



High Throughput Solid Phase Extraction and Concentration of PFAS/PFOS in Waste-Water Using EPA Method 533 and 537.1

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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 and 537.1 incorporate 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.

Instrumentation and Consumables

FMS, Inc. TurboTrace® PFC SPE system (Solid Phase Extraction)
FMS, Inc. SuperVap PFC-24 Concentrator
Waters Acquity H-class LC and Waters Xevo TQ MS
Relevant 533 and 537.1 PFAS spiking standards
FMS 1 g DVB PFC cartridges

Ultra pure DI water
Methanol pesticide grade
Isopropyl alcohol pesticide grade
Acetic acid
Ammonium acetate
Sodium phosphate (monobasic and dibasic)
Ammonium hydroxide



537.1 Procedure

Load 6 samples onto system 250 mL each and spike with relevant standards
Condition cartridges with 15 ml methanol (soak) and 18 mL water (soak)
Load samples across cartridges under vacuum (20 - 25 min, ~ 12 inch Hg)
Automatically rinse sample bottles with 2 x 7.5 mL water
Dry cartridges for 5 min with nitrogen
Rinse sample bottles with 2 x 4 mL methanol and elute
Evaporate to dryness and reconstitute as per method

533 Procedure

Load 6 samples on system 250 mL each and spike with relevant standards, add ammonium acetate
Adjust pH to 6-8
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)
Load samples across cartridges under vacuum (20 - 25 min, ~ 12 inch Hg)
Cartridges are rinsed with ammonium acetate, methanol and dried for 5 min (nitrogen)
Rinse sample bottles with 2 x 5 mL methanol/ammonium hydroxide and elute
Evaporate to dryness and reconstitute as per method

SuperVap PFC-24

Pre-heat for 5 min to 60-65 °C
Evaporate to dryness at 60-65 °C at 20-25 psi nitrogen
System can evaporate 24 samples at same time

