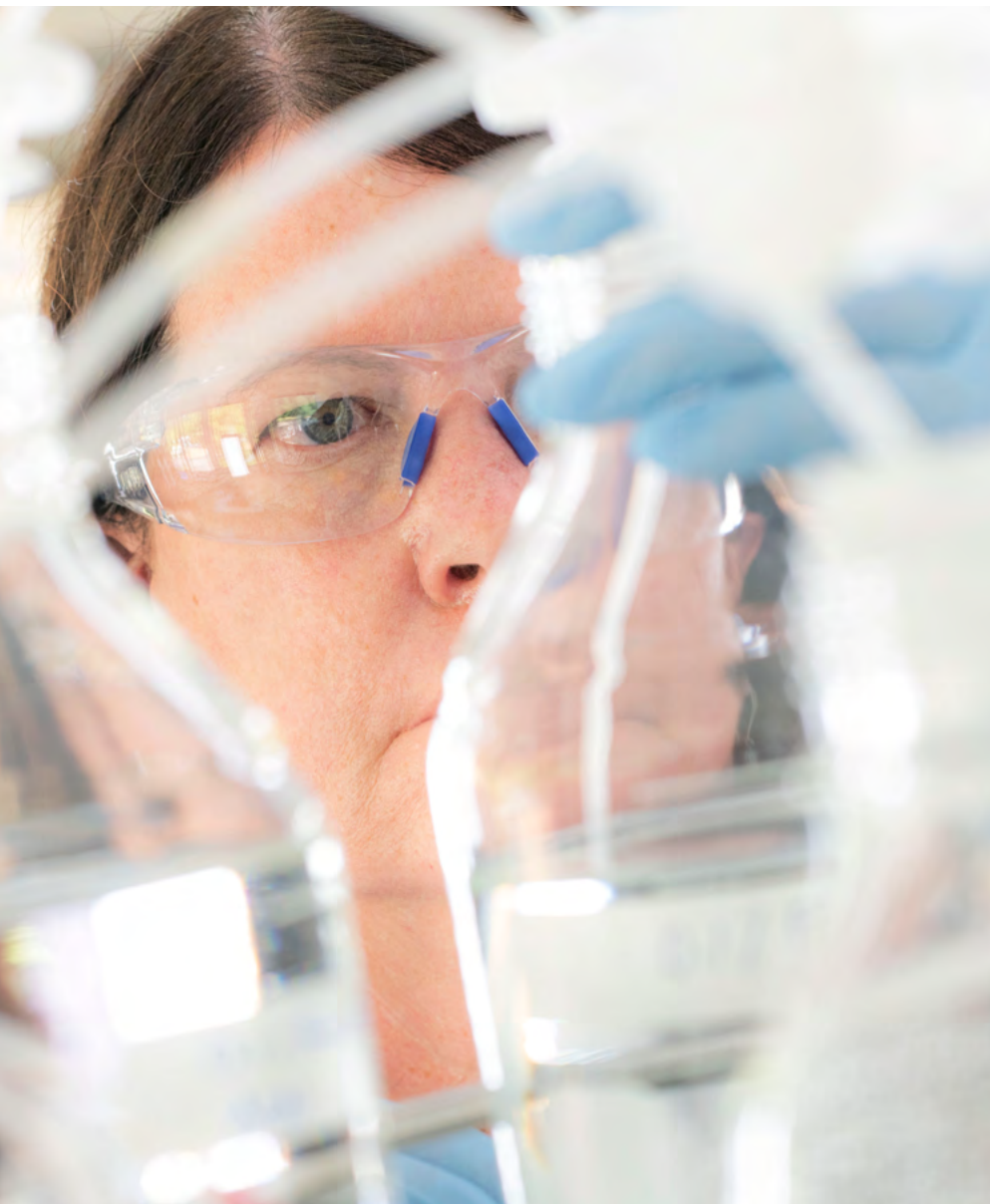# Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) Testing

Application Notebook

Waters

THE SCIENCE OF WHAT'S POSSIBLE.™

Introduction

EFFICIENT ANALYTICAL SOLUTIONS TO SUPPORT THE SAFETY OF FOOD, WATER SUPPLIES AND TO PROTECT THE ENVIRONMENT

Welcome to Waters' PFAS testing application notebook. The role of food safety, environmental protection and drinking water testing laboratories has never been more critical. Pollution, safety, sustainability and quality are of major concern to the public, governments, food industry and water companies. Inside this eBook you will find a compilation of our scientist's latest application notes, supporting your development and implementation of new testing methods and technologies for the analysis of PFAS in:

- Surface and ground waters
- Soil and sediments
- Drinking water
- Biological fluids
- Food

Trust in Waters to provide scalable application procedures and technologies to help you adapt quickly to challenges brought in by new regulations, new opportunities and competitive pressures. Improve internal efficiencies with less re-analysis and reduce waste, ensuring food and water safety and workflow optimization. By partnering with us you gain access to unmatched levels of application support and award-winning service which all aim to have your labs running effectively and consistently day-in-day-out.

Per- and polyfluoroalkyl substances (PFAS) are synthetic chemicals with a myriad of industrial and cosmetic uses. The original chemicals and their degradation products are highly persistent, toxic environmental contaminants that accumulate in humans, animals and the environment. They are characterized by their high chemical stability and enormous structural diversity. PFAS are ubiquitous in the aquatic environment and have been detected in air, soil, plants and biota. PFAS can persist in the environment for decades and are sometimes referred to as 'forever chemicals'.

Human biomonitoring has detected a range of PFAS in the blood of citizens across the globe. Human exposure to PFAS can occur in many ways, from consumer products and dust, to ingestion through contaminated drinking water and food grown on polluted land. The negative impacts on human health are extensive and continue to be investigated. In addition, PFAS pollution also affects ecosystems and generates costs through the need for remediation of polluted soil and water. Some countries impose regulatory or advisory limits on the concentration of PFAS in drinking and surface waters, and there are plans to set regulatory levels for PFAS in food in Europe.

CONTENTS

PFAS Testing in Environmental Samples

Per- and Polyfluorinated Alkyl Substances in Environmental Water Samples Using a Direct Injection Approach on Xevo TQ Absolute

Detection requirements for PFAS have been getting more challenging as advisory and regulatory limits, to protect public health and the environment, continue to be created and updated. For example, on June 15 2022, the US EPA tightened its lifetime health advisory levels in drinking water for perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS).

Using a simple direct injection approach for the determination of PFAS requires a highly sensitive mass spectrometer to reach the necessary performance criteria. The enhanced negative ion sensitivity of the Xevo TQ Absolute tandem quadrupole mass spectrometer allows for a reduction in the volume injected (10 µL) without compromising method performance. Analysis has been demonstrated in four types of water samples: drinking water, ground water, surface water, and influent wastewater. Method detection limits of 33 compounds (11 carboxylates, 10 sulfonates, 8 precursors, and 4 emerging) were determined to range from 0.8–2.0 ng/L.

