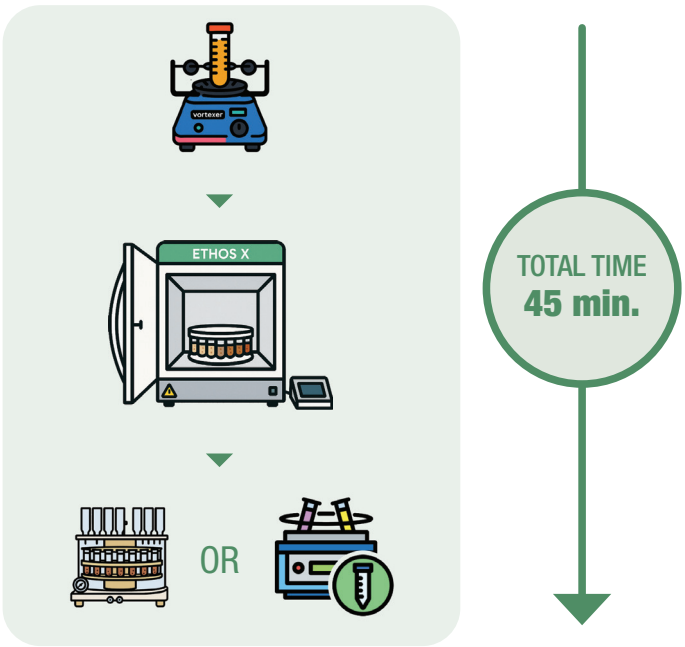


Figure 4. EPA 1633A Microwave-Assisted Extraction

EXPERIMENTAL

Validation Procedure

To validate the microwave-assisted extraction (MAE) as an alternative to the shaker table procedure, a comparative study was performed following the quality assurance framework outlined in EPA Method 1633A, Section 9.1.

According to section 9.2.1 of the method, laboratories may introduce modifications to the extraction, concentration, or cleanup steps provided that equivalent precision and recovery are demonstrated and all performance criteria are met.

The validation included the following steps:

- Initial Precision and Recovery (IPR) with method blank
- Low-Level Ongoing Precision and Recovery (LLOPR) for LOQ verification
- Batch analyses including blank, LLOPR, OPR, MS, MSD, and samples for comparison
- Method Detection Limit (MDL) studies (not performed here, as MDLs are laboratory-specific)

For reproducibility assessment, three samples were extracted in triplicate using both the shaker table and the microwave-assisted approach. The evaluation criteria followed Method 1633A Tables 7.0 and 8.0, which define recovery and precision limits for target PFAS and extracted internal standards (EIS).

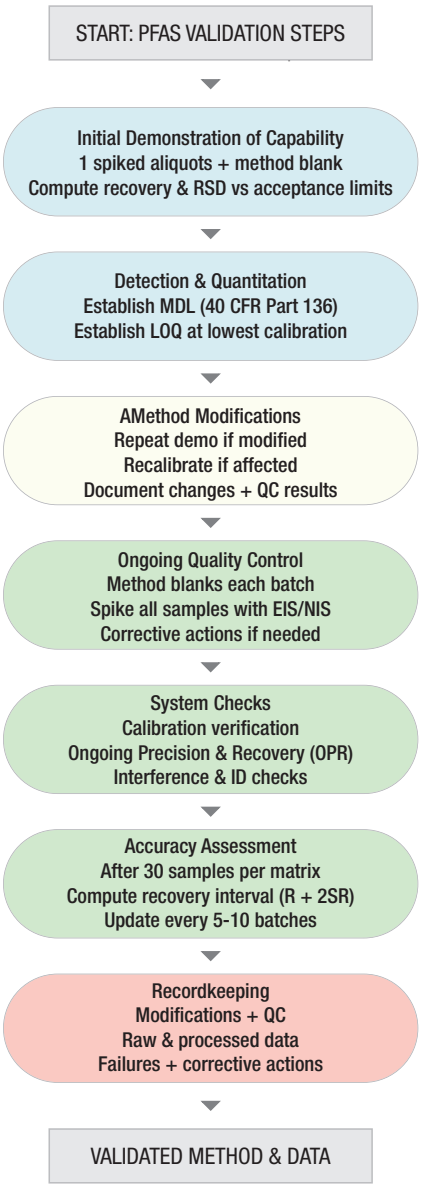
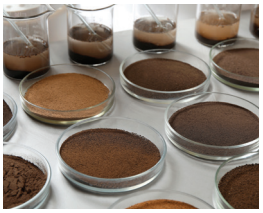


Figure 5. Quality assurance flow outlined in EPA Method 1633A



Extraction Procedure

Shaker Table Extraction

Following Method 1633A, 5 g of Ottawa sand (for blanks and QC samples) or soil were weighed into 50 mL polypropylene centrifuge tubes, spiked with extracted internal standard (EIS), and allowed to stand for 30 minutes.

