



EPA 3rd Draft Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Soil Samples by LC-MS/MS

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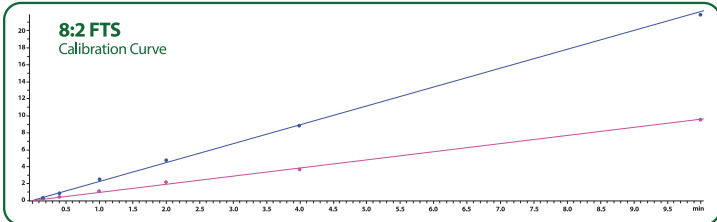
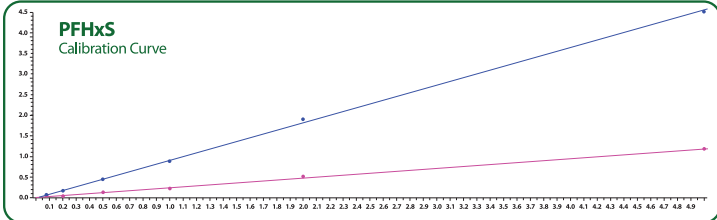
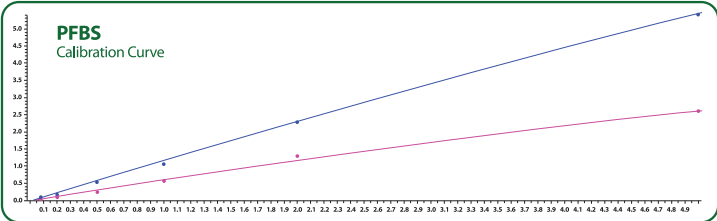


INTRODUCTION

At the end of 2022, the EPA released the 3rd draft of Method 1633. The complexity of all the matrices studied, presents a great challenge to analyte recovery. The diversity in chemical structure adds another layer of difficulty to the overall extraction. Clean extracts and acceptable recoveries were obtained using UCT ENVIRO-CLEAN® ECWAXCB206-P SPE columns.

LC-MS/MS PARAMETERS

HPLC system	SCIEX Exion LC	Detector	ABSciex Qtrap 6500+
Delay column	Selectra® C18 50 x 4.6 mm x 5 µm (p/n: SLC-1850ID46-5UM)		
HPLC column	SelectraCore® C18 50 x 2.1 mm x 2.7 µm (p/n: SCS27-C18521)		
Guard column	SelectraCore® 5 x 2.1 mm x 2.7 µm (p/n: SCS27-C18GDC21)		
Guard column holder	Selectra® (p/n: SLGRDHLDRI)		
Oven temperature	40°C	Flow rate	0.4 mL/min
Injection Volume	2 µL		
Mobile Phase	Bottle A: 20 mM Ammonium acetate in water / Bottle B: Acetonitrile		
Gradient (B Conc.)	0 min (2%), 0-7.5 min (100%), 7.5-8.5 min (100%), 8.5-8.51 min (2%), 8.51-11.5 min (2%)		



References:
[1] 3rd Draft Method 1633 Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. URL: https://www.epa.gov/system/files/documents/2022-12/3rd%20Draft%20Method%201633%20December%202022%2012-22_508.pdf

Disclosure: The speaker, author, moderator, planning member and/or presenter/s do have financial relationships with UCT, Inc., as defined in the AACC policy on potential bias or conflict of interest. The specific product/s: Enviro-Clean® WAX156-P, Enviro-Clean® Graphitized Carbon, SelectraCore® HPLC columns, SelectraCore® Guard Columns, Selectra® Delay Column and Selectra® Guard Column Holder will be mentioned and/or discussed.

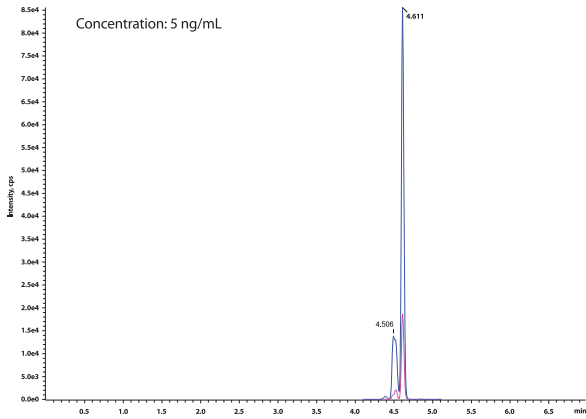


Figure 1: Linear and Branched Isomer Separation (PFOS)

