Analysis of Ultrashort Chain PFAS Using Automated SPE Process from Various Sample Matrices

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Abstract

Highlights:

- Automated solid phase extraction of ultrashort and short chain PFAS using PromoChrom SPE-03 and Aquaris™ cartridges.
- Capacity for simultaneous co-extraction of >60 PFAS compounds (C1–C20) and analysis with single LC-MS/MS injection.
- Extraction performed and is effective for various aqueous and food matrices.

Introduction

Ultrashort chain PFAS (C1–C3) are increasingly detected in environmental waters and food matrices. Although generally considered less bioaccumulative than long-chain PFAS, their high mobility, persistence, and widespread occurrence raise growing health and environmental concerns. Their strong C–F bonds make them difficult to remove through conventional water treatment, and their high polarity presents analytical challenges under traditional methods such as EPA Method 1633.

Current analytical approaches for ultrashort chain PFAS include:

- **Direct injection methods** (e.g., EPA L563 and ASTM D8421):
  These approaches are limited by matrices and sensitivity. EPA L563 is validated primarily for drinking water, while ASTM D8421 reports method reporting limits in the range of approximately 5–25 ng/L.
- **Solid phase extraction (SPE) methods** (e.g., ASTM D8628):
  This recently published method adopts extraction concepts similar to EPA 1633. However, it specifies a relatively small sample volume (20 mL) and has reported notable variability for isotopically labeled compounds such as TFA and PFPrA (RSD >30%).

In this presentation, we demonstrate an automated SPE workflow using the SPE-03 system that co-extracts more than 60 PFAS compounds (C1–C20) across drinking water, river water, and food matrices. The approach delivers high sensitivity and can be integrated with conventional PFAS methods for comprehensive analysis.

Instruments and Materials

SPE Cartridges: PromoChrom Aquaris™ PFAS WAX cartridge
(For details of the cartridge, please contact PromoChorm)

Instrument for Extraction: PromoChrom SPE-03 Plus automated extractor for PFAS analysis with upside-down sample loading

Instrument for Concentration: LCTech D-EVA Vacuum Concentrator

Instrument for Analysis: Thermo Scientific Vanquish Flex UHPLC + TSQ Quantis

Standards Spiking Concentration:

- Labelled compounds: **1.64-33.3 ng**
- 11Cl-PF3OUdS, 9Cl-PF3ONS, ADONA, HFPO-DA, NFDHA, PFEESA, PFMBA, PFMPA, PFPeA: **8 ng**; PFBA, 4:2FTS, 6:2FTS, 8:2FTS: **16 ng**; N-MeFOSE, N-EtFOSE: 5:3 FTCA, 7:3 FTCA: **80 ng**; FBSA-I, PFECHS, FHXSA-I, P37DMOA, FOUEA, 6:2 diPAP, 8:2 diPAP: **6.67 ng**; PFHxDA, PFODA: **3.33 ng**; L-PFUdS, L-PFTrDS: **2 ng**; 6:2 PAP, 8:2 PAP, PFDPA: **4 ng**
- All other PFAS: 4 ng (visible in the graph)

Aqueous Matrices

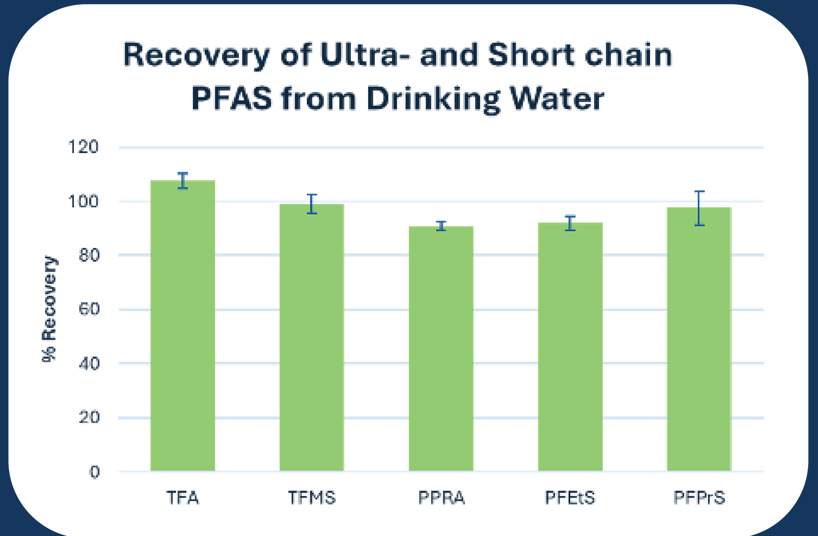
Drinking water: 50 mL drinking water (n=4) was spiked with ultrashort to long chain (C1-C20) PFAS. Sample concentration was enriched by a SPE cartridge procedure automated on the SPE-03 system. Evaporation was done in D-EVA (automated vacuum centrifuge with cold trap for solvents). The samples were subsequently analyzed in LC-MS/MS. For details of the extraction procedure, please contact PromoChrom.