The samples were extracted three consecutive times using 0.3% ammoniacal methanol (10 mL + 15 mL + 5 mL). Each extraction consisted of 30 minutes of shaking at 200 rpm, followed by centrifugation at 3000 rpm for 10 minutes and decanting of the supernatant into a clean tube.

Microwave-Assisted Extraction (MAE)

For MAE, 5 g of sample were weighed into 50 mL polypropylene tubes, spiked with extracted internal standard (EIS), and allowed to stand for 30 minutes. After adding 15 mL of 0.3% ammoniacal methanol and a rare-earth stirring bar (uncoated NdFeB), the tubes were placed into the XTR-44 rotor and then into the ETHOS X.

The microwave program consisted of a 5-minute ramp to 65 °C, followed by a 10-minute hold with active stirring. After a brief 5-minute cooling, samples were centrifuged at 3000 rpm for 10 minutes and the supernatant transferred to clean tubes for further processing.

Figure 6. Milestone ETHOS™ X

Post-Extraction Processing

Both extraction types followed the same downstream procedure:

Activated carbon treatment, solvent concentration at 60 °C (Organomation™ nitrogen evaporator) to a volume <10 mL, and dilution to 50 mL with PFAS-free water.

The final extracts underwent solid-phase extraction (SPE) cleanup using Phenomenex Strata-AW 33 µm PFAS Weak Anion Exchange cartridges. Extracts were then spiked with Non-Extracted Internal Standards (NIS) and transferred to autosampler vials for LC/MS-MS analysis.

LC/MS-MS Analysis

Extracts were analyzed on an Agilent 1290/6495C LC/MS-MS equipped with a Advanced Materials Technology (AMT) HALO 90 A, PFAS, 2.7um, 2.1X100 mm analytical column and an AMT guard column, HALO, 90A, C18, 2.1X5mm with the AMT HALO, 160A, and PFAS 2.7um, 3.0X50mm delay column.

4.0 uL of the extract was injected. The acquisition method was 0.4ml/min using this gradient: 0min – 98% A/2% B; 0.1min – 98% A/2% B; 18min – 10% A/90% B; 21min – 10% A/90% B; 22min – 98% A/2% B; 29min – 98% A/2% B. A mobile phase is 95/5 2mM ammonium acetate/LCMS acetonitrile; B mobile phase is LC/MS acetonitrile. Data processing was performed using Mass Hunter software.

A sample TIC-MRM scan is shown below in Figure 3.0 with a run time of approx. 16 minutes.

RESULTS & COMPARISON DATA

The validation study demonstrated that the microwave-assisted extraction (MAE) approach fully meets the precision, recovery, and reproducibility requirements established in EPA Method 1633A.

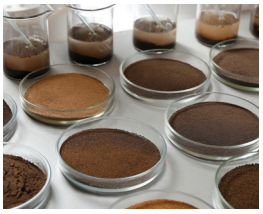
All performance criteria were satisfied using the Milestone ETHOS X system equipped with the XTR-44 rotor, confirming that MAE provides a reliable and compliant alternative to the shaker table extraction.

The following datasets summarize the validation outcomes:

- Table 1 – Initial Precision and Recovery (IPR) for target PFAS compounds compared to EPA 1633A limits
- Table 2 – Recoveries of extracted internal standards (EIS) according to EPA 1633A acceptance criteria
- Table 3 – Reproducibility results for three real soil samples analyzed in triplicate
- Table 4 – Comparative results between shaker table and microwave extraction methods for representative samples
- Table 5 – Matrix spike (MS) and matrix spike duplicate (MSD) recoveries
- Table 6 – Recovery data for Certified Reference Material (CRM) – soil

Across all studies, microwave extraction achieved recoveries and precision fully within the method's acceptance limits, with improved consistency across replicates and higher recoveries for strongly adsorbed PFAS such as PFTeDA and PFOSA.

These findings confirm the robustness of the MAE process and its suitability for a wide variety of solid matrices.