Read the Full Application Note

APPLICATION BENEFITS

- The Xevo TQ Absolute provides increased sensitivity to detect and quantify PFAS in various water samples using a simple, direct injection approach, without needing to resort to large volume injection.

- Reducing the sample injection volume prolongs column lifetime and source cleanliness while ensuring suitable chromatography.

- Avoiding any sample preparation improves laboratory throughput, while also avoiding potential sources of contamination.

- The MS Quan application in waters_connect™ for quantitation analysis software platform allows for easy visual representation and review of data quality.
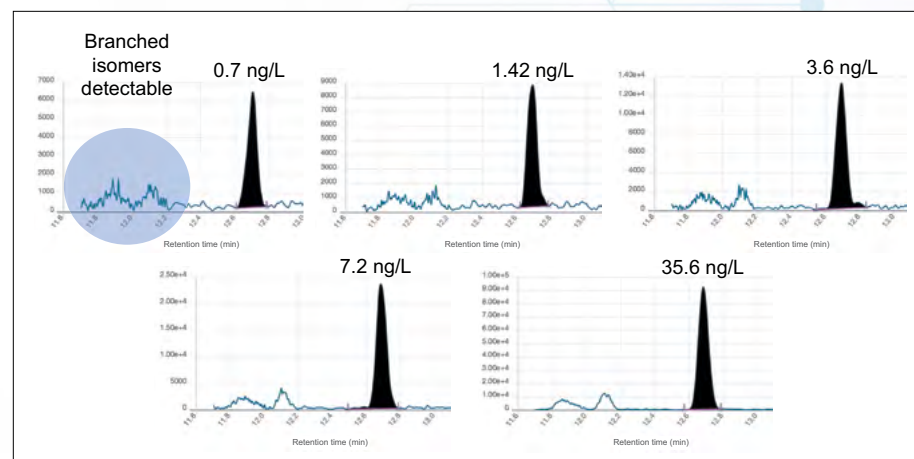
Chromatograms from the analysis of wastewater influent spiked with PFOS at different concentrations.

A Large Volume Injection Technique Using Simplified Sample Preparation for PFAS in Soils in Accordance with ASTM 7968

Soil is an important global reservoir for PFAS as it serves as a long-term contamination source to surface water, groundwater, the atmosphere, biota and ultimately, human exposure. Robust solutions for determining PFAS levels in the environment are required for monitoring programs, assessing the impact of remediation and source apportionment efforts. ASTM D7968 is a worldwide, consensus-standard method for PFAS testing and quantification in soil, sediment, and (bio)solids. The method uses isotope labelled standards, a quick solvent extraction, filtration and LC-MS/MS. It is not intended to generate an exhaustive accounting of the content of PFAS in difficult soil matrices.

In this application note, carried out in collaboration with US EPA Region 5 Laboratory, we describe the implementation of the ASTM D7968-17 method, using an ACQUITY™ UPLC I-Class PLUS, fitted with PFAS kit, and Xevo TQ-XS tandem quadrupole mass spectrometer, for the sensitive and reliable determination of PFAS in soil. Soil samples comprised of four types: sand, silt, lean clay, and fat clay. In addition to the 21 PFAS specified in the ASTM method, the scope was extended to include seven additional emerging PFAS (GenX, ADONA, 9Cl-PF3ONS, 11Cl-PF3OUdS, PFMBA, PFEESA, and NFDHA). All the performance parameters evaluated met the acceptance criteria given in the method.

Read the Full Application Note

APPLICATION BENEFITS

- This solution provided sensitive, accurate, efficient and high throughput analysis of PFAS in various soils and sediments utilizing very minimal sample preparation.

- It has been shown to be fit for purpose to achieve the requirements of the ASTM D7968 method, simplifying implementation into a routine laboratory setting.

- The emerging PFAS tested can easily be incorporated into the methodology.
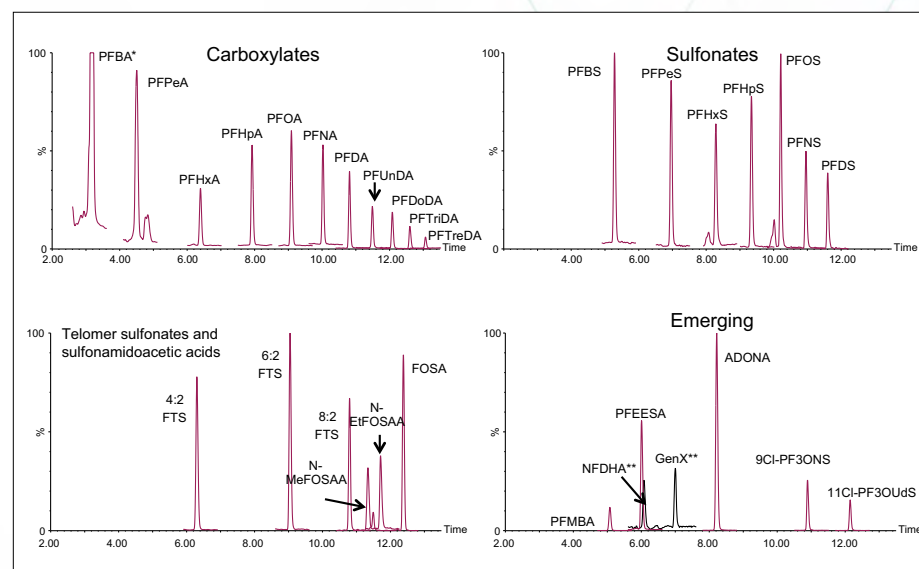
Chromatograms demonstrating the PFAS detected in a lean clay sample including emerging PFAS not currently specified in the ASTM 7968 method.

Large Volume Direct Injection Method for the Analysis of PFAS in Environmental Water Samples in Accordance with ASTM 7979

Numerous PFAS have been found ubiquitously in the environment. There is increasing concern about the occurrence, persistence, and fate of PFAS, and therefore better methods for identifying and quantifying PFAS in aquatic environments are urgently needed. ASTM D7979 is a standard test method for determination of PFAS in non-drinking water samples surface and ground water, sludge, influent and effluent wastewater) developed by the US EPA Region 5 Laboratory. ASTM D7979-17 is a direct injection LC-MS/MS method after some dilution with methanol. The scope of the method covers 21 PFAS and uses 14 isotope labelled standards for quantitation and quality control. In order to achieve low limits of detection required as there is no SPE concentration step, laboratories must commit to using instrumentation with high sensitivity and selectivity.