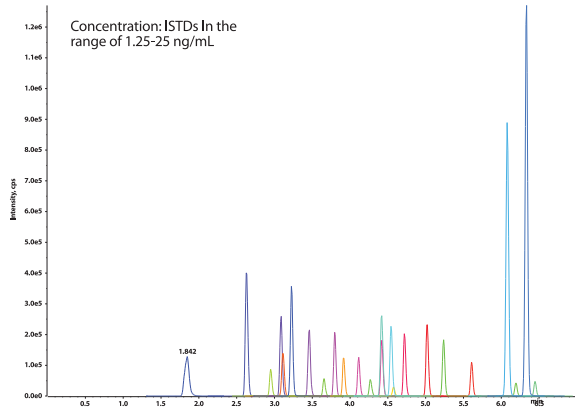


Figure 2: 1633 Extracted Internal Standards

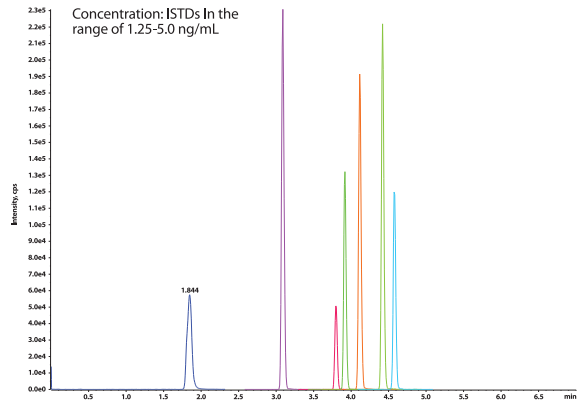


Figure 3: 1633 Non-Extracted Internal Standards

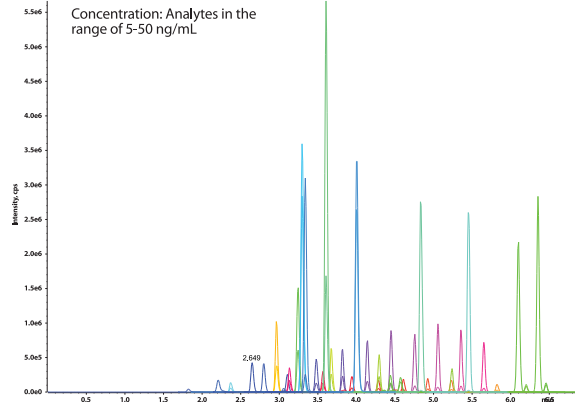


Figure 4: 1633 Targeted Analytes

Sample Preparation:

1. Prepare samples using 5 g of soil wetted with 2.5 g of reagent water. The addition of reagent water to the sand provides a matrix closer in composition to real-world samples.
2. Spike each sample with the reference standards and extracted internal standards (EIS).
3. Add 10 mL of 0.3% methanolic ammonium hydroxide to each centrifuge tube.
4. Vortex mix for 30 minutes. Centrifuge at 2800 rpm for 10 minutes and transfer the supernatant to a clean 50-mL polypropylene centrifuge tube.
5. Repeat step 3 with 15 mL of 0.3% methanolic ammonium hydroxide.
6. Repeat Step 3 with 10 mL of 0.3% methanolic ammonium hydroxide.
7. Combine all extract and evaporate the sample to the 5 mL mark at a very gentle nitrogen flow and a warm water bath at 40°C.
8. Make sure not to evaporate all the methanol in the sample which can cause lower recoveries. Dilute the sample in 50 mL of reagent water and adjust pH if necessary to 6.5 using 2% formic acid solution.

Extraction Procedure:

1. Pack clean silanized glass wool to half the height of the ECWAXCB206-P SPE cartridge barrel
2. Set up the vacuum manifold with one SPE cartridge plus a reservoir and reservoir adaptor for each cartridge for each sample
3. Condition the cartridges by washing them with 15 mL of 1% methanolic ammonium hydroxide
4. Condition the cartridges by washing them with 5 mL of 0.3M formic acid
5. Do not allow the cartridges to go dry.
6. Load the sample.
7. Flow rate should not exceed 2-3 mL per minute.
8. Wash the cartridges with 5 mL reagent water.
9. Wash the cartridges with 10% MeOH in formic acid solution.
10. Dry the cartridge under a high vacuum (15-20 in.Hg) for 5-10 minutes.
11. Add Non-Extracted Internal Standards (NIS) solution to each clean collection tub.
12. Rinse the inside of the sample bottle with 5 mL of 2% methanolic ammonium hydroxide.
13. Transfer the rinse to the SPE reservoir, washing the walls of the reservoir.
Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion.
14. Add 25 µL of concentrated acetic acid to each sample eluted in the collection tubes and vortex.
15. Vortex and transfer a portion of the extract into a 1-mL polypropylene autosampler vial for LC-MS/MS analysis.