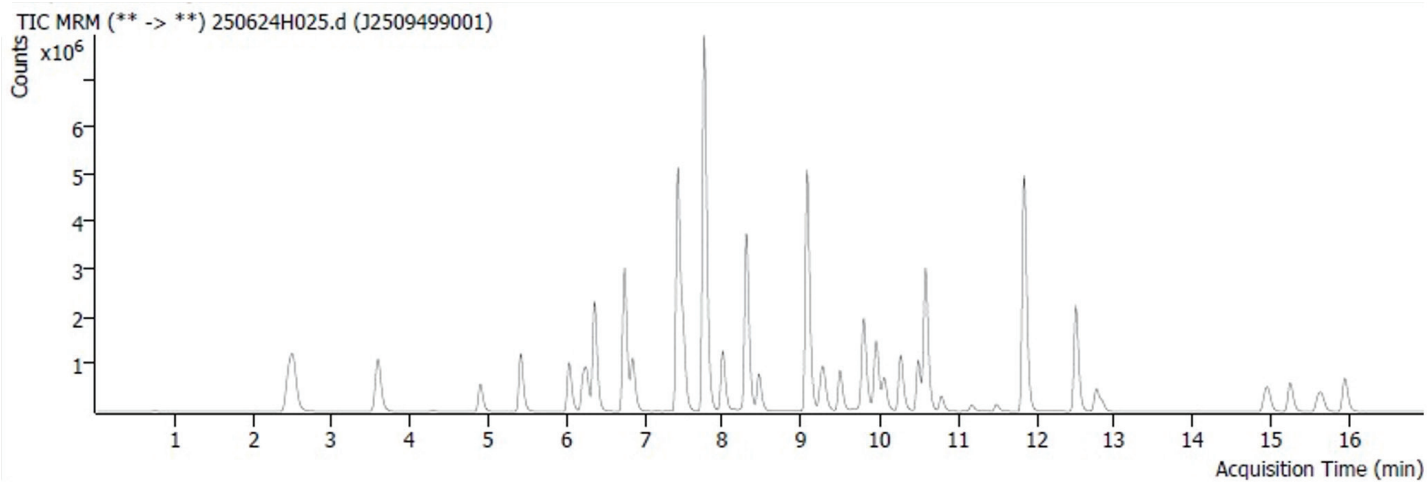


Figure 7. TIC-MRM scan

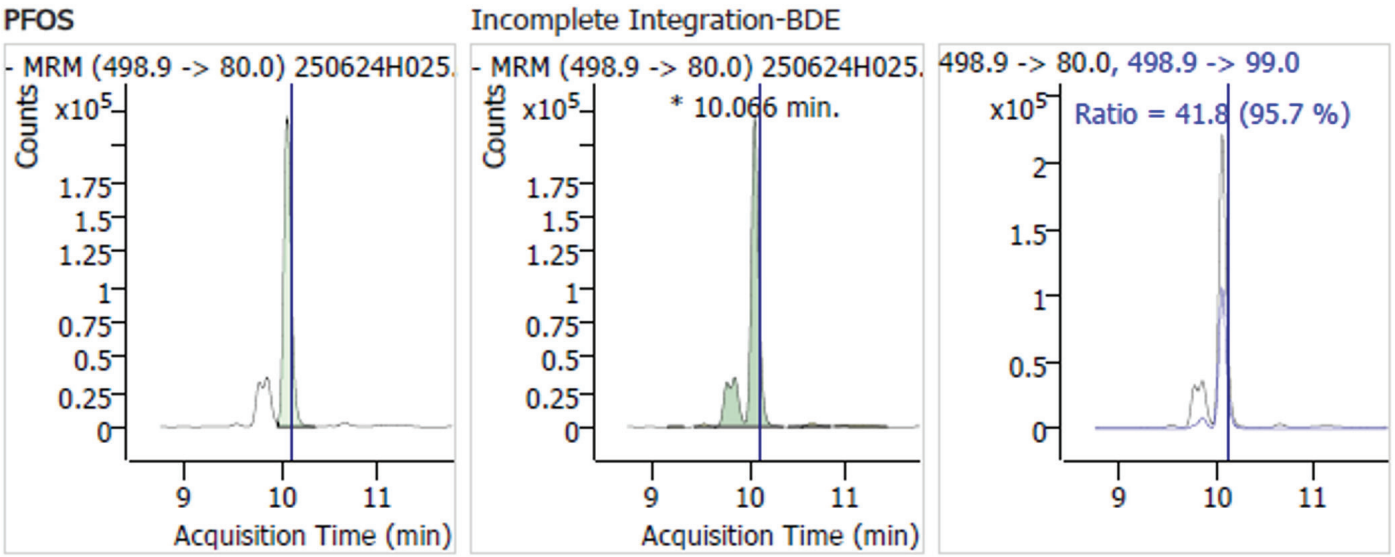


Figure 8. PFOS branched and linear isomers

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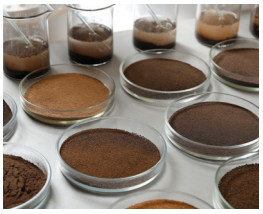
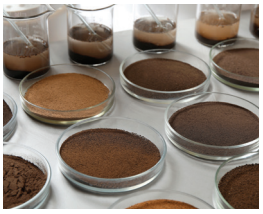


Table 1. Initial Precision and Recovery (IPR) for target PFAS compounds compared to EPA 1633A limits