Results

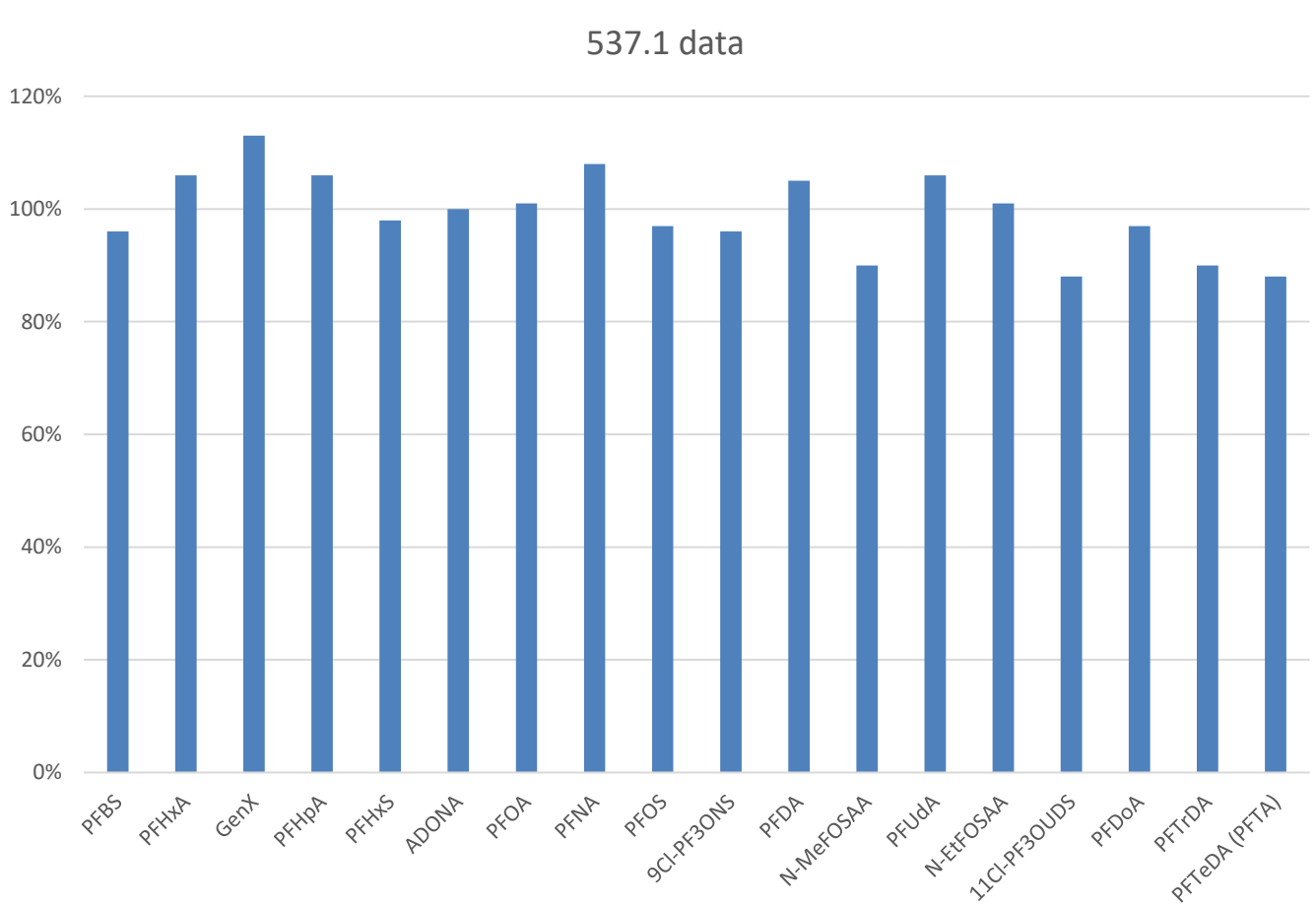


Figure 1. Native PFAS recoveries for 537.1 method (%). 50 ppt.

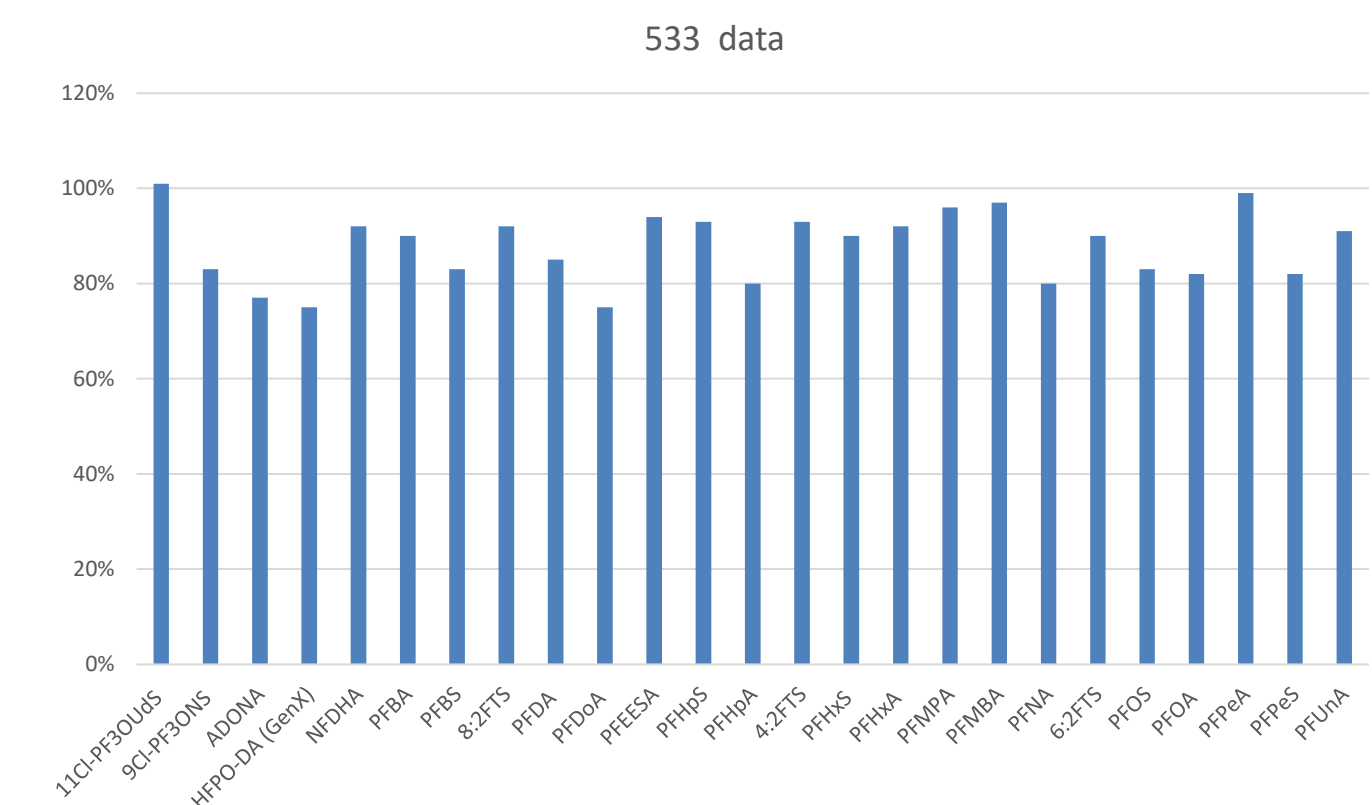


Figure 2. Native PFAS recoveries for 533 method (%). 50 ppt.

Conclusion

The FMS TurboTrace PFC SPE system produces reliable, reproducible results for PFAS in water. The system, by design, has very low background PFAS allowing for analysis of samples without any significant interference. Both EPA method 533 and 537.1 give excellent recoveries.

The SuperVap PFC-24 can accommodate 24 samples for final blow down to dryness as per these two EPA methods. Final extract size can be up to 15 mL.

For waste-water samples Delrin filtration wool can be used in the cartridge barrel to prevent plugging. This greatly reduces chance of slowing down the loading and elution procedure because of cartridge problems with samples containing a lot of particulate.



For additional information please contact:

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SPE TurboTrace® System for PFAS extraction