Analysis of PFAS in soils via automated soil sample preparation and LC-MS/MS as a screening procedure for EPA method 1633

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Introduction

Per- and Polyfluoroalkyl Substances (PFAS) are of considerable interest to regulatory agencies across the globe. In January 2024, the US EPA released method 1633 to test 40 PFAS compounds in a variety of matrices, including soils. An automated screening method for 51 PFAS compounds in soils has been developed and tested with Ottawa sand, topsoil, and humic peat soil. Sixty samples can be extracted in parallel with a specialized, grinding magnetic stirrer and automatically controlled magnetic stirring plate. Samples are extracted with methanolic ammonium hydroxide, cleaned using a carbon SPE cartridge, then concentrated prior to LC-MS/MS analysis. Extracted internal standard recoveries were within EPA 1633 guidelines. Reproducibility of the automated sample preparation workstation were < 10% RSD in laboratory control samples. Carryover was measured with a solvent blank after a mid-level spike and is generally < 1%.

Extraction workflow

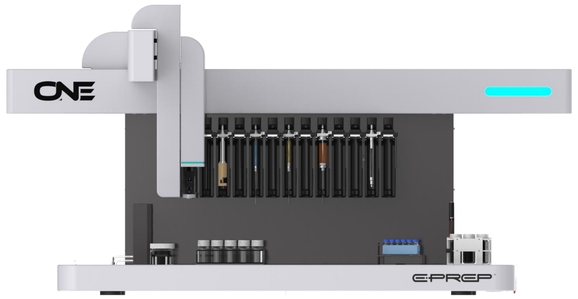
Soil Extraction
- 2 g soil, polypropylene (PP) sealed vials, PP stirring magnets (added manually)
- Add Extraction Standard at 10 ppb
- Add PFAS spikes
- Add 15 mL Extraction solvent 3% ammonium hydroxide 1:1 MeOH:Acetone
- Magnetic Stir 6 mins at 600 RPM
- Pause for 10 min settling time

SPE
- Condition carbon SPE cartridge with 5 mL MeOH followed by 5 mL of extraction solvent
- Load 6mL of sample extract onto carbon cartridges, collect in 15 mL centrifuge tubes
- Elute with 3mL of extraction solvent, collect in 15 mL centrifuge tubes

LC-MSMS Analysis
- Concentrate sample to 1 mL
- Add Non-extracted standard
- LC-MSMS Analysis

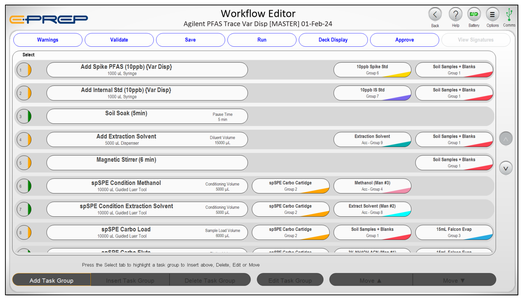
Automated

ePrep ONE Workstation

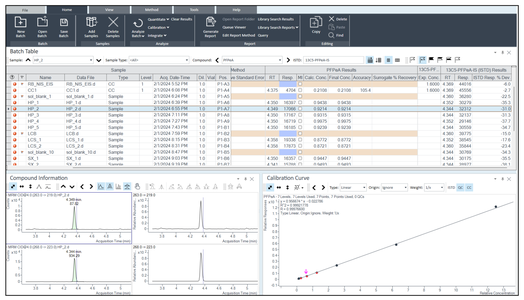


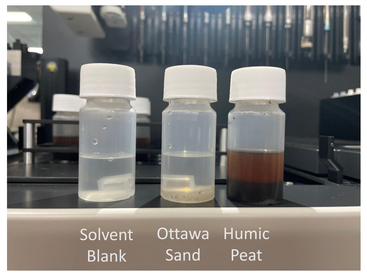
Agilent 1290 Infinity II LC with PFC Free Kit and 6475 TQ

