The Determination of Per- and Polyfluorinated Alkyl Substances (PFAS): Answers to Frequently Asked Questions

TECHNICAL BULLETIN

Perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA) and related per- and polyfluorinated alkyl substances (PFAS) continue to receive a substantial amount of attention from environmental practitioners and regulatory bodies, not only because they are recognized as ubiquitous environmental contaminants, but also because these compounds persist, bioaccumulate and cause toxicity in some animal studies. PFAS are of particular interest because of their emergence as compounds of environmental concern at an increasing number of sites across North America.

Introduction

Over the last several years, requirements for PFAS analyses, and the ability to use the resultant data for risk assessment and management, as well as remedial decisions have increased at an extraordinary rate. Because of the recognized challenges associated with proper sampling and analysis of these compounds, questions about sampling, analysis and correct interpretation of analytical results provided by laboratories have also increased. It is important that the laboratory industry respond not only with reliable, defensible and comparable analytical results, but also consistent responses to these questions to ensure a sound and uniform decision making framework for the data user.

PFAS Naming Conventions

What is PFAS?

PFAS is an acronym for the entire class of per- and polyfluorinated alkyl substances. The class of compounds eencompasses a whole family of man-made chemicals used in industry and consumer products worldwide since the 1950s. PFAS represents over 3,000 substances that contain a carbon and fluorine atom backbone. Many are extremely persistent and mobile in the environment.

The most commonly studied PFAS are perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS). Then next most studied are perfluorohexane sulfonic acid (PFHxS) and perfluorononanoic acid (PFNA). PFAS, as an abbreviation, is a plural noun. It should be noted that the acronym PFCs (perfluorinated compounds) is **no longer used** as it is poorly defined and does not capture the polyfluorinated compounds which are being increasingly recognised as environmental contaminants.

Sample Contamination

The drive to decrease PFAS criteria to low ng/L has resulted in the need for increased sensitivity in the analyses.

What are common sources of sample contamination?

Sampling Equipment

A known source of background contamination is the presence of fluoropolymers, such as polytetrafluoroethylene (PTFE) compounds in sampling equipment such as pump tubing.

Sample Containers

Glass containers are not suitable for the collection and storage of samples due to the potential for adsorption of PFAS on the walls of these containers.

Samples should be collected in high density polyethylene (HDPE) bottles, provided by the laboratory and fitted with an unlined (Teflon-free) polypropylene screw cap.

Because of the ubiquitous nature of PFAS compounds in many modern materials, all batches of sample containers provided by Maxxam, used for collecting samples for PFAS determinations, are “proofed” to demonstrate that they are PFAS-free prior to sampling.

Field / Wash Water

Water used in the field to generate quality control (QC) samples should be PFAS-free. Maxxam will provide for a fee, PFAS-free water that has been “proofed” by the laboratory.

Other Sources of Contamination

There are reports that some personal care products such as cosmetics, moisturizers and sunblock contain PFAS and should not be worn by the sampler to limit any potential contamination.

Sample Preservation

What is the purpose of Trizma preservative?

USEPA methods for regulated drinking water typically use sample preservatives to prevent microbial degradation (e.g. $CuSO_4$, DZU and NaHSO4) and to dechlorinate (e.g. ascorbic acid, Trizma buffer and Na_2SO_3) at the time of sampling.

Trizma buffer was selected by Maxxam as the preferred preservative as it yields recoveries of PFAS between 92 – 108% with excellent precision. It has the added benefit of buffering the sample at pH 7.

Sample Handling

What is the procedure for handling turbid samples or samples containing sediment?

Turbid samples should either be centrifuged or allowed to settle prior to sampling the supernatant. In situations where low levels of PFAS are anticipated, the whole bottle is extracted. It should be recognized that this potentially introduces a high bias because PFAS that is adsorbed to the particulate material may contribute to the total PFAS concentration

Should a sample containing a lot of sediment be filtered?

No. The accepted industry best practice is that samples collected for PFAS determinations should not be filtered as it has been demonstrated that significant PFAS loss can occur due to adsorption on to the surface of the filter (see below).

PFAS	Filtered (ng/L)	Centrifuged (ng/L)
PFOS	29.3	96.6

Quantifying PFAS

What are the important considerations when calculating PFAS?

Isotope Dilution Mass Spectrometry (IDMS)

IDMS provides greater accuracy than other calibration methods because it compensates for any matrix effects that may suppress recovery of the parameters being measured.

Simply put, the recovery of the labeled compound, which is not naturally present in the sample, is an exact representation of the recovery of the native compound which is present in the sample. IDMS involves using the isotopically labelled analogue for each target compound determined.

Direct Injection vs. Solid Phase Extraction (SPE)

Water samples containing low levels of PFAS undergo a solid phase extraction (SPE), to extract, clean up and concentrate the parameters of concern. The extract is then analysed by isotope dilution liquid chromatography coupled with tandem mass spectrometry (LC/MS/MS).

Water samples with higher PFAS concentrations may be analysed by direct injection isotope dilution (LC/MS/MS).

Soil, solids and tissues are homogenised, followed by a solid/liquid extraction. Interferences are removed from the liquid extract using SPE. The extract is then concentrated and analysed by isotope dilution LC/MS/MS.

Why is it important to distinguish between linear and branched isomers of specific PFAAs?

Most environmental contamination by PFAS is due to technical mixtures, not the just the linear isomer. Therefore, it is important to understand if PFAS such as PFOS was quantified using only the linear isomer or a technical mixture of the linear and branched isomers. If the calibration of the measurement system was performed using only the linear isomer, the final result for may be significantly biased (40 – 80%).

In Summary

This bulletin lists some of the most frequently asked questions received by Maxxam regarding PFAS sampling and analysis. As technology advances and our knowledge surrounding the science of PFAS in the environment increases, new questions arise. For any enquiries concerning sampling and analysis for PFAS, please contact Maxxam's experts here.

About Us

Maxxam is a leading North American provider of analytical services and solutions to the energy, environmental, food, Industrial Hygiene and DNA industries. We are a member of the Bureau Veritas Group of companies – a world leader in testing, inspection and certification services. We support critical decisions made by our customers through the application of rigorous science and the knowledge and expertise of over 2,500 employees.

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