Application Note

Automated Low Background Solid Phase Extraction of Poly- and Perfluoroalkyl Substances in Drinking Water via EPA Method 537.1

Introduction

Poly- and Perfluoralkyl Substances (PFAS) compounds which are largely comprised of or contain a polyfluorinated or perfluorinated carbon chain moiety such as $F(CF_2)_n$- or $F(CF_2)n$-$(C_2H_4)_n$. PFOS and other PFAS 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 chemicals, such as PFOS (perfluorosulfonate) and PFOA (perfluoro-octanoicacid), in the global environment and their fate and possible adverse effects in the environment.

Hence, they are classified as emerging 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 537.1 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 automating these processes apparent.

Instrumentation and Consumables

- FMS, Inc. Turbotrace® PFC SPE system (Solid Phase Extraction)
- FMS, Inc. SuperVap Concentrator
- FMS Inc PFC 500 mg DVB cartridge
- Waters Acquity H-class LC and Waters Xevo TQ MS.
- Fisher Optima® Methanol
- Ultrapure DI water
- PFAS spiking standards

Method Summary

- Prepare six 500 mL DI water samples and spike with standards
- Condition cartridges with 15 mL methanol
- Condition cartridges with 18 mL DI water
- Load 500 mL of water samples across cartridges under vacuum (25-30 min)
- Rinse sample bottles twice with 7.5 mL DI water and load across cartridges
- Dry cartridges under nitrogen for 5 min
- Rinse sample bottles twice with 4 mL methanol and elute cartridges

FMS SuperVap®

■Pre-heat temp: 60-65 ºC
■ Pre-heat time: 20 minutes
■ Heat in Time mode at 60-65 ºC under nitrogen (7-10 psi)
■ Reduce to dryness

Analysis

■ Reconstitute as per method
■ Analyze with LC/MS

TurboTrace® System for PFAS

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Table 1 Average recoveries for 537.1 analytes (50 ng/L).

Analyte	Average Recoveries (%)
PFBS	83
PFHxA	92
PFHxS	86
PFHpA	86
ADONA	85
PFOA	103
PFOS	82
PFNA	82
9Cl-PF3ONS	100
PFDA	111
NMeFOSAA	92
PFUnA	108
NEtFOSAA	98
PFDoA	101
PFTrDA	91
PFTA	85

Conclusion

The FMS PFC SPE system is made of peek and stainless steel components and produces reliable, reproducible results for PFAS in drinking water. The system, by design, has very low background PFAS allowing for high throughput analysis of samples without any significant interference.

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