EPA 537.1 PFAS in Drinking Water with Parallel/Sequential Automated Solid Phase Extraction



Introduction

Poly- and Perfluoralkyl Substances (PFAS) compounds which are largely comprised of or contain a polyfluorinated or perfluorinated carbon chain moiety such as F(CF₂)_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. This application note describes the method for parallel/sequential automated Solid Phase Extraction. Up to 30 samples can be processed with this system in a 6 h period fully automated and unattended.

Instrumentation and Consumables

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

Method Summary

- Prepare up to thirty 500 mL DI water samples and spike with standards (2 and 50 ppt)
- 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 °C
- Pre-heat time: 20 minutes
- Heat in Time mode at 60-65 °C under nitrogen (7-10 psi)
- Reduce to dryness

Analysis

- Reconstitute as per method
- Analyze with LC/MS



TurboTrace® Parallel/Sequential System for PFAS Extraction





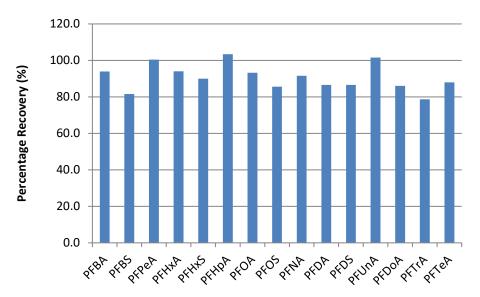


Figure 1. Average recoveries for the parallel/sequential SPE system (%).

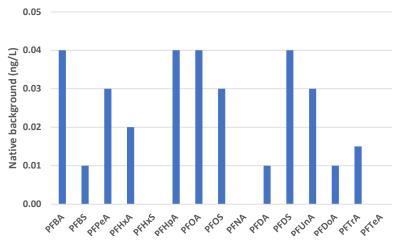


Figure 2. Native PFAS background from parallel/sequential system.

Conclusion

The automated extraction and analysis of PFAS with the parallel/sequential TurboTrace SPE system holds great promise for laboratories. Drinking water can be analyzed quickly and reproducibly with this system. The recoveries for PFAS easily meet the U.S. EPA Method 537.1 requirements of the 70-130% window (Figure 1).

The background contribution from the parallel/sequential system is very low (Figure 2) and is <0.04 ng/L for all PFAS. The design of the systems, with all PEEK and stainless-steel surfaces, ensures sample extraction with very low background contamination.

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