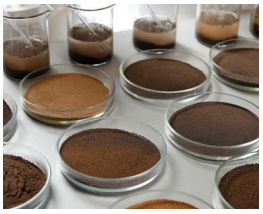
PFAS Compound	*ug/Kg*	*% Recovery*					EPA 1633A Table 7 Limits		
	Method Blank, Reported to MDL	IPR-1	IPR-2	IPR-3	IPR-4	Avg % R	% RSD	Control Limits %R	Control Limits % RSD
11Cl-PF3OUdS	<0.5	91	102	84	72	87	14,4	45-160	17
9Cl-PF3ONS	<0.5	87	106	104	106	101	9,1	70-150	26
ADONA	<0.5	105	113	112	114	111	3,7	70-160	23
4:2 FTS	<0.5	113	110	109	112	111	1,6	60-150	21
6:2 FTS	<0.5	114	105	115	105	110	5,0	55-200	23
8:2 FTS	<0.5	107	118	121	109	114	6,0	70-150	24
HFPO-DA	<0.5	111	112	115	115	113	1,8	70-145	26
NFDHA	<1.0	127	127	129	126	127	1,0	60-155	26
PFBS	<0.5	98	106	107	102	103	4,0	65-145	25
PFBA	<0.5	110	112	112	113	112	1,1	70-140	26
PFDA	<0.5	126	124	124	115	122	4,0	70-155	24
PFDS	<0.5	112	102	90	87	98	11,8	40-155	25
PFDoA	<0.5	120	121	122	132	124	4,5	70-150	29
PFHpS	<0.5	122	117	117	113	117	3,1	65-155	28
PFHpA	<0.5	109	111	115	119	114	3,9	65-145	27
PFHxS	<0.5	101	106	102	107	104	2,8	60-150	27
PFHxA	<0.5	120	121	121	119	120	0,8	65-140	27
PFMBA	<0.5	120	124	124	125	123	1,8	60-150	31
PFMPA	<0.5	121	124	125	126	124	1,7	30-140	40
PFNA	<0.5	127	126	130	126	127	1,5	70-155	27
PFOS	<0.3	111	105	106	108	108	2,5	65-160	50
PFOA	<0.15	116	117	121	118	118	1,8	70-150	27
PFPeS	<0.5	118	117	119	124	120	2,6	55-160	19
PFPeA	<0.5	117	120	120	121	120	1,4	60-150	26
PFUnA	<0.5	114	117	121	122	119	3,1	70-155	19
PFEESA	<0.5	108	109	106	108	108	1,2	70-140	31
PFNS	<0.5	110	115	108	112	111	2,7	55-140	31
FOSA	<0.5	128	125	124	125	126	1,4	70-140	19
PFTeDA	<0.5	103	103	112	108	107	4,1	65-150	17



PFTrDA	<0.5	121	121	98	116	114	9,6	65-150	25
NEtFOSAA	<0.5	110	124	125	121	120	5,7	65-165	26
NMeFOSAA	<0.5	116	123	131	121	123	5,1	65-155	25
PFDoS	<1.0	94	72	78	81	81	11,4	25-160	33
NMeFOSA	<0.5	143	144	144	139	143	1,7	70-155	27
NEtFOSA	<0.5	112	108	114	113	112	2,4	70-140	23
NMeFOSE	<5.0	103	105	104	104	104	0,8	70-140	31
NEtFOSE	<5.0	104	106	107	106	106	1,2	70-135	20
3:3FTCA	<2.0	108	108	111	110	109	1,4	45-130	32
5:3FTCA	<10.0	105	106	104	109	106	2,0	60-130	28
7:3FTCA	<10.0	89	103	85	97	94	8,6	60-150	39

Table 2. Recoveries of extracted internal standards (EIS) according to EPA 1633A acceptance

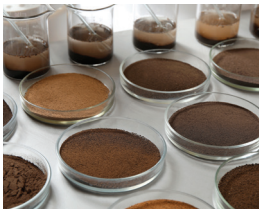
PFAS EIS Isotopes	***ug/Kg***	***% Recovery***					**EPA 1633A Table 8 Limits**		
	Method Blank, Reported to MDL	**IPR-1**	**IPR-2**	**IPR-3**	**IPR-4**	**Avg % R**	**% RSD**	**Control Limits %R**	**% RSD**
Extracted Internal Standards (EIS)									
13C4-PFBA	86	93	95	86	92	92	4,2	10-130	NA
13C5-PFPEA	84	93	93	85	91	91	4,2	35-130	NA
13C3-PFBS	84	91	92	85	90	90	3,5	40-135	NA
13C2-4:2FTS	73	71	77	70	73	73	4,3	40-165	NA
13C5-PFHXA	83	89	93	86	91	90	3,3	40-130	NA
13C4-PFHPA	85	94	96	88	91	92	3,8	40-130	NA
13C3-PFHXS	83	80	85	81	80	82	2,9	40-130	NA
13C2-6:2FTS	78	72	82	73	79	77	6,3	40-215	NA
13C8-PFOA	86	84	93	82	86	86	5,6	40-130	NA
13C9-PFNA	81	74	91	75	85	81	10,1	40-130	NA
13C8-PFOS	101	83	100	93	95	93	7,7	40-130	NA
13C2-8:2FTS	83	70	77	73	78	75	5,0	40-275	NA
13C6-PFDA	81	70	84	76	86	79	9,4	40-130	NA
13C7-PFUNA	88	75	83	69	64	73	11,2	40-130	NA
13C3-HFPO-DA	85	95	92	86	90	91	4,2	40-130	NA



