



Automated Solid Phase Extraction of Per- and Polyfluoroalkyl Substances in Wastewater with a Parallel-Sequential System Using EPA Method 1633

Ruud Addink, Tom Hall,

Fluid Management Systems, 900 Technology Park Drive, Billerica, MA 01821

www.fms-inc.com

Introduction

Per- and polyfluoroalkyl substances (PFAS) constitute a group of compounds characterized by perfluorinated or polyfluorinated carbon chain moieties, typically denoted by structures such as $F(CF_2)_n-$ or $F(CF_2)_n-(C_2H_4)_n$. Due to their unique properties, these substances have found extensive application in various industrial and consumer products.

Many industrial and consumer applications utilize perfluorooctane sulfonate (PFOS) and other PFAS compounds. These include but are not limited to, stain-resistant coatings for textiles, leather, and carpets; grease-proof coatings for food-contact paper products; firefighting foams; surfactants for mining and oil-well operations; floor polishes; and insecticide formulations. Their widespread usage has led to their ubiquitous presence in the environment.

In recent years, mounting concerns have emerged regarding the widespread distribution and potential adverse effects of PFAS, particularly notable compounds like PFOS and perfluorooctanoic acid (PFOA). These concerns have prompted intensified scrutiny of these substances' environmental occurrence, fate, and potential impacts.

Recent developments in the United States have led to the introduction of EPA method 1633, which addresses the need for robust methodologies to monitor and analyze PFAS. This method, unveiled in early 2024, enables comprehensive analysis across various matrices, including wastewater, surface water, groundwater, soil, biosolids, sediment, landfill leachate, and fish tissue. EPA Method 1633 represents a significant advancement in the analytical toolkit for assessing PFAS contamination and understanding their distribution and behavior in diverse environmental compartments.

Materials and methods

Instrumentation

- FMS, Inc. TurboTrace® Parallel/Sequential PFAS SPE system (Solid Phase Extraction) is a modular expandable system designed for handling wastewater samples. The system can have from one to six modules. Each module can run 5 samples sequentially, the system can be expanded from one to 6 modules to run a total of 5 to 30 samples.
- FMS Vacuum pump
- Agilent 6470 TripleQuad LC/MS

Consumables

- FMS Inc PFAS WAX 150 mg cartridges
- Ultrapure DI water
- Methanol pesticide grade
- Ammonium hydroxide
- Formic acid
- Relevant PFAS spiking standards

Method Parallel/Sequential

- Ten synthetic wastewater samples (500 mL) spiked with native PFAS standards and relevant internals
- Load sample bottles onto system and install cartridges
- Rinse bottles are automatically filled during procedure
- Use positive pressure (nitrogen) for pumping solvents and mixes through the system and use vacuum to load the samples
- Condition cartridges with 15 mL 1% methanolic ammonium hydroxide followed by 5 mL of 0.3M formic acid.
- Load samples across the cartridges at 5-10 mL/min (~ 8-inch Hg)
- Sample bottles rinsed with 5 mL reagent water (twice) followed by 5 mL of 1:1 0.1M formic acid/methanol and load rinses across the cartridges
- Dry 10 min
- Rinse sample bottles with 5 mL 1% methanolic ammonium hydroxide
- Load rinses across cartridges and collect in polypropylene tubes
- Cleanup over 10 mg of loose carbon
- As per the method no further concentration is carried out.
- Further relevant standards were added prior to LC/MS analysis.