ePrep Software

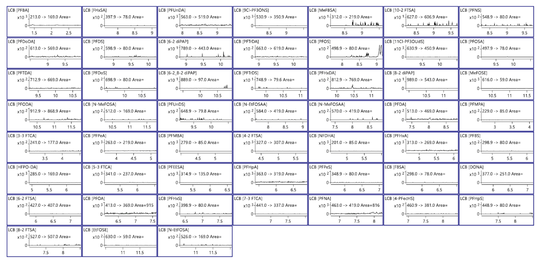


MassHunter 12.1

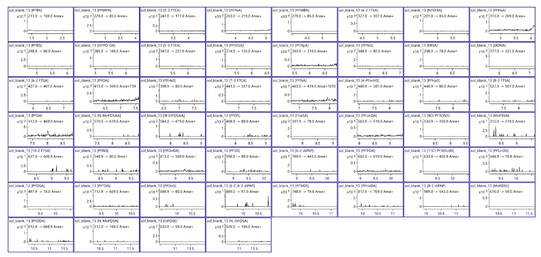




Blank Evaluation
Ottawa Sand Blank

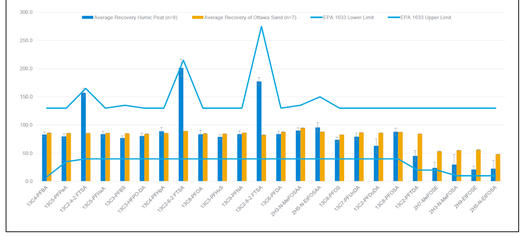


Carryover Evaluation
Solvent Blank extracted after mid-level spike

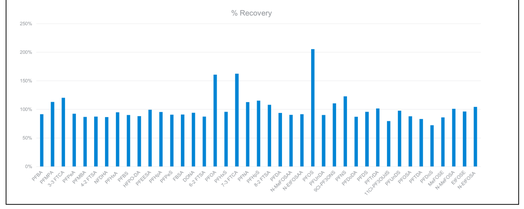


Results

Extraction Standard Recovery
0.5 – 10 ng/g spike



Spike Recovery in Humic Peat Samples
0.5-12.5 ng/g



Reproducibility

Extraction Standard	RSD in Humic Peat (n=8)	RSD in Ottawa Sand (n=7)
13C4-PFBA	5%	0.2%
13C5-PFPeA	6%	0.1%
13C2-4-2 FTSA	5%	0.1%
13C5-PFHxA	7%	0.0%
13C3-PFBS	5%	0.0%
13C3-HFPO-DA	6%	0.2%
13C4-PFHpA	8%	0.1%
13C2-6-2 FTSA	8%	0.1%
13C8-PFOA	8%	0.1%
13C3-PFHxS	5%	0.0%
13C8-PFNA	7%	0.0%
13C2-8-2 FTSA	4%	0.2%
13C8-PFDA	6%	0.0%
2H-3-N-MeFOSAA	6%	0.2%
2H5-N-EtFOSAA	8%	0.2%
13C8-PFOS	8%	0.0%
13C7-PFUnDA	8%	0.0%
13C2-PFDoDA	19%	0.0%
13C8-PFOSA	7%	0.0%
13C2-PFTDA	21%	0.0%
2H7-MeFOSE	42%	2.0%
2H3-N-MeFOSA	59%	0.4%
2H9-EtFOSE	31%	1.9%
2H5-N-EtFOSA	64%	0.4%

Likely caused by variability in the Humic Peat

Conclusion

PFAS in soils screening analysis can be performed in an automated method to ensure reliable data for sound decision making.

Up to 60 soil samples can be processed on an ePrep workstation and an Agilent LC-MSMS with low carryover and no system interference.

Minimal sampling handling is required. Laboratory control sample spiking, internal standard addition, extraction solvent addition, magnetic stirring, and SPE cleanup is performed by the robot in 8 – 10 minutes per sample.