

Analysis of Poly and Perfluoro Alkylsubstances in Soil with Pressurized Liquid Extraction (PLE[®]) and Semi-Automated Solid Phase Extraction (EZPFC[®])

Introduction

Per and polyfluoroalkylated substances (PFAS) is a general term used to describe compounds that are largely comprised of or contain a perfluorinated or polyfluorinated carbon chain moiety such as $F(CF_2)_n-$ or $F(CF_2)_n-(C_2H_4)_n-$. Perfluorooctane sulfonate (PFOS) and other PFAS are widely used in industrial and consumer applications, including stain-resistant coatings for textiles, leather, and carpets; grease-proof coatings for paper products approved for food contact; firefighting foams; mining and oil-well surfactants; floor polishes; and insecticide formulations. In recent years, there has been increasing concern over the levels of PFAS such as PFOS and PFOA (perfluorooctanoic acid) in the global environment and their fate and possible adverse effects.

In addition to extraction of drinking and waste water, also soil is being analyzed for PFAS. In this application note we present a simple and reliable method for fast automated extraction and semi-automated cleanup of soil samples for PFAS testing.

Instrumentation

- FMS Pressurized Liquid Extraction (PLE[®]) System
- FMS EZPFC[®] System
- FMS SuperVap[®] PFC
- Vacuum pump
- Acquity UPLC coupled to a Q-TOF (Xevo G2-XS) and HR-MS (Waters, Milford, MA)

Consumables

- Hydromatrix[®]
- Methanol pesticide grade
- Acetonitrile pesticide grade
- FMS, Inc. 500 mg PFC SDVB cartridge
- Ultra pure DI water
- Methanol pesticide grade
- Isopropyl alcohol pesticide grade

- Wellington Labs native PFAS standard
- Wellington Labs labeled PFAS standard

PLE

- 2 g of soil mixed with 10 g inert Hydromatrix and spiked with surrogates at 50 ppt
- Sample placed in extraction cell
- Capped with re-usable stainless steel end caps
- Heated with 80% methanol/20% acetonitrile for 15 min at 100 °C and 1500 psi
- 15 min cool down
- Nitrogen flush to transfer analytes and extract to collection tubes
- Typical final volume is 40 mL

EZPFC Procedure

- Samples were loaded onto EZPFC system
- Put sample bottles in place and fill rinse bottles with 7.5 mL water
- Cartridges are installed in each of the 12 positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 15 mL methanol (soak 2 min) and 18 mL water (soak 2 min)
- Samples are loaded across cartridges under vacuum (20 - 25 min, ~ 8 inch Hg)
- Rinse sample bottles with 7.5 mL water from rinse bottles and load across cartridge; repeat process (15 mL water total)
- Cartridges are dried with nitrogen for 5 min

Stage 2:

- Fill rinse bottles with 4 mL methanol and use to rinse sample bottles
- 4 mL methanol from sample bottles is loaded across the cartridges with nitrogen and the eluent is collected for analysis into collection vessels
- Repeat process so 8 mL methanol total is collected across cartridge as eluent



FMS PFC SuperVap®

- Pre-heat temp: 65 °C
- Pre-heat time: 20 minutes
- Heat in Time mode at 65 °C under nitrogen (10-15 psi)
- Reduce to dryness

Analysis

- Reconstitute as per method
- Analyze with LC/MS



Figure 1. PLE System used for soil extraction

PFBS	96%	PFDoA	96%
PFHxA	103%	PFTTrDA	91%
GenX	107%	PFTeDA (PFTA)	88%
PFHpA	102%	4:2FTS	98%
PFHxS	98%	6:2FTS	99%
ADONA	98%	8:2FTS	96%
PFOA	108%	NFDHA	97%
PFNA	106%	PFBA	101%
PFOS	96%	PFEESA	98%
9Cl-PF3ONS	93%	PFHpS	100%
PFDA	102%	PFMPA	98%
N-MeFOSAA	94%	PFMBA	101%
PFUdA	102%	PFPeA	104%
N-EtFOSAA	100%	PFPeS	93%
11Cl-PF3OUDS	88%	PFUnA	102%

Table 1. Average recoveries for 50 ppt PFAS spike in soil (%).



Conclusions

The results of the soil samples in Table 1 demonstrate the ability of the FMS PLE and EZPFC systems to deliver accurate results with excellent recoveries. The single cycle automated soil extraction and the subsequent Semi-Automated Solid Phase Extraction (here mostly used as cleanup) provide a fast method for PFAS analysis in soil with the complete sample prep taking about 2.5 hours. Native PFAS background contributions from the systems (both PLE and EZPFC) are negligible. The EZPFC system can run 12 samples in parallel.

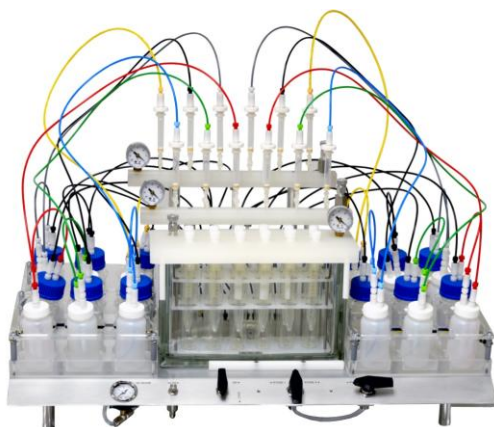


Figure 2. EZPFC System.

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