In this application note, carried out in collaboration with US EPA Region 5 Laboratory, we describe the implementation of the ASTM D7979-17 method for the sensitive and reliable determination of PFAS in surface (river) water, ground water, influent waste water, and effluent waste water. A number of newer PFAS (ADONA, 9Cl-PF3ONS, and 11Cl-PF3OUdS) were added to the scope. The method proved to be accurate and sensitive and met the acceptance criteria for quality control for all the PFAS except 6:2 FTS, which was impacted by solvent contamination issues.

Read the Full Application Note

APPLICATION BENEFITS
- The ASTM D7979-17 method uses minimal sample preparation and requires smaller sample volumes than the SPE approaches, which takes less time, uses less consumables and improves sample throughput.
- The use of the ACQUITY UPLC I-Class PLUS, fitted with PFAS kit, and Xevo TQ-XS provides the high sensitivity needed to implement the ASTM D7979-17 method to determine PFAS at the low ng/L levels range to meet regulatory requirements.
- The solution has been shown to be suitable for monitoring PFAS compounds in non-drinking water matrices, in an effective and efficient manner.
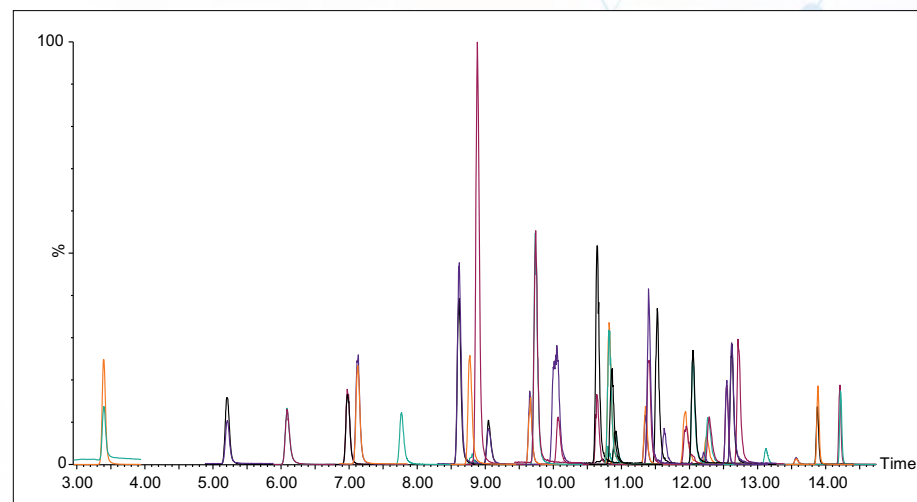
Overlay of all PFAS compounds analyzed in the method.

Analysis of Legacy and Emerging PFAS in Environmental Water Samples Using Solid Phase Extraction and LC-MS/MS

There are two types of approaches used for the monitoring of water samples using LC-MS/MS; direct injection of water samples and the use of a pre-concentration step with solid-phase extraction (SPE). In this application note, carried out in collaboration with University of Massachusetts, Amherst, we explored the performance possible using the second approach, using a method adapted from ISO 25101 for 35 PFAS. SPE was carried out Oasis Weak Anion Exchange (WAX) cartridges prior to LC-MS/MS on ACQUITY UPLC I-Class PLUS, fitted with PFAS kit, and Xevo TQ-S micro tandem quadrupole mass spectrometer. For the most part, detection limits were sub ng/L (ppt), reaching to the pg/L (ppq) levels. Ensuring contamination of the sample is kept to a minimum is essential when SPE is incorporated in to PFAS methods. The sensitivity, recovery and repeatability of the method, which uses isotope labelled standards for quantitation, was shown to be suitable for the monitoring for PFAS at very low concentrations in surface water, ground water, influent waste water, and effluent waste water.

Read the Full Application Note

APPLICATION BENEFITS

- The use of SPE with Oasis WAX cartridges provides a significant pre-concentration factor and hence the high sensitivity needed to determine PFAS at the sub ng/L levels to meet regulatory requirements.

- A robust and reliable solution for monitoring PFAS compounds in environmental water matrices was shown to be possible using an LC-MS/MS system based upon the Xevo TQ-S micro.
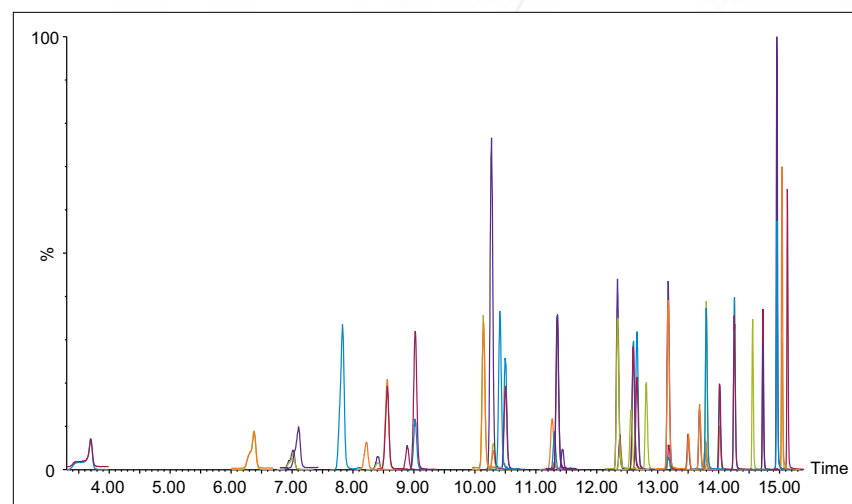
Overlay of all PFAS compounds analyzed in the method.

An Alternative Ionization Technique for PFAS Analysis: Evaluating UniSpray for Water and Soil Samples

Detection requirements for PFAS in environmental samples are typically in the ng/L or parts per trillion (ppt) range, requiring sensitive analysis methods, most typically LC-MS/MS. PFAS respond well using negative electrospray ionization (ESI-) and this has become the preferred method of analysis. Although this ionization technique works well, the increasingly stringent requirements for detection can benefit from any boost in signal that a technique like UniSpray could provide. UniSpray is a novel atmospheric ionization technique that allows for multimode ionization of both polar and non-polar analytes in a single injection. The column effluent is nebulized as it exits a grounded, heated probe. The spray is directed onto a stainless-steel pin that is held at high voltage. This creates smaller droplets than ESI, leading to increased desolvation of ions. The nebulized flow bends around the surface of the impactor pin into the sample cone due to the Coanda effect. This mechanism allows for increased ionization and sampling efficiency.

This application note describes the evaluation of UniSpray ionization for analysis of PFAS in various environmental samples, as well as showing a direct comparison to typical electrospray ionization (ESI) for the same set of samples.

