Analysis of Per- and Polyfluoroalkyl Substances in Drinking Water Using EPA Method 533 with Semi-Automated Solid Phase Extraction (EZPFC®)

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Introduction

Per- and polyfluoroalkyl substances (PFAS) are a family of diverse, yet interrelated, synthetic compounds, first developed in the 1940s. PFAS are used in various products, ranging from Teflon to firefighting foams to food packaging. However, in recent years, these ubiquitous chemicals have been found to persist in groundwater and drinking water, due to their resistant molecular structure. Hence, they are classified as frontier pollutants, and the EPA has recently developed certain methods for their extraction and analysis. The extraction method outlines the use of solid phase extraction for drinking water matrix samples employing WAX cartridges.

Consistent with other EPA 500 series methods, EPA 533 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of semi-automating these processes apparent.

To meet demands for a low-cost method that requires less financial investment than fully automated systems, FMS developed a simple semi - automated system which is fast, inexpensive and yields high quality data.

Instrumentation

- FMS 12-position EZ-PFC® System
- FMS SuperVap-24 PFC®
- Vacuum pump
- Agilent 6475 LCMS

Consumables

- FMS, Inc. 500 mg WAX PFC cartridge
- Ultra-pure DI water
- Fisher Pesticide Grade Methanol
- Acetic acid
- Ammonium acetate
- Sodium phosphate (monobasic and dibasic)
- Ammonium hydroxide
- Method 533 native and labeled spiking standards
- 15 mL polypropylene tubes

Method

Procedure

- 12 samples (250 mL water each) are prepared, containing 1g/L ammonium acetate
- Acetic acid is used to adjust pH to ~6-8
- Spike with various 533 standards
- Cartridges are installed in each of the twelve positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 10 mL methanol (keep wet), 10 mL phosphate buffer (keep wet), and 3 mL phosphate buffer with 2 mL of water (keep wet)
- Samples are loaded across cartridges under vacuum, at 5 mL/min.
- Cartridges are rinsed with 10 mL 1 g/L ammonium acetate in water, then 1 mL methanol
- Cartridges are dried under nitrogen for 5 min

Stage 2:

- Methanol with 2% ammonium hydroxide is added to the rinse bottles (2 x 5 mL) and sprayed across the sample bottles.
- The 5 mL methanol aliquots are pulled drop wise across the cartridges and the eluent is collected.

FMS SuperVap-24 PFC®

- Pre-heat temp: 60-65 °C
- Pre-heat time: 5 minutes
- Heat in Sensor mode at 60-65 °C under nitrogen (15-20 psi)
- Direct to LC Vial Vessel Reduce to dryness and reconstitute to 1 mL as per method (20% water - 80 % methanol)
- Samples are now ready for analysis
- LC/MS analysis.

Results

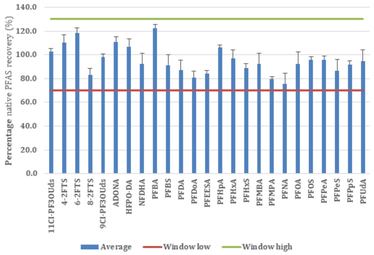


Figure 1. Recoveries (%) and RSDs (%) for 25 native PFAS in drinking water (533) using EZPFC (spiked with 10 ng/L, n=12).

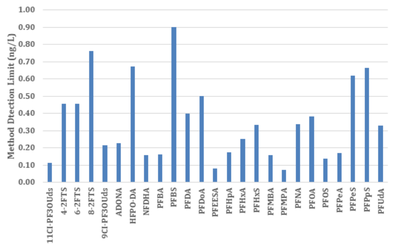


Figure 2. Method Detection Limit values (ng/L) for 25 native PFAS in drinking water (533) using EZPFC (spiked with 1.0 ng/L, n=12).

Discussion and Conclusions

Figure 1 presents the Demonstration of Capability for method 533 using the EZPFC system, showing recoveries between 70–130% for all 25 native PFAS (within the method acceptance window) and RSDs below 10%, demonstrating the reliability of this simple semi-automated system that uses only a vacuum pump as its mechanical component.

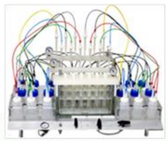
The Method Detection Limit study for these 25 native PFAS showed that most MDLs were below 0.50 ng/L, confirming the system's reproducibility and indicating minimal risk of native background cross-contamination (Figure 2).

This finding aligns with the native background measurements for the EZPFC, where concentrations for all 25 compounds were below 0.03 ng/L.

Drinking water sample results further verified that the FMS EZPFC system delivers accurate data with excellent reproducibility, while remaining inexpensive and easy to operate, making it a strong alternative to manual techniques, liquid–liquid extraction, and glass manifolds.

Using two EZPFC units with a SuperVap-PFC-24 enables processing of 24 PFAS samples in approximately 2–3 hours.

EZPFC system



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