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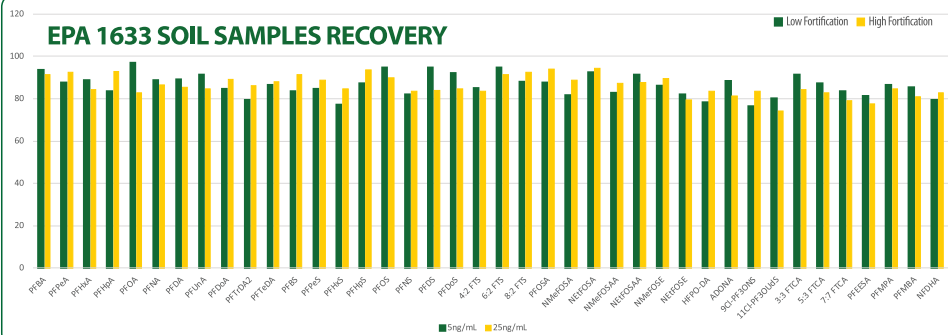
SPE RESULTS IN SOIL SAMPLES

(n=5)

Analyte	Low Fortification		High Fortification	
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
PFBA	93.5	5.4	91.4	6.8
PFPeA	87.6	3.6	92.4	5.1
PFHxA	88.9	4.5	84.1	2.4
PFHpA	83.6	2.9	92.7	4.8
PFOA	96.8	6.7	82.7	1.8
PFNA	88.6	8.2	86.7	6.1
PFDA	89.1	4.7	85.4	8.9
PFUnA	91.4	3.1	84.7	4.7
PFDoA	84.7	7.3	89.2	1.6
PFTraD2	79.4	8.4	86.3	6.9
PFTeDA	86.4	6.1	88.1	4.7
PFBS	83.5	2.3	91.4	5.6
PFPeS	84.6	5.2	88.9	8.1
PFHxS	77.3	6.3	84.6	6.4
PFHpS	87.4	3.7	93.6	4.9
PFOS	94.6	4.8	89.7	9.3
PFNS	81.9	4.5	83.4	4.7
PFDS	94.6	2.7	83.8	1.9
PFDoS	92.3	4.6	84.7	6.3
4:2 FTS	85.1	8.1	83.5	5.5

Analyte	Low Fortification		High Fortification	
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
6:2 FTS	94.6	6.5	91.4	2.9
8:2 FTS	88.1	6.1	92.6	4.6
PFOSA	87.6	7.8	93.8	3.8
NMeFOSA	81.8	5.3	88.9	13.6
NetFOSA	92.6	6.1	94.5	2.4
NMeFOSAA	82.9	10.3	87.3	12.6
NetFOSAA	91.4	6.5	87.6	6.4
NMeFOSE	86.1	4.1	89.5	8.6
NetFOSE	82.2	6.3	79.3	9.9
HFPO-DA	78.3	8.4	83.4	5.8
ADONA	88.2	6.1	81.3	7.3
9Cl-PF3ONS	76.5	7.6	83.6	4.6
11Cl-PF3OUds	80.2	9.4	74.1	8.2
3:3 FTCA	91.5	5.1	84.1	6.9
5:3 FTCA	87.1	4.8	82.6	8.5
7:7 FTCA	83.4	9.6	78.9	4.9
PFEESA	81.4	3.6	77.5	5.8
PFMPA	86.5	4.1	84.7	4.3
PFMBA	85.5	6.7	80.9	2.1
NFDHA	79.3	8.3	82.8	7.4

EPA 1633 SOIL SAMPLES RECOVERY



CONCLUSION

This poster outlines the analysis of PFAS in soil samples according to EPA 1633 third draft method utilizing UCT's Enviro-Clean® double layer polymeric weak-anion exchange (WAX) and Graphitized Carbon Black (GCB) SPE cartridges (ECWAXCB206-P). LC-MS/MS analysis was carried out using a SelectraCore® C18 HPLC analytical column (SCS27-C18521), while a short (5 cm) C18 delay column (SLC-1850ID46-5UM) was used to reduce potential PFAS contamination from the HPLC system. The chromatography was optimized to obtain maximum resolution with minimum co-elution of isomers, including the critical linear and branched isomers of PFHxS and PFOS. For quantitation, a six-point calibration was performed, and all compounds were found to be linear with R² values >0.98. The extraction method was evaluated by spiking soil samples with PFAS at 5 ng/L and 25 ng/L. Recoveries of all analytes were within a range of 70-100% and RSD values <15%. Some Steps in the extraction have proven to be very critical in analyte's recovery. The evaporation cycle after the sample preparation step is a primary source of low analyte recovery.