Read the Full Application Note

APPLICATION BENEFITS

- Increased response for PFAS when using UniSpray ionization in environmental samples making required detection limits easier to achieve.

- A possible reduction in the requirements for sample preparation prior to injection where allowable by regulations and guidelines.

- More robust and accurate results at lower concentrations with the potential for lower Limits of Quantitation.

- An accurate and robust alternative technique for the routine analysis of PFAS in environmental samples.
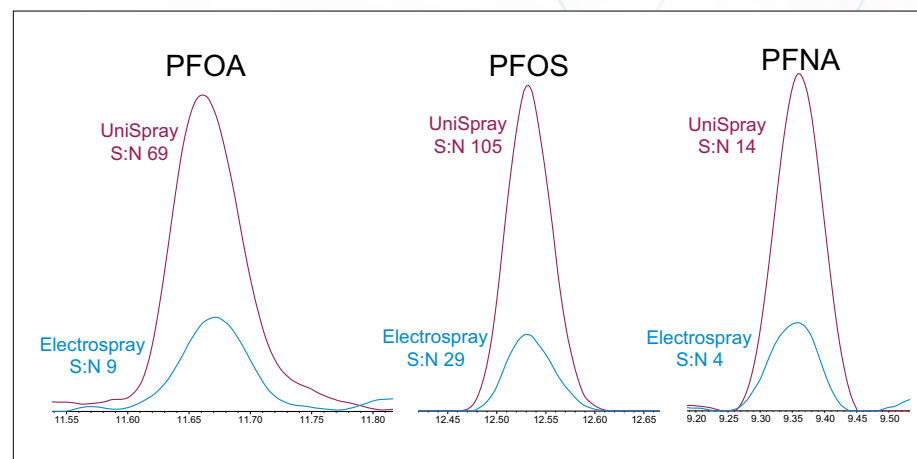
Signal:Noise (S:N) and peak response comparison between UniSpray (red) and Electrospray (blue) demonstrated with PFOA, PFOS, and PFNA peaks.

Approaches to Non-targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) in Environmental Samples

PFAS are an important class of toxic environmental contaminants. From an analytical point of view, the limited availability of authentic reference standards considerably limits the number and kind of routinely analyzed PFAS. Over 9000 PFAS exist, but reference standards are available for less than 2% of compounds. Non-targeted approaches to analysis using liquid chromatography—high-resolution tandem mass spectrometry (LC-HRMS) is therefore an essential technique for increasing the analytical coverage of PFAS present in environmental samples and to help identify the presence of new compounds and transformation products.

In this study, workflows for non-targeted analyses for PFAS are demonstrated using a Xevo G2-XS QTof coupled with an ACQUITY UPLC I-Class PLUS modified with the PFAS Analysis kit. In-house PFAS reference libraries were prepared and used to assign putative identities to the compounds detected in wastewater and soil samples. Discovery of PFAS not present in the UNIFI™ libraries can be achieved by automatic searching of external databases, aided using additional software tools including common fragments, neutral loss, and mass defect searching.

Read the Full Application Note

APPLICATION BENEFITS

- Local PFAS accurate mass libraries with over 4,000 compounds that are easily customizable.

- An analytical solution that uses multiple attributes for component putative identification increasing confidence in the results for legacy and emerging PFAS in environmental samples.

- The presented non-targeted methodology can be useful in various scenarios including, but not limited to, discovery of novel PFAS compounds, a better understanding of PFAS contamination in the environment, and source fingerprinting for remediation purposes.
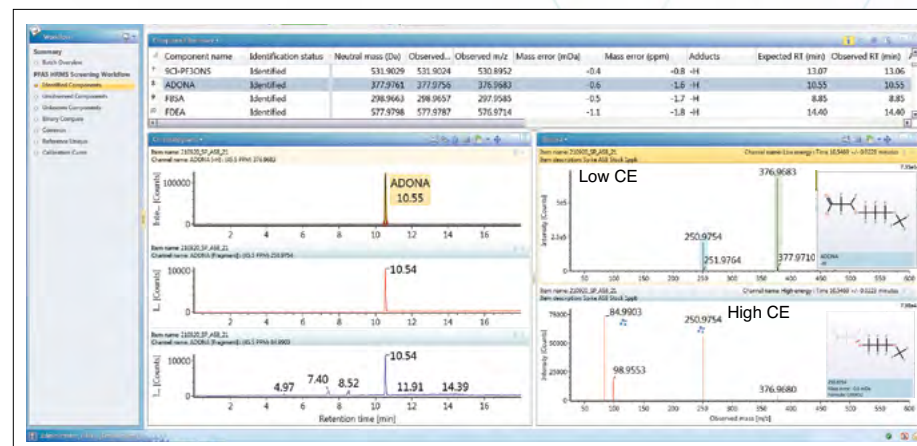
Identification of ADONA in a wastewater sample extract at 1000 ng/L, 30 µL injection (0.03 ng injected).

PFAS Testing in Drinking Water

Oasis WAX for Extraction of Per- and Polyfluorinated Alkyl Substances (PFAS)

EPA method 533 complements EPA method 537.1 and can be used to test for 11 additional PFAS. Using both methods, a total of 29 unique PFAS can be effectively measured in drinking water. EPA Method 533 is a method for determination of PFAS in drinking water, which uses SPE to provide a pre-concentration step prior to LC-MS/MS. Unlike in EPA method 537.1, this method uses a mixed-mode SPE sorbent, isotope dilution and includes 25 PFAS, incorporating both short chain and emerging compounds.
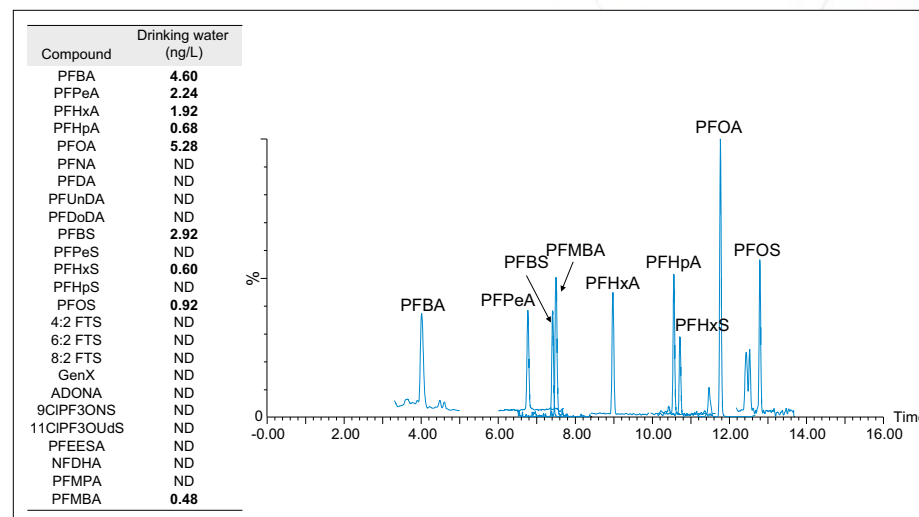
In this application note, we explored the performance of the EPA method 533 for the determination of PFAS in drinking water, using the same conditions as in previous work (720006471EN). The Oasis WAX cartridge, containing a mixed-mode sorbent with a pKa of ~10 (p/n: 186009568), is a direct equivalent to the chemistry mandated in EPA method 533, and meets the necessary analytical requirements mandated by the method. The accuracy, demonstrated by the measured recovery, and repeatability, were determined to be within the required range for all the PFAS in the method.