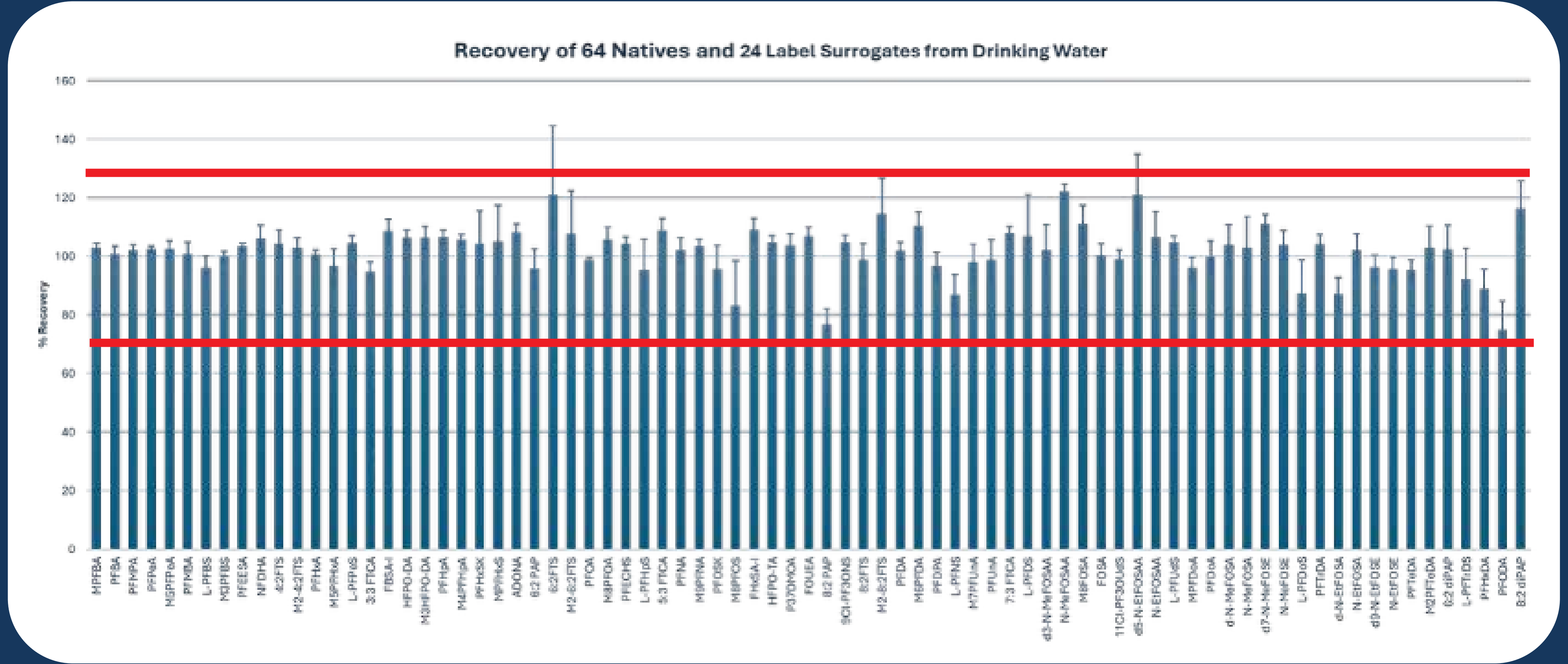
Drinking water results

All results shown represent absolute average recoveries without isotopic correction.

All 5 ultrashort chain PFAS achieve recoveries above 90% and RSD less than 10%.

All other native and surrogate PFAS recoveries fall within the 70–130% internal QC limits and meet the requirements of EPA Method 1633.





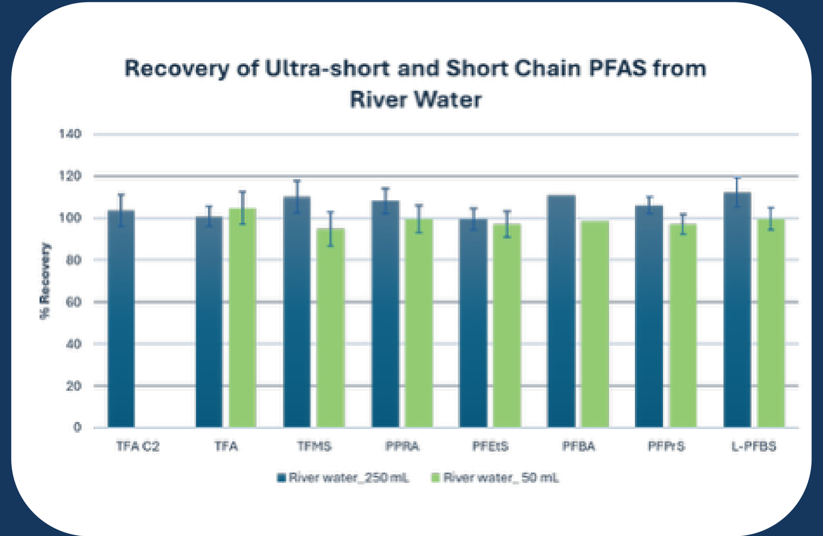
River water: 50 mL (n=4) and 250 mL (n=4) river water was spiked with ultrashort to long chain (C1-C20) PFAS. Sample concentration was enriched by a SPE cartridge procedure automated on the SPE-03 system. Evaporation was done in D-EVA (automated vacuum centrifuge with cold trap for solvents). The samples were subsequently analyzed in LC-MS/MS. For details of the extraction procedure, please contact PromoChrom.