Analysis

- Take aliquot from final 5 mL extract
- Analyze with LC/MS



Parallel-Sequential system

Results

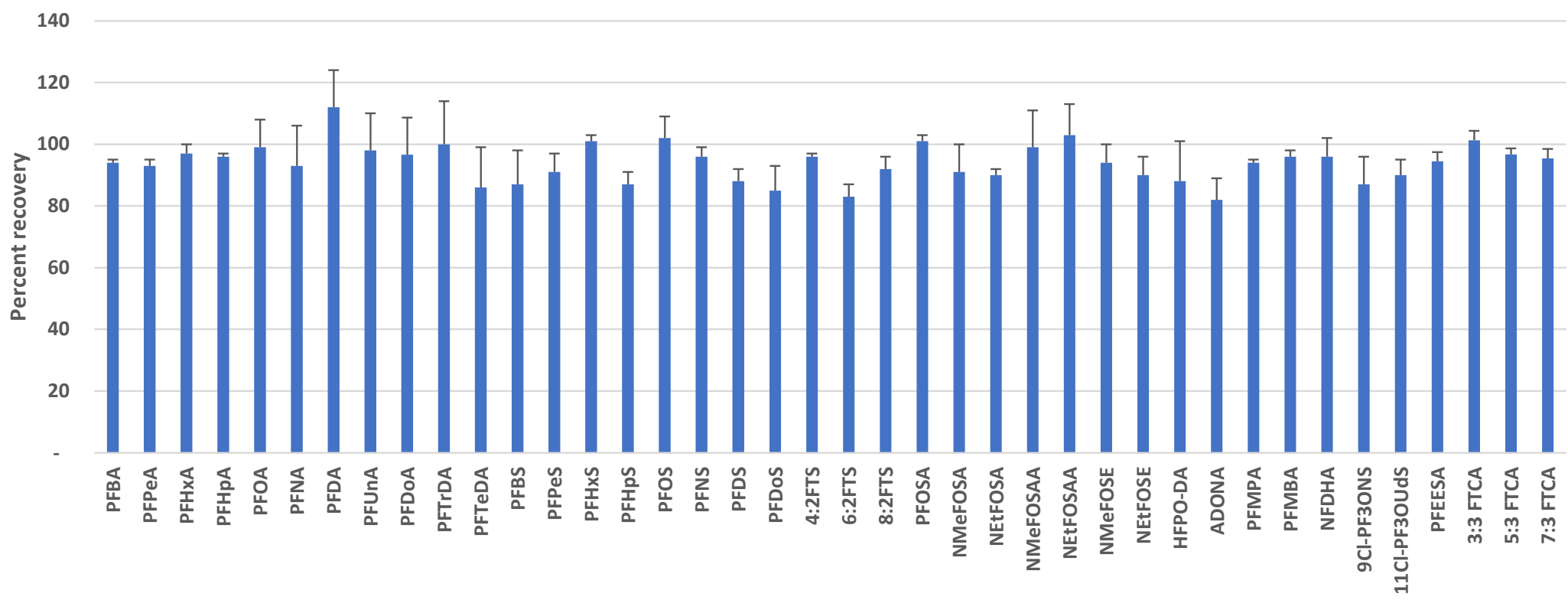


Figure 1. Demonstration of Capability for 40 native PFAS compounds. (Recoveries of native PFAS)