13C2-PFDOA	79	75	67	73	68	71	5,5	40-130	NA
D3-NMEFOSAA	79	69	81	67	62	70	11,6	40-135	NA
D5-NETFOSAA	65	61	70	66	63	65	6,0	40-150	NA
13C8-FOSA	84	76	89	82	81	82	6,5	40-130	NA
13C2-PFTEDA	66	72	66	69	71	70	3,8	20-130	NA
D3-NMEFOSA	77	74	79	74	74	75	3,3	10-130	NA
D5-NETFOSA	78	66	80	73	71	73	8,0	10-130	NA
D7-NMEFOSE	77	74	80	76	74	76	3,7	20-130	NA
D9-NETFOSE	79	72	81	75	75	76	5,0	15-130	NA

Table 3. Reproducibility Study on Actual Soil Samples- Detected Compounds only

Target Compounds	Sample J2509157004 49.7 % Moisture			Sample J2509157018 33.7% Moisture			Sample J2507676006 60.4% Moisture		
	Sample	Sample Dup	Sample Dup 2	Sample	Sample Dup	Sample Dup 2	Sample	Sample Dup	Sample Dup 2
	ug/kg, dry	*ug/kg, dry*	*ug/kg, dry*	*ug/kg, dry*	*ug/kg, dry*	*ug/kg, dry*	*ug/kg, dry*	*ug/kg, dry*	*ug/kg, dry*
11Cl-PF3OUdS	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
9Cl-PF3ONS	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
ADONA	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
4:2FTS	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
6:2FTS	<0.51	<0.50	<0.51	<0.45	<0.44	<0.44	<0.74	<0.79	<0.76
8:2FTS	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
HFPO-DA	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
NFDHA	<1.0	<0.50	<1.0	<0.75	<0.74	<0.73	<1.2	<1.3	<1.3
PFBS	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
PFBA	0.731 J	0.717 J	0.768 J	0.803 J	0.765 J	0.880 J	<0.62	<0.66	<0.63
PFDA	0.914 J	1.10 J	0.982 J	< 0.38	< 0.37	0.369 J	<0.62	<0.66	<0.63
PFDS	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
PFDoA	1.01 J	0.914 J	1.03 J	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
PFHpS	<0.71	<0.70	<0.71	<0.53	<0.52	<0.51	<0.87	<0.92	<0.88
PFHpA	0.694 J	0.546 J	0.747 J	1.02 J	1.12 J	1.28 J	<0.62	<0.66	<0.63
PFHxS	<0.51	<0.50	<0.51	< 0.38	<0.37	<0.36	11,3	11,5	11,4
PFHxA	0.639 J	0.521 J	0.573 J	1.26 J	1.31 J	1,5	1.15 J	1.07 J	1.22 J



PFMBA	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
PFMPA	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	<0.62	<0.66	<0.63
PFNA	0.877 J	0.798 J	0.815 J	< 0.38	< 0.37	0.419 J	<0.62	<0.66	<0.63
PFOS	21,5	21,0	20,7	25,3	33,5	34,5	57,7	57,2	56,5
PFOA	2,89	2,35	3,14	2,51	2,74	3,03	1,38	1,38	1,37
PFPeS	<0.51	<0.50	<0.51	< 0.38	< 0.37	<0.36	0.799 J	<0.66	0.709 J
PFPeA	0.575 J	0.515 J	0.596 J	1.30 J	1.26 J	1,5	<0.62	<0.66	<0.63
PFUnA	1.13 J	1.03 J	1.15 J	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
PFEESA	<0.51	<0.50	<0.51	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
PFNS	<0.81	<0.80	<0.81	<0.60	<0.59	<0.58	<0.99	<0.66	<0.63
PFOSA	<0.51	<0.50	<0.51	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
PFTeDA	0.526 J	<0.50	0.542 J	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
PFTrDA	0.640 J	0.547 J	0.666 J	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
NEtFOSAA	<0.51	<0.50	<0.51	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
NMeFOSAA	<0.51	<0.50	<0.51	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
PFDoS	<1.0	<0.99	<1.0	<0.75	< 0.37	<0.73	<1.2	<1.3	<1.3
NMeFOSA	<0.51	<0.50	<0.51	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
NEtFOSA	<0.51	<0.50	<0.51	< 0.38	< 0.37	< 0.36	<0.62	<0.66	<0.63
NMeFOSE	<5.1	<5.0	<5.1	<3.8	<3.7	<3.6	<6.2	<6.6	<6.3
NEtFOSE	<5.1	<5.0	<5.1	<3.8	<3.7	<3.6	<6.2	<6.6	<6.3
3:3FTCA	<2.0	<2.0	<2.0	<1.5	<1.5	>1.5	<2.5	<2.6	<2.5
5:3FTCA	<10.0	<9.9	<10.0	<7.5	<7.4	<7.3	<12	<13	<13
7:3FTCA	<10.0	<9.9	<10.0	<7.5	<7.4	<7.3	<12	<13	<13