Read the Full Application Note

APPLICATION BENEFITS

- Using Oasis WAX SPE along with the ACQUITY UPLC I-Class PLUS and Xevo TQ-S micro provides a system solution that meets and exceeds the requirements prescribed in EPA method 533.

| Compound	Drinking water (ng/L)
PFBA	4.60
PFPeA	2.24
PFHxA	1.92
PFHpA	0.68
PFOA	5.28
PFNA	ND
PFDA	ND
PFUnDA	ND
PFDoDA	ND
PFBS	2.92
PFPeS	ND
PFHxS	0.60
PFHpS	ND
PFOS	0.92
4:2 FTS	ND
6:2 FTS	ND
8:2 FTS	ND
GenX	ND
ADONA	ND
9ClPF3ONS	ND
11ClPF3OUdS	ND
PFEESA	ND
NFDHA	ND
PFMPA	ND
PFMBA	0.48

PFAS detected in drinking water.

Perfluorinated Alkyl Substances (PFAS) in Drinking Water: Extraction Using the PS2 Cartridge in Accordance with EPA 537.1

PFAS have been detected in drinking water and drinking water sources across the world. Their chemical properties have made PFAS difficult to treat and remove using conventional water treatment processes. Driven by concern over the different possible health effects, some countries have imposed regulatory or advisory limits on the concentration of PFAS in drinking water. In the USA, both government and private laboratories can effectively determine PFAS in drinking water using EPA methods 533 and 537.1, which were developed with particular attention to accuracy, precision, and robustness and have been through multi-lab validation and peer review. Method 537, used originally as the approved PFAS method to support UCMR 3 (2013-2015), was updated to method 537.1 in 2018 to include emerging PFAS. EPA 537.1 specifies the use of a styrene divinylbenzene (SDVB) substrate for solid phase extraction (SPE) in a prescribed manner without modifying the method. The method covers 18 PFAS, including legacy carboxylate and sulfonates, like PFOA and PFOS, as well as the addition of four emerging PFAS, including GenX (HFPO-DA).

In this application note, we explored the performance of the EPA method 537.1 for the determination of PFAS in drinking water. The Sep-Pak PS2 Cartridge (pn WAT200610) is a direct equivalent to the chemistry mandated in the 537.1 method and meets all the necessary analytical requirements.

APPLICATION BENEFITS

- Demonstration of the Sep-Pak PS2 SPE cartridge to determine PFAS in drinking water according to EPA method 537.1.
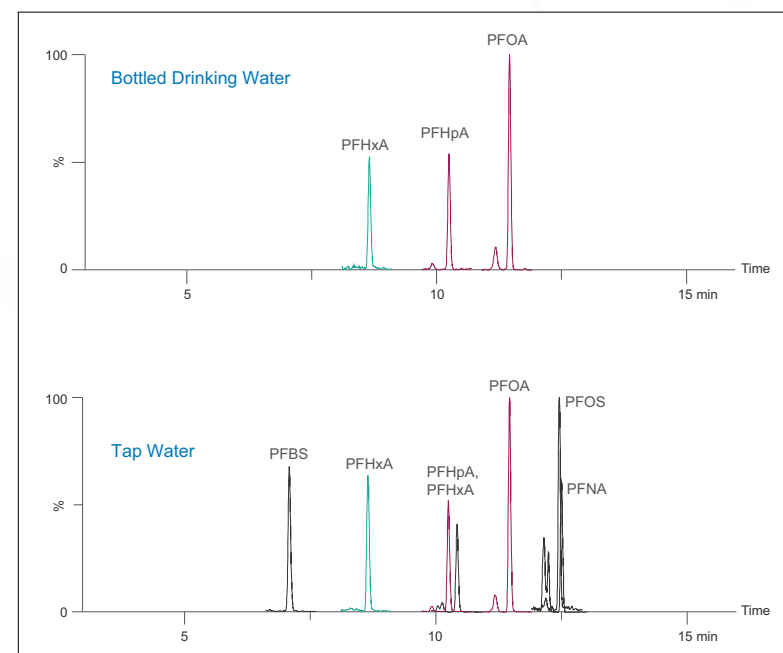
Results from the analysis of two samples of drinking water.
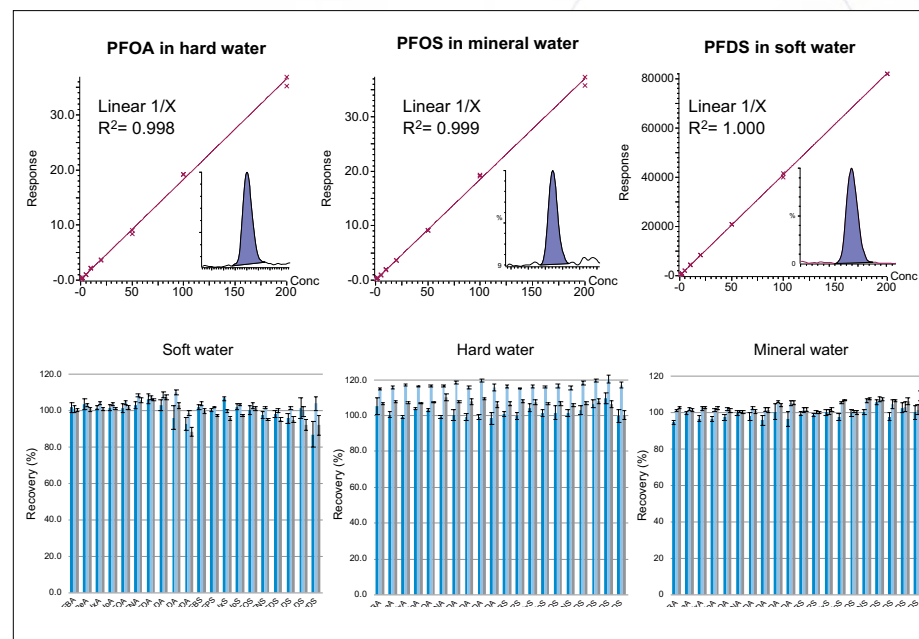
Read the Full Application Note

Determination of PFAS in Drinking Water by Direct Injection UPLC-MS/MS Suitable for the EU Drinking Water Directive 2020/2184

The European Union (EU) has now included a set of 20 PFAS compounds in the updated Drinking Water Directive 2020/2184. It provides a parametric value Sum of PFAS of 0.1 µg/L for a selection of 20 PFAS that are considered a concern in water intended for human consumption. The 20 individual substances included in the parameter Sum of PFAS cover a wide range of PFAS with chain lengths from C_4 to C_{13}.

