

Nota applicativa

Sample Preparation and Analysis Workflow to Address the PFAS Primary National Drinking Water Regulation

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Questa relazione è un Application Brief e non contiene una sezione dettagliata sull'esperimento.

Abstract

The newly announced PFAS National Primary Drinking Water Regulation has set enforceable levels of six PFAS compounds in drinking water for the first time in the United States. Regulatory levels of individual compounds range from 4–10 ng/L (or ppt). The Waters™ PFAS Solution for sample preparation through sample analysis is presented to make implementation of the new PFAS regulations straightforward and accurately reach far below the lowest regulated Maximum Contamination Level of 4 ng/L.

Benefits

- The Xevo™ TQ-S micro Mass Spectrometer is demonstrated to have more than enough sensitivity to meet the new PFAS National Primary Drinking Water Regulations when following EPA 537.1 and EPA 533 methods
 - Both the Sep-Pak™ PS2 and Oasis™ WAX for PFAS SPE Cartridges provide excellent recovery for the six regulated PFAS in drinking water
 - A full workflow using Waters solutions for sample preparation and analysis for accurate and reliable testing to
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conform with the new PFAS drinking water regulations

Introduction

The United States Environmental Protection Agency (EPA) finalized the first ever enforceable PFAS drinking water levels on April 10, 2024.¹ The PFAS National Primary Drinking Water Regulation (NPDWR) will regulate the maximum contamination levels (MCLs) of six PFAS in public water supplies. Included in the list of regulated PFAS are PFOA, PFNA, PFBS, PFHxS, PFOS, and HFPO-DA (commonly called GenX). Individual MCL values of four parts per trillion (ppt) are set for PFOA and PFOS and ten ppt for PFNA, PFHxS, and HFPO-DA. Additionally, when PFBS, PFNA, PFHxS, and HFPO-DA occur as a mixture of at least two or more in a drinking water sample, a Hazard Index must also be calculated and reported to represent the additive health effects of the mixture. The Hazard Index cannot exceed one under the implementation of this regulation.

By 2027 all public water systems will be required to have implemented routine monitoring of these six PFAS compounds. Results from an initial monitoring period between 2024–2027 on a quarterly or twice per year basis (depending on the number of customers supplied by the system) will determine the frequency of monitoring required from 2027 onward. Currently, two EPA methods are available for the testing of drinking water for PFAS: EPA 537.1 and EPA 533. Both of these methods cover the six newly regulated PFAS. The Waters PFAS Solution for sample preparation through sample analysis is presented to make implementation of the new PFAS regulations straightforward.

Experimental

Method Information

All samples were collected and prepared in accordance with the protocols outlined in EPA 537.1 and EPA 533.^{2,3} For the spiked samples, a reagent water previously tested, and verified to contain minimum levels of PFAS contamination was utilized to best represent sensitivity and recovery. Final drinking water samples were collected locally. For EPA 537.1 the Sep-Pak PS2 SPE cartridge (p/n: [WAT200610](#) < <https://www.waters.com/nextgen/global/shop/sample-preparation--filtration/wat200610-sep-pak-ps2-6-cc->

[cartridge-500-mg-sorbent-per-cartridge-80--m-30.html](#)) was utilized and for EPA 533 the Oasis WAX for PFAS 500 mg, 30 µm SPE cartridge (p/n: 186009568 <<https://www.waters.com/nextgen/global/shop/sample-preparation--filtration/186009568-oasis-wax-for-pfas-analysis-6-cc-vac-cartridge-500mg-sorbent-per.html>>) was utilized.

Analysis was performed on an ACQUITY™ UPLC™ I-Class PLUS FTN with PFAS Analysis Kit installed, coupled to a Xevo TQ-S micro Mass Spectrometer.

Results and Discussion

To achieve the parts per trillion (ppt) or ng/L measurements required by the NPDWR PFAS Regulations, the mass spectrometer must have sufficient sensitivity. Not only should the regulatory levels be easily detected and quantifiable, but a sufficient level below the regulated levels is also necessary for continual water quality monitoring purposes. Sample preparation using solid phase extraction (SPE) also increases method sensitivity by providing a sample enrichment effect. For sample preparation and analysis of both EPA 537.1 and 533 methods, a sample enrichment of 250x is achieved by concentrating 250 mL of the drinking water sample to a final sample extract volume of 1.0 mL. In combination with this sample preparation, the Xevo TQ-S micro Mass Spectrometer has excellent sensitivity to provide reliable and accurate detection of the six regulated PFAS down to the parts per quadrillion (ppq) levels. Figure 1 demonstrates overlays of the quantitation ion for the six regulated PFAS in the method blank, the lower-level calibration standard of 0.2 ng/L, a 0.5 ng/L sample extract, and a 4 ng/L sample extract (concentrations reported are sample equivalent concentrations). As demonstrated in Figure 1, there is minimal to no contamination in the method blanks, easily detected peaks with signal:noise values greater than 20 and easy to integrate peaks at each level.

