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

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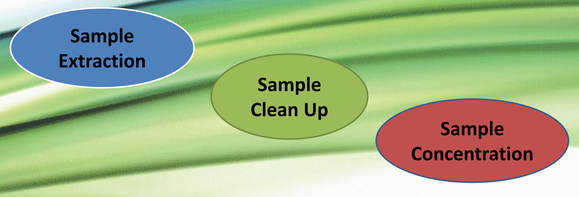
Introduction

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 SDVB 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®
- Vacuum pump
- Agilent 6475 LC/MS

Consumables
- FMS, Inc. 250 mg PFC cartridge
- 15 mL Falcon tubes
- Ultra pure DI water
- Fisher Pesticide Grade Methanol
- Acetic acid
- Ammonium acetate
- Sodium phosphate (mono- and dibasic)
- Method 533 spiking standards



Material and Methods
SPE Procedure

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 six 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, rinses loaded
- 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®
- Pre-heat temp: 55 ºC
- Pre-heat time: 15 min
- Heat in Sensor mode at 60-65 ºC under nitrogen (up to 20-25 psi)
- Direct to LC Vial Vessel Reduce to dryness and reconstitute to 1 mL as per method
- Samples are now ready for LC/MS analysis

Results

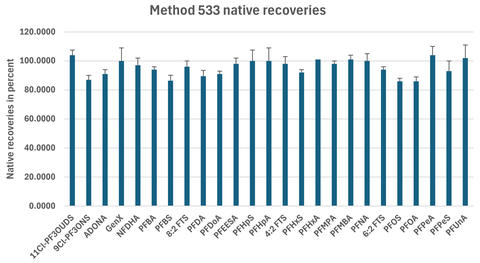


Figure 1. Recoveries of native PFAS compounds in method 533 extracts at 50 ng/L.

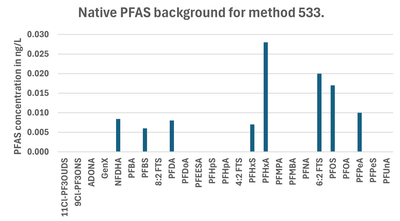
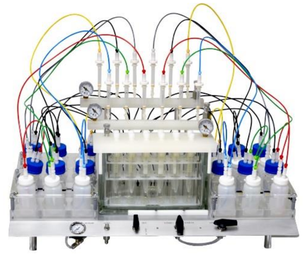


Figure 2. Native background of various PFAS using the semi-automated system.

Discussion and Conclusions

Reviewing the sample data shows high recoveries for the 25 spiked analytes, demonstrating excellent efficiency for the PFAS covered under EPA 533 with results well within the 70-130% acceptance windows. Samples can be taken from sample bottle to LC vial in one quick, consistent, reproducible process that will save laboratories both time and money.



EZPFC system

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