Instruments with high sensitivity are needed to detect PFAS at concentrations in the parts per trillion (ppt) range. PFAS respond well using electrospray ionization in negative ion mode (ESI-) but here we evaluate whether an alternative, UniSpray, can provide greater sensitivity. This application note demonstrates the performance of the ACQUITY UPLC I-Class PLUS coupled with a Xevo TQ-XS, fitted with UniSpray, for the analysis of three types of drinking water matrices: soft tap water, hard tap water, and mineral water. The method was based upon direct injection, after addition of some acidified organic solvent, and used stable isotope analogues as internal standards. UniSpray provided consistent gains in sensitivity; mean increase of 18x peak area and 5x signal to noise ratio. Overall, the mean for trueness of the method was 89 to 112% across the types of water and RSDs were all at or below 14%.

Read the Full Application Note

APPLICATION BENEFITS

- Provides a solution using a direct injection, UPLC-MS/MS method suitable for the determination of all 20 PFAS with an LOQ of 1 ng/L per compound, which is significantly lower than the limit in the 2020 EU Drinking Water Directive, without the need for lengthy clean-up or concentration steps.

Recovery and repeatability for the 20 PFAS in the method from analysis of soft, hard, and mineral water spiked at 2, 10, and 100 ng/L

Evaluation of the Performance of a UPLC-MS/MS Method for the Determination of PFAS in Drinking Water by Interlaboratory Study

Waters previously developed a method for the determination of per-and polyfluoroalkyl substances (PFAS) in drinking water based on direct injection liquid chromatography with tandem quadrupole mass spectrometry (LC-MS/MS) that was suitable for checking compliance with the revised EU Drinking Water Directive. The method benefited from direct injection of drinking water, after some dilution with acidified organic solvent, and stable isotope analogues as internal standards. This application brief shows the successful evaluation of the performance of that method by interlaboratory study. A reference material containing twenty PFAS was sent to seven Waters laboratories, along with standard solutions containing native PFAS compounds and isotopically labelled analogues. The reference material was diluted upon receipt to create a test sample with the PFAS compounds at 0.01 μg/L. All the laboratories used ACQUITY UPLC systems, fitted with the PFAS Analysis Kit, and Xevo TQ-XS tandem quadrupole mass spectrometers with the UniSpray ion source. No assessment of the results for perfluorobutanoic acid (PFBA) was possible due to contamination experienced by most laboratories. For the other PFAS, the trueness of the method was determined to be within the range of 96 to 110%. Close agreement was observed with the repeatability within each laboratory and the reproducibility between laboratories both being <20% RSD.

Read the Full Application Note

APPLICATION BENEFITS

- The method was successfully implemented, in seven different Waters laboratories, with varying levels of experience of PFAS analysis.

- The performance of the method, as demonstrated by this interlaboratory study, provides users with confidence in the ease of implementation and its suitability for testing drinking water for PFAS for regulatory compliance.

Summary of the values for repeatability (blue) within each laboratory (RSDr) and for reproducibility (orange) between laboratories (RSD$_{RL}$) for PFAS from the analysis of the water test sample at 0.01 μg/L

PFAS Testing in Biological Fluids

Extracting and Analyzing PFAS from Human Serum

Due to the bioaccumulative nature of per- and polyfluorinated alkyl substances (PFAS), monitoring levels in human biofluids can help gain understanding into exposure levels and pathways. For some individual PFAS, adverse health effects have been described or suspected. Since PFAS are not rapidly metabolized or removed from the body, we can get an understanding of these levels of exposure by studying serum content. Therefore, sensitive, reliable and accurate methods are needed to continue our understanding of the burden of PFAS in humans for purposes such as occupational health monitoring, environmental exposure monitoring, and toxicology studies.

In this work we developed an optimized solid phase extraction (SPE) method for the extraction of PFAS from human serum utilizing Oasis WAX µElution 96-well plates. Analysis was performed on Xevo TQ-S micro coupled with an ACQUITY UPLC I-Class PLUS, fitted with the PFAS Analysis Kit. An ACQUITY HSS T3 Column provided crucial separations of PFAS from serum steroid sulphate interferences. Method verification was performed using six different pooled lots of human serum. In addition, the performance of the optimized method was successfully verified using a standard reference material from NIST (SRM 1957). The final method proved to be sensitive, accurate and robust for the extraction and determination of 30 PFAS from a variety of different common PFAS chemistry classes.

Read the Full Application Note

APPLICATION BENEFITS

- An optimized protocol was devised for efficient sample clean up using Oasis WAX 96-well plates.

- Robust method for accurate determination of a range of different PFAS in human serum using the Xevo TQ-S micro, suitable for studying and understanding human exposure levels to PFAS.

- This methodology can be implemented in various types of laboratories to understand levels and trends of PFAS in serum samples for purposes such as occupational health monitoring, understanding human exposure levels, source fingerprinting, exposure pathways, and understanding impacts on humans based on regional and/or epidemiological differences.
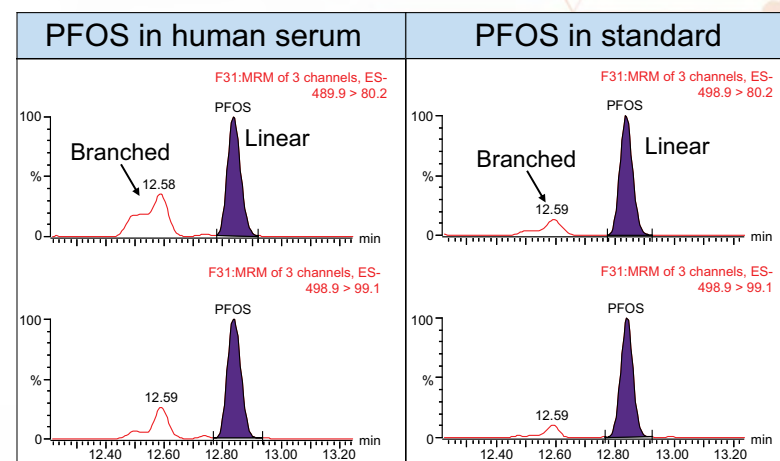
Comparison of the ratio of branched and linear PFOS isomers in human serum (left) and analytical standard (right).