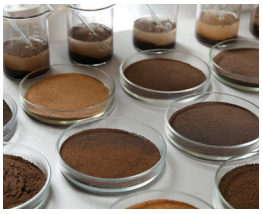
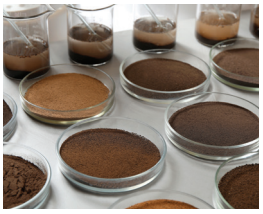


Table 4. Comparison of Sample extractions using EPA 1633 Shaker Table vs. Microwave Extraction

Data Reported to MDL (listed as < values) and J-Flagged1 between MDL and verified LOQ						
Target Compounds	**Shaker Table Data**	**Microwave Data**	**Shaker Table Data**	**Microwave Data**	**Shaker Table Data**	**Microwave Data**
	2509157004 Sample	**2509157004 C Sample Dup 2**	**J2509157018 Sample**	**J2509157018 C Sample Dup 2**	**J2507676006 Sample**	**J2507676006 A Sample**
	ug/kg, dry	*ug/kg, dry*	*ug/kg, dry*	*ug/kg, dry*	*ug/kg, dry*	*ug/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
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
PFDoA	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
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
PFTrDA	<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
PFDoS	<0.97	<1.0	1.1 J	<0.73	<1.2	<1.2
NMeFOSA	<0.49	<0.51	< 0.36	< 0.36	<0.62	<0.62
NEtFOSA	<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

J = estimated value > MDL < LOQ
Note: Values for MDL and LOQ will change per sample based upon Percent moisture.

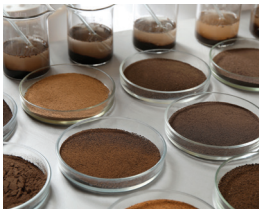
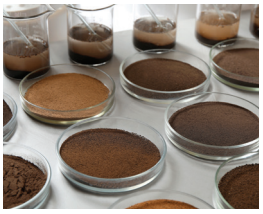


Table 5. Matrix Spike/Matrix Spike Duplicate Data

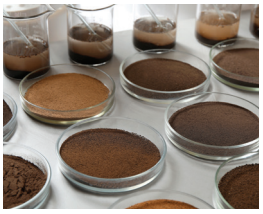
EPA 1633 Compounds	J2509157004 A Sample	J2509157004 A Sample MS	J2509157004 A Sample MSD	Precision for MS/MSD	MS/MSD recovery and Precision Limits	
	ug/kg, dry	*% Recovery*	*% Recovery*	*RPD*	*Recovery*	*RPD*
11Cl-PF3OUdS	<0.51	116	117	2,6	45 - 160	31
9Cl-PF3ONS	<0.51	119	118	2,6	70 - 150	23
ADONA	<0.51	119	115	5,4	70 - 160	26
4:2FTS	<0.51	111	113	0,5	60 - 150	27
6:2FTS	<0.51	112	109	5	55 - 200	50
8:2FTS	<0.51	110	113	0,6	70 - 150	27
HFPO-DA	<0.51	116	114	3,7	70 - 145	25
NFDHA	<1.0	138	135	4,6	60 - 155	27
PFBS	<0.51	115	114	3	65 - 145	25
PFBA	0.731 J	120	116	4,8	70 - 140	17
PFDA	0.914 J	134	117	15	70 - 155	26
PFDS	<0.51	114	109	6,2	40 - 155	31
PFDoA	1.01 J	123	131	3,8	70 - 150	25
PFHpS	<0.71	117	123	3,1	65 - 155	27
PFHpA	0.694 J	119	120	1,2	65 - 145	21
PFHxS	<0.51	131	130	2,7	60 - 150	28
PFHxA	0.639 J	126	127	0,8	65 - 140	23
PFMBA	<0.51	132	132	1,7	60 - 150	23
PFMPA	<0.51	134	134	2,4	30 - 140	25
PFNA	0.877 J	138	129	8	70 - 155	24
PFOS	21,5	112	98	5,2	65 - 160	27
PFOA	2,89	131	132	1,4	70 - 150	23
PFPeS	<0.51	124	124	2,5	55 - 160	29
PFPeA	0.575 J	128	126	3,6	60 - 150	26
PFUnA	1.13 J	141	127	12	70 - 155	26
PFEESA	<0.51	115	112	5,3	70 - 140	20
PFNS	<0.81	113	115	0,2	55 - 140	27
PFOSA	<0.51	131	141	5,2	70 - 140	19
PFTeDA	0.526 J	108	109	1,5	65 - 150	24
PFTrDA	0.640 J	123	123	2,2	65 - 150	28
NEtFOSAA	<0.51	134	137	0,3	65 - 165	31