River water results

The extraction procedure was slightly modified from the drinking water method by introducing an organic–water wash step.

Results for the ultrashort and short-chain PFAS are shown. All recoveries fall within the 70–130% internal QC limits, with RSDs below 10%.

Results for other PFAS are available but not shown.



Food Matrices

Carrots and cucumber: Grinded 1 g cucumber (n=4) and 2 g carrot (n=4) were extracted by following the US EPA Method 1633 tissue extraction procedure.

Add 0.3% basic MeOH, shake for 5 min at 800 rpm, centrifugate at 2700 rpm for 10 min and transfer the extract

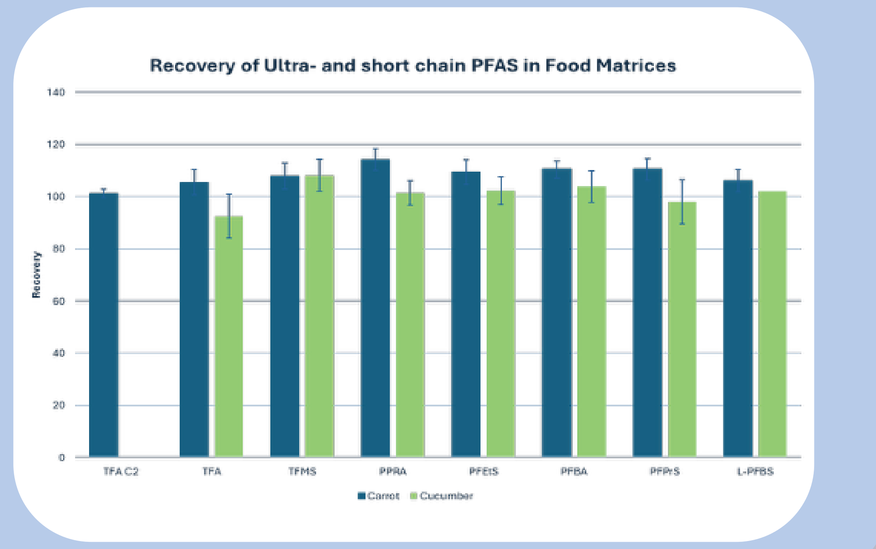
Do above 3 x with 10 mL, 15 mL and 5 mL of 0.3% basic MeOH

The final extract is evaporated to less than 5 mL and diluted with water to 50 mL.
50mL samples are concentrated by SPE using the same procedure as the river water extraction.
For details of the extraction procedure, please contact PromoChrom.

Food matrices results

Results for the ultrashort and short-chain PFAS show no significant differences between the two matrices, demonstrating the versatility of the procedure for handling varied sample types.

The same extraction approach is likely applicable to solids and biosolids. All recoveries fall within the acceptable range.



LC-MS/MS Method

The LC–MS/MS method is provided for reference. It is capable of separating and quantifying all 60 PFAS compounds, including ultrashort-chain analytes.

LC-Parameter

Parameter	Value	
LC	Thermo Scientific Vanquish Flex UHPLC system	
Analytical column	Acclaim 120, C18, 3 µm, 2.1 x 150 mm	
Delay column	Acclaim RSLC, PolarAdvantage II, 3.0 mm x 33 mm, 3 µm	
Column temperature	40 °C	
Injection volume	5 µL	
Mobile Phase	A) H2O B) 2 mM ammonium acetate MeOH with 2 % H2O and 0.1 % acetic acid	
Gradient Flow rate	0.4 mL/min	
Gradient	Time (min)	% B
	0	10
	1	10
	4	50
	14	100
	17	100
	18	10
	20	10
	22	10

MS/MS -Parameter

Parameter	Value
MS	TSQ Quantis (Thermo)
Polarity	Negative
Spray voltage	2500 V
Sheath Gas	50 Arb
Aux Gas	10
CID Gas	2 mTorr
Ion transfer tube temp	325 °C
Vaporizer Temp	300 °C
Q1 resolution	0.7 FWHM
Q3 resolution	1.2 FWHM
Cycle time	0.5 sec
Chromatographic peak width	6. sec

Conclusion

PromoChrom's SPE-03 system, coupled with Aquaris™ PFAS WAX cartridges, provides an effective solution for extracting ultrashort PFAS from a wide range of sample matrices. In addition, more than 64 other PFAS compounds can be co-extracted in a single run, eliminating the need for multiple extractions of the same sample for different groups. Automation with PromoChrom offers a time- and cost-efficient solution for laboratories.