

Automated Low Background Solid Phase Extraction of Perfluorinated Compounds in Water

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Introduction

- Perfluoralkylated compounds contain a perfluorinated or polyfluorinated carbon chain moiety such as $F(CF_2)_n$ or $F(CF_2)n-(C_2H_4)_n$.
- Use in industrial and consumer applications: stainresistant coatings for textiles and carpets; greaseproof coatings for paper products approved for food contact; firefighting foams; mining and oil well surfactants; floor polishes; and insecticide formulations.

Concern over levels in the global environment: fate and possible adverse effects.



Health concerns (1)

• Results from animal studies:

- -Disruption of endocrine activity.
- -Reduced immune function.
- Damage to liver and pancreas.
- Developmental problems in off-spring.





Health concerns (2)

- Data from human studies have been contradictory: some found effects on human health, some did not.
- Additional work is needed.
- Two compounds most studied: perfluorooctane sulphonate (PFOS) and perfluorooctanoic acid (PFOA).



Health concerns (3)

• These are final environmental degradation products of (and contaminants in) a wide range of other perfluorinated products and have therefore been of most interest.





Regulation

 PFOS is now subject to varying but increasing levels of control in a number of countries. PFOA, also a widespread contaminant but with a far lower bioaccumulation potential, is still under evaluation.





SPE System for PFC extraction







Extraction procedure (1)

- 500 mL water samples are spiked with 25 uL of 1 ug/mL PFC standard solution.
- Use Waters HLB Plus 225mg cartridge.
- Condition cartridge with 15 mL methanol.
- Condition cartridge with 40 mL water.





Extraction procedure (2)

- Load samples on the FMS PFC SPE system.
- Pass across cartridge under -12 psi vacuum.
- Rinse bottle with 25 mL of water and load onto the cartridge under negative pressure.





Extraction procedure (3)

- Dry cartridges under nitrogen until no residual water is present (~ 20 min).
- Elute with 15 mL methanol.











SuperVap Evaporation

- Pre-heat temp: 50 °C
- Pre-heat time: 20 minutes
- Heat in Sensor mode: 50 °C
- Nitrogen pressure: 9 PSI
- The extracts were concentrated to 500 uL, after which internal standard was added. The samples were diluted to a final volume of 1 mL of water for LC/MS analysis.









Analysis (1)

- UPLC Conditions
 - Waters Acquity H-Class UPLC
 - Column: Waters BEH C_{18} , 2.1 x 50 mm, 1.7 um
 - Column temperature: 50 °C





Analysis (2)

- Solvent A: (98:2) 2 mM ammonium acetate : methanol
- Solvent B: methanol + 2 mM ammonium acetate





Analysis (3)

- Xevo TQD mass spectrometer
- Ionization mode: ESI-
- Acquisition mode: Dual Scan MRM
- Capillary voltage: 0.44 kV
- Source: 150 °C
- Data: acquisition and analysis using MassLynx v.4.1 software





Results

Compound	Amount Spiked ng/L	Mean Percent Recovery	Background from FMS SPE
Perfluorinated	50 ng/L		
Compounds			
PFHxA		61%	0.068
PFHxS		77%	0.06
PFOA		88%	0.214
PFNA		74%	0.062
PFOS		72%	0.048
PFDA		84%	0.054
PFUdA		65%	
PFDoA		57%	0.011





Conclusions

- It is possible to automate the sample preparation of Perfluorinated Compounds with the FMS PFC SPE system to get reliable and reproducible results for perfluorinated compounds in water.
- The system, by design, has very low background PFC allowing for analysis of samples without any significant interference.
- Fully automated features make for rapid and reliable sample processing with same-day-analysis.