NMeFOSAA	<0.51	125	126	2,1	65 - 155	31
PFDoS	<1.0	93	100	5,3	25 - 160	40
NMeFOSA	<0.51	147	151	0,6	70 - 155	26
NEtFOSA	<0.51	116	113	4,6	70 - 140	19
NMeFOSE	<5.1	107	105	3,3	70 - 140	19
NEtFOSE	<5.1	111	111	1,7	70 - 135	17
3:3FTCA	<2.0	104	111	4,5	45 - 130	32
5:3FTCA	<10.0	128	126	3,3	60 - 130	28
7:3FTCA	<10.0	150	156	1,9	60 - 150	39

Table 6. Recovery data for Certified Reference Material (CRM) – soil

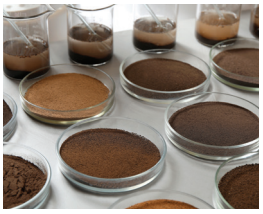
EPA 1633 Microwave Extraction Data	**Results and Acceptance Ranges (PT Limits)**			
PFAS Compound-EPA 1633 40 compound list	**Certified Value**	**EPA 1633A Microwave**	**Acceptance Range[1]**	**Pass/Fail**
	µg/kg	*ug/kg*	*µg/kg*	
11-chloroeicosafluoro-3- oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	<2.50	<0.5	ND[2]	Pass
9-chlorohexadecafluoro-3- oxanonane-1-sulfonic acid (9Cl- PF3ONS)	17,9	20,4	8.95 - 26.8	Pass
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	24,9	30,4	12.4 - 37.3	Pass
N-Ethylperfluorooctanesulfonamide (NEtFOSA)	30,4	34,5	15.2 - 45.5	Pass
N-ethyl perfluorooctanesulfonamidoaceti c acid (NEtFOSAA)	36,0	40,7	18.0 - 53.9	Pass
N-Ethylperfluorooctanesulfonamido ethanol (NEtFOSE)	32,8	34,8	16.4 - 49.1	Pass
Hexafluoropropylene oxide dimer acid (HFPO-DA)	37,6	39,9	18.8 - 56.2	Pass
N-Methylperfluorooctanesulfonami de (NMeFOSA)	32,8	45,4	16.4 - 49.1	Pass
N-methyl perfluorooctanesulfonamidoaceti c acid (NMeFOSAA)	32,0	33,1	16.0 - 47.9	Pass
N-Methylperfluorooctanesulfonami doethanol (NMeFOSE)	33,6	39,6	16.8 - 50.3	Pass
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	18,0	21,9	9.00 - 26.9	Pass
1H, 1H, 2H, 2H-Perfluorodecanesulfonic acid (8:2 FTS)	47,9	54,6	24.0 - 71.7	Pass
1H, 1H, 2H, 2H-Perfluorohexanesulfonic acid (4:2 FTS)	33,6	37,2	16.8 - 50.3	Pass
1H, 1H, 2H, 2H-Perfluorooctanesulfonic acid (6:2 FTS)	47,4	52,5	23.7 - 70.9	Pass
2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA)	48,0	33,8	24.0 - 71.8	Pass
2H, 2H, 3H, 3H-perfluorohexanoic acid (3:3 FTCA)	10,4	7,1	5.20 - 15.6	Pass
2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA)	<2.50	<10	ND	Pass