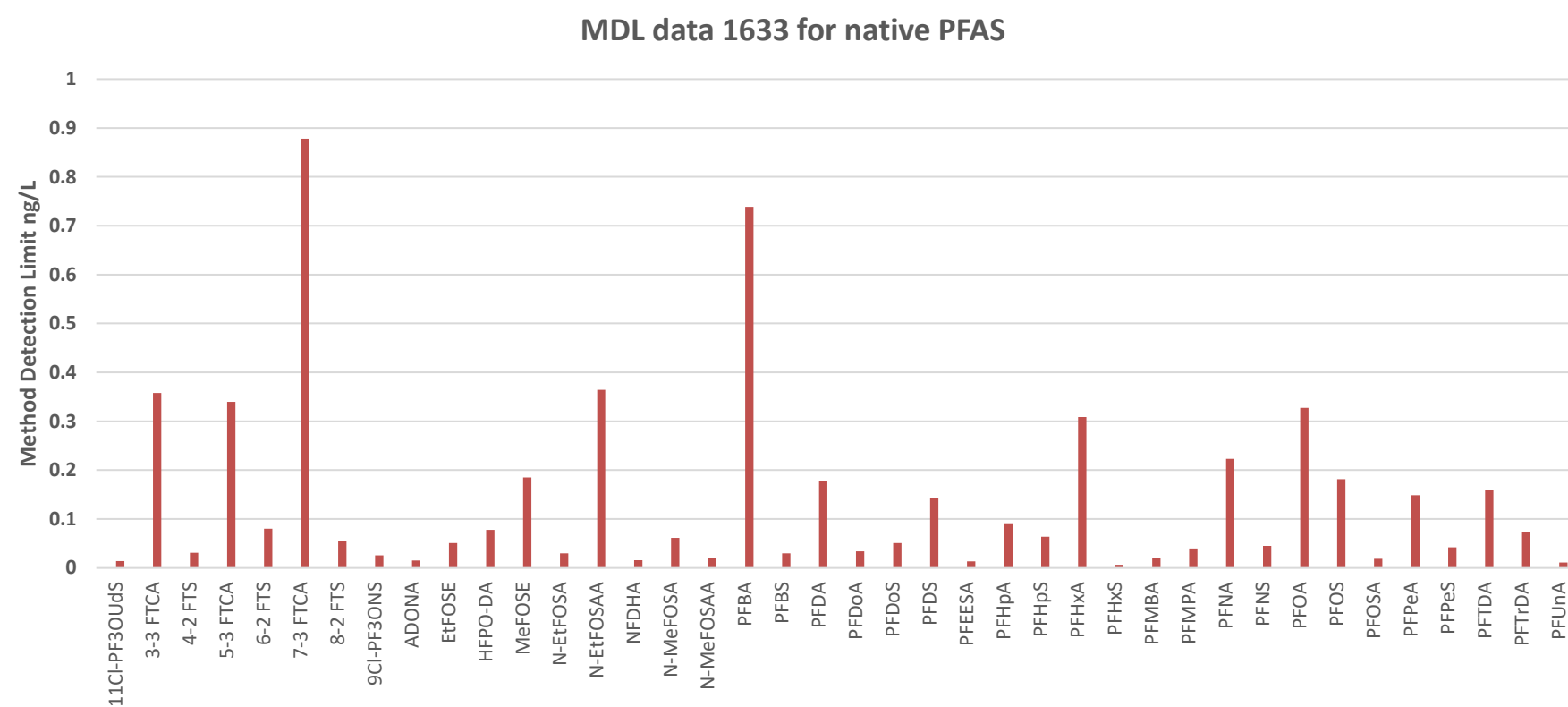


Figure 2. Method Detection Limit for 40 native PFAS compounds on Parallel-Sequential system.

Conclusions

All PFAS data were within the acceptance windows (different for each compound) required by the method. The total run time of the automated system is < 70 min. The fully automated extraction system produces excellent data with low standard deviations.

The background contribution from the parallel/sequential system is minimal (< 0.02 ng/L, Figure 3). The system's design, with stainless-steel surfaces, PEEK, and polypropylene tubing, ensures sample extraction with minimal native background contamination.

The problem with ground and wastewater extraction is particulate matter, which can easily plug up cartridges. Using plastic filtration wool in the barrel of the cartridges can eliminate this problem. In this work, no clogging of cartridges occurs.

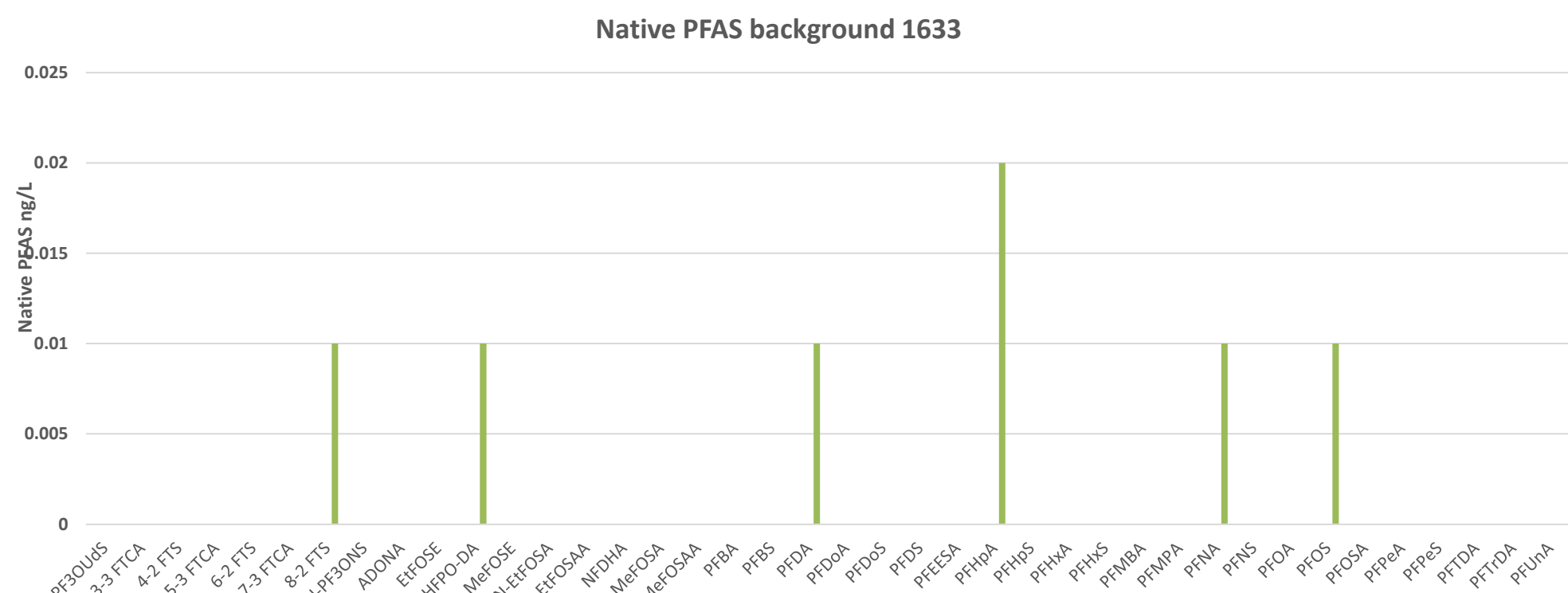


Figure 3. Native PFAS background contribution from system.

For additional information please contact:

Tom Hall
FMS, Inc.
thall@fms-inc.com
www.fms-inc.com

Sample
Extraction

Sample
Clean Up

Sample
Concentration