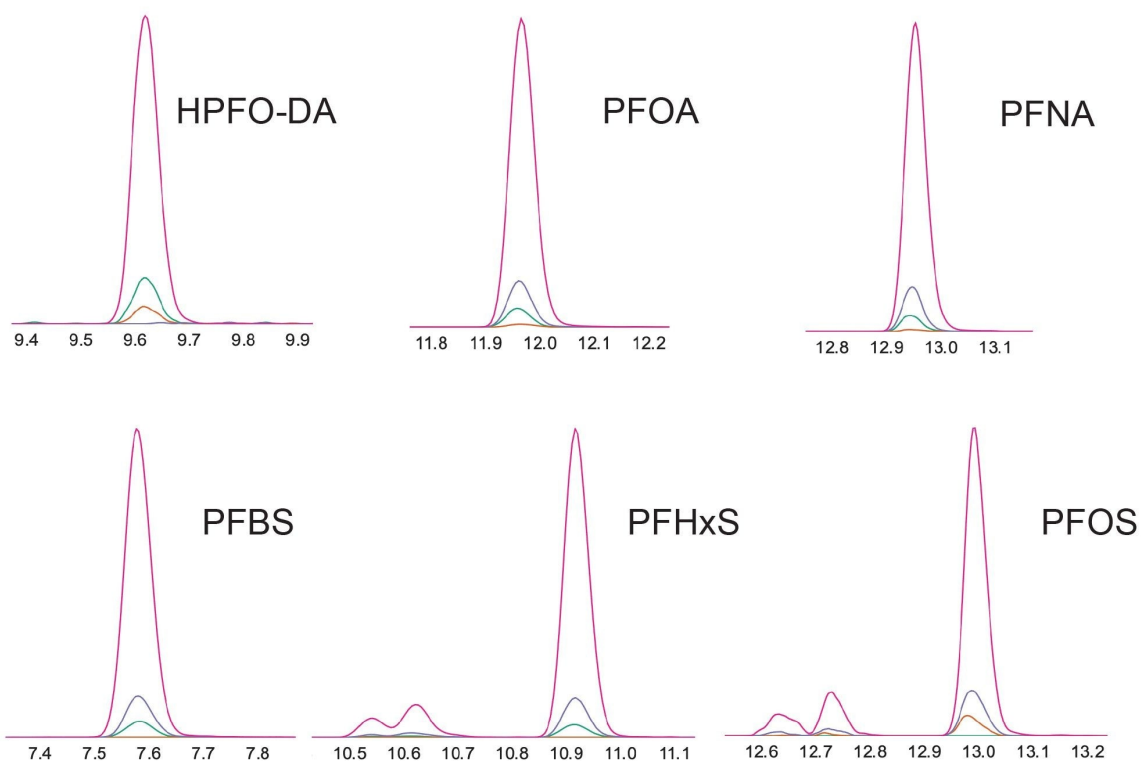


Figure 1. Chromatogram overlays of each regulated PFAS showing the method blank, 0.2 ng/L standard, 0.5 ng/L sample extract, and 4 ng/L sample extract.

Sample preparation performance is also important to providing accurate and reliable drinking water results for routine monitoring. The two EPA drinking water methods (537.1 and 533) utilize different SPE cartridges and protocols, so the recovery achieved is demonstrated for both methods in Figure 2. The recovery reported in Figure 2 is from reagent water spiked at the 4 ng/L regulatory level. The average recovery for EPA 537.1 on the Sep-Pak PS2 Cartridge was 107% (range of 103–115%) and the average recovery for EPA 533 on the Oasis WAX for PFAS Cartridge was 93% (range of 90–95%). Both SPE cartridges easily perform within the requirements of each EPA drinking water method.