Perfluorobutanesulfonic acid (PFBS)	16,3	19,7	8.15 - 24.4	Pass
Perfluorobutanoic acid (PFBA)	21,6	24,3	10.8 - 32.3	Pass
Perfluorodecane sulfonic acid (PFDS)	9,45	10,4	4.72 - 14.1	Pass
Perfluorodecanoic acid (PFDA)	12,4	14,7	6.20 - 18.6	Pass
Perfluorododecanesulfonic acid (PFDoS)	19,4	19,0	9.70 - 29.0	Pass
Perfluorododecanoic acid (PFDoA)	<2.50	<0.5	ND	Pass
Perfluoro(2- ethoxyethane)sulfonic acid (PFEESA)	12,1	14,4	6.05 - 18.1	Pass
Perfluoroheptane sulfonic acid (PFHpS)	13,0	14,9	6.50 - 19.4	Pass
Perfluoroheptanoic acid (PFHpA)	39,2	45,5	19.6 - 58.6	Pass
Perfluorohexanesulfonic acid (PFHxS)	24,0	26,6	12.0 - 35.9	Pass
Perfluorohexanoic acid (PFHxA)	12,8	15,5	6.40 - 19.1	Pass
Perfluoro-4-methoxybutanoic acid (PFMBA)	25,6	33,1	12.8 - 38.3	Pass
Perfluoro-3-methoxypropanoic acid (PFMPA)	38,4	45,8	19.2 - 57.4	Pass
Perfluorononane sulfonic acid (PFNS)	16,2	18,0	8.10 - 24.2	Pass
Perfluorononanoic acid (PFNA)	26,4	34,0	13.2 - 39.5	Pass
Perfluorooctanesulfonamide (PFOSA)	14,4	20,2	7.20 - 21.5	Pass
Perfluorooctanesulfonic acid (PFOS)	21,5	21,6	10.8 - 32.2	Pass
Perfluorooctanoic acid (PFOA)	16,8	24,9	8.40 - 25.1	Pass
Perfluoropentane sulfonic acid (PFPeS)	45,1	53,7	22.6 - 67.5	Pass
Perfluoropentanoic acid (PFPeA)	<2.50	<0.5	ND	Pass
Perfluorotetradecanoic acid (PFTDA)	21,6	22,8	10.8 - 32.3	Pass
Perfluorotridecanoic acid (PFTrDA)	40,0	49,7	20.0 - 59.8	Pass
Perfluoroundecanoic acid (PFUnA)	8,40	10,1	4.20 - 12.6	Pass

1. Ranges were determined by ERA based upon shaker table extraction, not microwave
2. ND = not detected down to RL

IMPLEMENTATION AND VALIDATION OF MICROWAVE-ASSISTED EXTRACTION FOR **PFAS IN SOLID MATRICES** UNDER **EPA METHOD 1633A**



DISCUSSION

The validation data confirms that the microwave-assisted extraction (MAE) procedure meets or exceeds all quality assurance and performance requirements of EPA Method 1633A.

By integrating controlled heating and active stirring, MAE provides faster, more consistent, and more reproducible extractions compared to the conventional shaker table method.

Under Method 1633A, laboratories are permitted to modify extraction procedures provided that equivalent accuracy and precision are demonstrated (Section 9.1). The results obtained with the Milestone ETHOS X system demonstrate full compliance with these requirements while delivering substantial operational advantages. Traditional shaker table extraction involves multiple manual transfers and long agitation times, increasing the risk of analyte loss, contamination, and matrix-dependent variability. In contrast, microwave extraction enables complete solvent penetration within sealed polypropylene tubes, significantly improving recoveries even for strongly bound PFAS compounds such as PFTeDA, N-EtFOSAA, and PFOSA. The controlled temperature (65 °C) and uniform stirring accelerate mass transfer and ensure consistent extractions across diverse matrices. In practice, this results in approximately 75% reduction in total extraction time, a major improvement for laboratories managing large sample loads or short turnaround times. The table below summarizes the comparative advantages of the two approaches:

Table 7. Comparative Summary: Shaker Table vs. Microwave Extraction

Criteria	Shaker Table Extraction	Microwave-Assisted Extraction (ETHOS X + Stirring)
Extraction Time	~3 hours; multiple shaking, centrifuging and decanting steps	15 minutes total (plus cooling); up to 44 samples per batch
Throughput	Limited by manual handling and batch size	High-throughput automated operation
Robustness	Ambient temperature only; matrix-dependent variability	Controlled heat and stirring ensure reproducibility
Recoveries – Heavy PFAS (PFTeDA)	Often low due to strong matrix adsorption	Excellent recoveries due to enhanced solvent–matrix interaction
Recoveries – Precursors (N-EtFOSAA, PFOSA, PFOSE)	Incomplete extraction, lower consistency	High recoveries and reproducibility
Overall Reproducibility	Variable across sample types	Highly consistent across all tested matrices

CONCLUSIONS

The validation performed by Advanced Environmental Laboratories (AEL) demonstrates that microwave-assisted extraction provides a fully compliant and more efficient alternative to the traditional shaker table procedure described in EPA Method 1633A. The ETHOS X system ensures robust, reproducible, and faster extractions across a variety of solid matrices, allowing laboratories to increase analytical throughput while maintaining data integrity and regulatory compliance.

ACKNOWLEDGMENT

Milestone, INC would like to acknowledge our collaboration partners at AEL: Shaun West, PFAS Lab Manager, Evan Ged, P.E., Operations Manager and Brandon Beck, VP of Operations.

AEL - Advanced Environmental Laboratories headquartered in Jacksonville, FL, was founded in 1994 and has grown to be Florida's largest laboratory network. They are NELAC/TNI and DOD certified and provide full service conventional analyses and PFAS analyses according to EPA methods 537.1, 533 and 1633A.

Milestone Srl - Via Fatebenefratelli, 1/5, 24010 Sorisole (BG) - Italy
Tel: +39 035 573857 - Fax +39 035 575498
www.milestonesrl.com - email: marketing@milestonesrl.com