A Semi-Automated Extraction of PFAS from Human Serum and Plasma Using Otto™ SPEcialist

PFAS persist in the environment and exposure in humans can occur by consuming PFAS-contaminated water or food or via contact with dust or household products that contain PFAS. There is a growing need for high throughput monitoring of human exposure towards a wide spectrum of PFAS in the general population including determining temporal trends of the burden of PFAS in human blood plasma samples.

In a previous application note, we have developed a method for quantification of representative PFAS in human serum and plasma for biomonitoring and epidemiological studies of human health effects of PFAS exposure. To provide higher throughput, the method was transferred onto the Otto SPEcialist positive pressure manifold, which provides a more efficient and reliable solution than traditional negative pressure SPE manifolds. The same SPE protocol and instrument methods described in Waters Application Note 720007114 were used for the evaluation of the Otto SPEcialist. The performance of the new, semi-automated method was evaluated for the analysis of both human serum and plasma. The Otto SPEcialist performed similar to, and in some cases better than, the previous negative pressure manifold in terms of accuracy. Otto SPEcialist also was shown to have high precision with low %RSD values for replicate extractions of both human serum and plasma.

APPLICATION BENEFITS

- The new method, using the Otto SPEcialist was shown to be suitable for the extraction of PFAS from human serum and plasma.

- The use of the Otto SPEcialist for the analysis of human biofluids for PFAS can provide laboratory efficiency while maintaining confidence in high quality results.
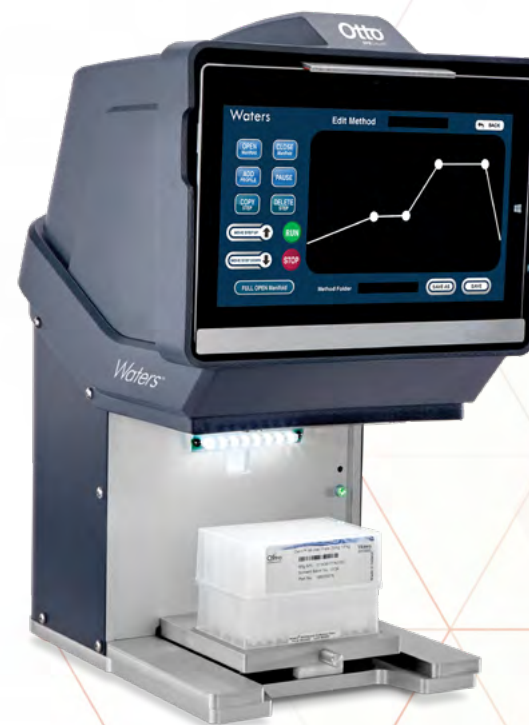
Otto™ SPEcialist positive pressure manifold

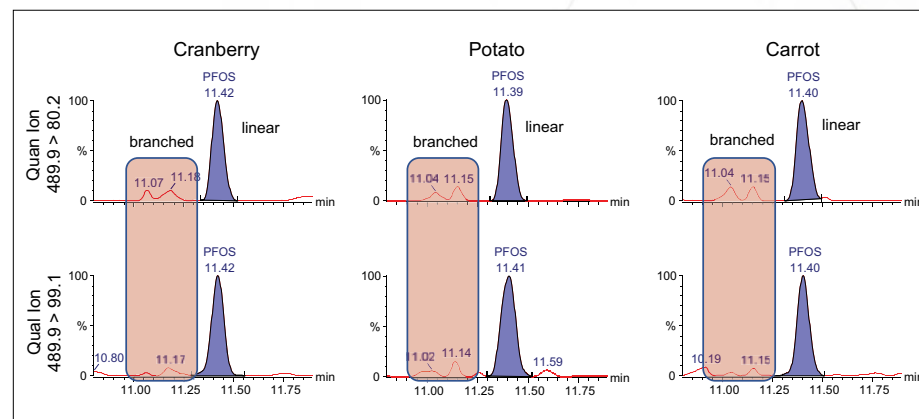**Read the Full Application Note**

PFAS Testing in Food

QuEChERS Extraction of Per- and Polyfluoroalkyl Substances (PFAS) from Edible Produce with Sensitive Analysis on Xevo TQ-XS

The environmental impact of PFAS is now well known from the prevalent usage of these compounds in everyday products. Potential routes of human exposure include air inhalation, dust ingestion, dermal absorption, and dietary intake, including food and migration through food contact substances and from food packaging materials. Cultivating agricultural produce using PFAS contaminated water, biosolids and soils can lead to the uptake of these compounds into the edible portions of fruit and vegetables. Some countries impose regulatory or advisory limits on the concentration of PFAS in water and indicative levels for some PFAS have been set in food in Europe. To investigate the contribution of dietary intake to PFAS exposure, reliable methods are needed to generate more occurrence data. Methods will also be needed to check regulatory compliance when maximum limits are introduced. Such methods must have sufficient sensitivity to detect PFAS at the very low concentrations likely to be found in food.

In this application note, we evaluated and modified an existing straightforward approach the analysis of fresh produce. The original FDA C-010.01 method, based on QuEChERS, was implemented for extraction of PFAS using DisQuE dispersive solid phase extraction (dSPE) products followed by highly sensitive LC-MS/MS analysis on ACQUITY UPLC I-Class PLUS coupled to Xevo TQ-XS. The performance of the method was successfully evaluated in five different commodity types; lettuce, strawberry, cranberry, carrot, and potato.

APPLICATION BENEFITS

- An efficient and simple approach to the determination of 30 PFAS in edible produce utilizing QuEChERS and dSPE clean-up.
- The Xevo TQ-XS provides enough sensitivity to detect PFAS at sub-ng/g levels suitable for checking compliance with regulatory levels and to produce data for risk assessment.

Detection of branched and linear PFOS isomers in 0.05 ng/g cranberry, potato, and carrot matrix.

Read the Full Application Note

Total Workflow for the Sensitive Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Fish, Meat, Edible Offal, and Eggs

There have been extensive investigations into estimating the dietary intake of PFAS in humans, which have shown that seafood, meats, and other products of animal origin were most likely to make a significant contribution to human exposure. To investigate the contribution of dietary intake to PFAS exposure, reliable methods are needed to generate occurrence data and methods are also needed to check compliance with regulatory limits.

