Application Note

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

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 6-position EZ-PFC® System
- FMS SuperVap®
- Vacuum pump
- Waters Acquity UPLC
- Waters Xevo TQD

Consumables

- FMS, Inc. 500 mg PFC cartridge
- Ultra pure DI water
- Fisher Pesticide Grade Methanol
- Method 533 spiking standards
- 15 mL Falcon tubes
- Acetic acid
- Ammonium acetate
- Sodium phosphate (dibasic and monobasic)
- Ammonium hydroxide

Procedure

- 6 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
- 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 minutes
- Heat in Sensor mode at 55 ºC under nitrogen (5-7 psi)
- Direct to LC Vial Vessel Reduce to dryness and reconstitute to 1 mL as per method
- Samples are now ready for analysis

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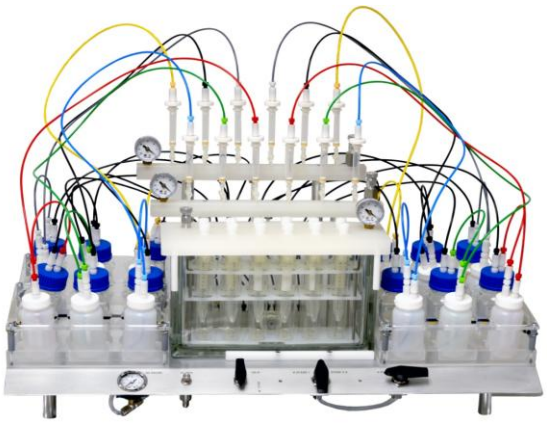
Table 1 with average recoveries (%) and RSDs (%) for 533 PFAS analytes (50 ng/L) n=4

Analyte	Average Recoveries (%)	RSDs (%)
11Cl-PF3OUdS	104.0	3.5
9Cl-PF3ONS	86.9	3.0
ADONA	80.2	2.9
HFPO-DA (GenX)	78.7	3.7
NFDHA	97.0	5.0
PFBA	101.2	12.3
PFBS	86.4	3.7
8:2FTS	96.0	4.0
PFDA	89.5	4.0
PFDoA	80.6	8.9
PFEESA	98.0	4.0
PFHpS	100.2	7.5
PFHpA	83.0	3.5
4:2FTS	98.0	5.0
PFHxS	92.0	1.9
PFHxA	101.2	10.0
PFMPA	98.0	2.0
PFMBA	101.0	3.0
PFNA	81.3	3.0
6:2FTS	99.0	11.0
PFOS	86.2	2.0
PFOA	85.7	2.9
PFPeA	104.3	6.3
PFPeS	92.8	11.1
PFUnA	101.6	9.2



FMS EZPFC® System

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 collection bottle to LC vial in one quick, consistent, reproducible process that will save laboratories both time and money.

For more information contact FMS:
FMS, Inc.
580 Pleasant Street
Watertown, MA 02472
Phone: (617) 393-2396
Fax: (617) 393-0194
Email: onlineinfo@fms-inc.com
Web site: www.fms-inc.com