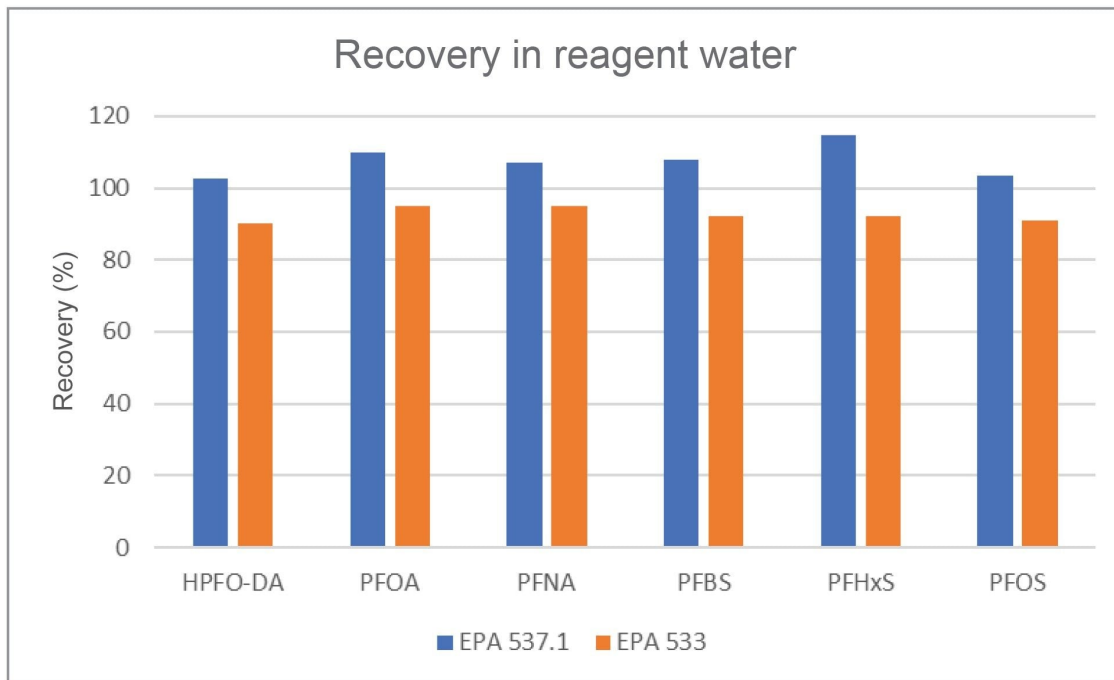


Figure 2. Percent recovery for the six regulated PFAS spiked at 4 ng/L in reagent water following both EPA 537.1 and EPA 533 methods.

Two locally collected drinking water samples were analyzed to demonstrate the workflow using authentic drinking water samples. Sample 1 followed the EPA 537.1 method, while Sample 2 followed the EPA 533 method. The results are reported in Table 1 compared to the individual regulated Maximum Concentration Levels (MCLs) and the Hazard Index. The Hazard Index was calculated using the following equation:

$$\text{Hazard Index} = (\text{HPFO-DA}_{\text{ppt}}) / 10_{\text{ppt}} + (\text{PFBS}_{\text{ppt}}) / (2000_{\text{ppt}}) + (\text{PFNA}_{\text{ppt}}) / 10_{\text{ppt}} + (\text{PFHxS}_{\text{ppt}}) / 10_{\text{ppt}}$$

Sample 1 was found to contain PFOA, PFOS, PFBS, PFHxS, and PFNA. Sample 2 was found to contain PFOA, PFOS, PFBS, and PFHxS. The Hazard Index for both samples were well below the regulated level of 1.0. Individual PFAS detected in Sample 1 were all within the new regulatory limits, whereas Sample 2 had a quantified level of PFOS that was over the new 4.0 ng/L MCL.

Compound	NPDWR MCL ng/L	Sample 1 ng/L	Sample 2 ng/L
PFOA	4	3.0	5.3
PFOS	4	0.53	0.92
HFPO-DA	10	0	0
PFBS	–	2.0	2.9
PFNA	10	0.39	0
PFHxS	10	0.63	0.60
Hazard Index	1.0	0.10	0.06

Table 1. Levels and calculated Hazard Index of six regulated PFAS detected in two drinking water samples compared to the National Primary Drinking Water Regulation.

Conclusion

A complete workflow for routine monitoring to easily satisfy the new PFAS Primary National Drinking Water Regulation is presented. Sample preparation following either EPA 537.1 or EPA 533 using the Sep-Pak PS2 or Oasis WAX for PFAS cartridges has been demonstrated to achieve maximum recovery. Analysis utilizing ACQUITY UPLC I-Class PLUS coupled to Xevo TQ-S micro MS and modified with the PFAS Kit to delay any contamination has been demonstrated to easily and accurately reach far below the lowest regulated MCL of 4 ng/L. In combination with Waters on site application training and certified reference materials provided by Waters ERA™, the PFAS solution for drinking water testing is easy to establish in both new and skilled laboratories.

References

1. Per- and Polyfluoroalkyl Substances (PFAS) Final PFAS National Primary Drinking Water Regulation. *US Environmental Protection Agency*. [PFAS <https://www.epa.gov/sdwa/and-polyfluoroalkyl-substances-pfas>](https://www.epa.gov/sdwa/and-polyfluoroalkyl-substances-pfas) . Accessed 10 April 2024.
 2. Shoemaker J., Dan Tettenhorst. Method 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS). *US Environmental Protection Agency*. November 2018.
 3. Method 533: Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry. *US Environmental Protection Agency*. December 2019.
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[Oasis WAX, WAX/GCB, and GCB/WAX for PFAS Analysis <https://www.waters.com/nextgen/global/products/sample-preparation/oasis-wax-for-pfas-analysis.html>](https://www.waters.com/nextgen/global/products/sample-preparation/oasis-wax-for-pfas-analysis.html)

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