There are a variety of approaches available for the extraction of PFAS from meat, fish, and similar products of animal origin, which can be more challenging matrices than fresh produce. For this application note, we chose alkaline extraction (sodium hydroxide in methanol) of the sample followed by solid phase extraction (SPE) clean-up on Weak Anion Exchange (WAX) cartridges. Analysis was performed on an ACQUITY UPLC I–Class PLUS modified with PFAS Analysis Kit, coupled to a Xevo TQ-XS to fit the highly sensitive analysis requirements. The performance of the method was evaluated for the determination of 30 PFAS in six different challenging food matrices; salmon, tilapia, ground beef, beef liver, beef kidney, and egg. Recoveries were within FDA criteria for all compounds except the C_{13} and C_{14} carboxylates. Utilization of the isotope dilution method takes the recovery into account and allows for accurate correction of recovery during calculation of PFAS concentration in samples.

Read the Full Application Note

APPLICATION BENEFITS

- A single extraction method, with selective clean-up can be utilized for a large suite of PFAS from a variety of food matrices to allow for better monitoring and understanding of the environmental impact of PFAS on our food sources.

- Sensitive and accurate analysis of extracts was performed using the Xevo TQ-XS allowing for detection and quantitation limits in the sub-ng/g levels suitable for checking compliance with regulatory levels and to produce data for risk assessment.

PFAS	Liver (ng/g)	Egg (ng/g)
PFOS linear	0.52	–
PFOS branched	0.24	–
PFOS (total)	0.76	–
PFPeA	–	0.18
PFHxA	–	0.25
PFHpA	–	0.29
PFOA	–	0.13
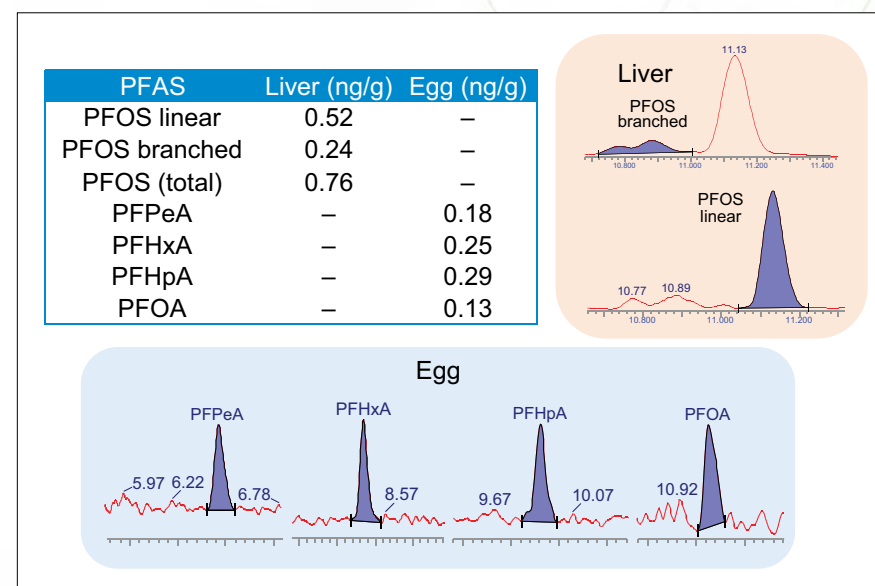
PFAS detected in samples of beef liver and egg purchased in local grocery stores.

Matrix Matching or Isotope Dilution? A Comparison of Two Quantitation Approaches to Determine PFAS in Cow's Milk

The application of water and biosolids contaminated with PFAS to fertilize produce feed crops for livestock has greatly impacted the quality of dairy products farmed from these animals. Hence, it is crucial to have sensitive and accurate analytical methods to monitor PFAS levels in cow's milk to establish occurrence data for dietary risk assessment and to check compliance with any regulatory limits.

In this application brief, PFAS analysis in cow's milk was successfully performed by applying the same QuEChERS approach previously used for the analysis of edible produce. The performance of the method was evaluated in store bought cow's milk and proved to be accurate and robust for a range of 30 PFAS compounds of varying chemistry classes. Matrix effects can be the Achilles heel for analysis of foodstuffs using LC-MS/MS, as they can have a significant impact on quantitation. Here, matrix matched calibration and solvent based isotope dilution calibration curves were compared, with both proving to be appropriate techniques for calculating compound concentrations in samples.

Read the Full Application Note

APPLICATION BENEFITS

- A time efficient and simple extraction of 30 PFAS from cow's milk utilizing a QuEChERS extraction method and dSPE clean up.

- Sensitive analysis on the Xevo TQ-XS to detect PFAS at sub-ng/g levels to effectively monitor PFAS exposure in cow's milk.

- The choice of approach to quantitation (matrix matched or isotope dilution) can be decided based upon specific laboratory needs and can be readily set up in the instrument software.
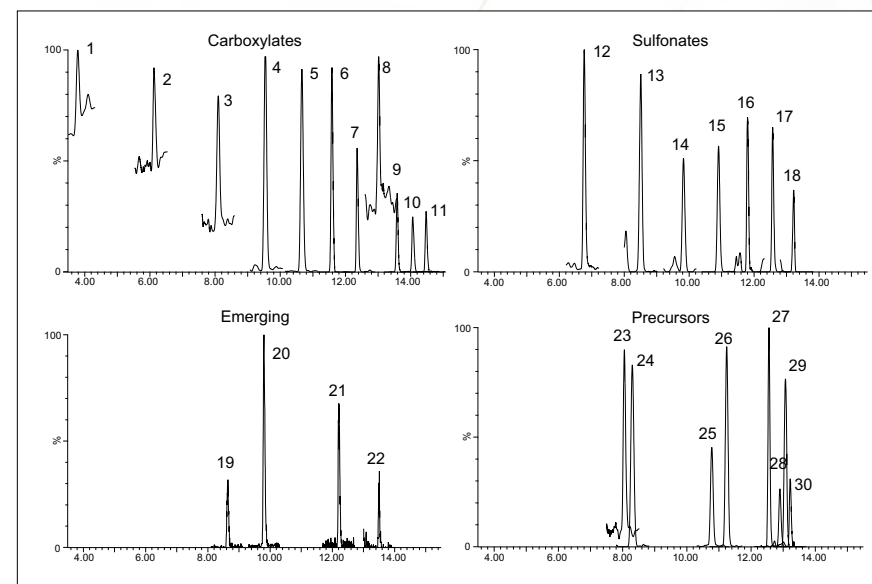
Chromatograms showing the detection for each PFAS in the 0.1 ng/g spike in milk.

Links to other Useful Materials

- Adapting to Challenging PFAS Analysis with Focused Analytical Workflow Solutions

- PFAS Analysis Solutions

- PFAS Analysis Solutions Ordering Information

- Startup Guide for the Analysis of Perfluorinated Alkyl Substances (PFAS) in Environmental Samples

- Improving Negative Ion Detection for Tandem Quadrupole Mass Spectrometry

- Benefits of waters_connect MRM Processing Application, MS Quan

- ERA's website for PFAS PT, CRM & QRs

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