
# 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 with not only 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.

## 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 laboratory equipment. Instead, it is recommended to use high density polyethylene (HDPE) or silicone tubing materials.

Sample Containers

Polypropylene and glass containers are not suitable for the collection and storage of samples; this is due to the potential adsorption of PFAS on the walls of these containers.

Samples should be collected in 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” by Maxxam to demonstrate that they are PFAS-free before 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.

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## Sample Preservation

**What is the purpose of Trizma preservative?**

Regulated EPA drinking water methods typically use sample preservatives to prevent microbial degradation (e.g., $CuSO_4$, DZU, $NaHSO_4$) and to dechlorinate (e.g., ascorbic acid, Trizma buffer, $Na_2SO_3$) at the time of sampling.

Trizma buffer was selected as the preservative as it yields recoveries of 92 – 108% with excellent precision. It had the added benefit of buffering the aqueous sample so that the pH effects were controlled at approximately 7 and removed free chlorine from the sample.

## Sample Hold Times

**If PFAS are such persistent compounds, why is there a 14 day hold time for samples?**

While investigating the best way to preserve a PFAS sample, it was found that the antimicrobial agent was having an effect on the holding times of samples. The use of $CuSO_4$ led to a 20% decrease in mean recovery after 14 days. As no suitable antimicrobial was found that did not adversely affect the recovery of the analytes, it was decided that no antimicrobial would be used. Without the addition of an antimicrobial, 14 days is the maximum holding time that is preferred in current drinking water methods.

## Sample Handling

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

The procedure for handling turbid samples is to centrifuge the sample or alternatively, allow the sample to settle prior to sampling the supernatant. In situations where low levels are anticipated, the whole bottle is extracted. This makes the sediment a non-issue but will potentially introduce a high bias.

**Should a sample containing a lot of sediment be filtered?**

No. Best practices have indicated that the samples should not be filtered as it impacts the amount of PFAS detected when compared with centrifugation

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

## Quantifying PFAS

**What are the important considerations when calculating PFAAs?**

Isotope dilution Mass Spectrometry (IDMS)

IDMS provides a 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. This involves using the isotopically labelled analogue for each compound determined (isotopically labeled standard are commercially available for almost all PFAAs measured).

Which Multiple Reaction Monitoring (MRM) transitions are being used to quantify individual compounds?

It is known that some MRMs are more sensitive than others depending on the isomer being measured. This is an important consideration when it comes to linear vs. branched isomers.

MRM use offers the combination of selectivity and sensitivity.

Direct injection vs. Solid Phase Extraction (SPE)

Low-level water samples undergo 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 contaminant 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 then 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?**

It is important to distinguish between linear and branched as during the interpretation of data, it is important to understand if the sample is being quantified as the linear or branched chain isomers.

It is important to consider the impact of bias on quantitative results. The mix of branched PFOS isomers in the technical standard is important as biases may be seen in individual PFOS

## Quantifying PFAS (continued)

There are three scenarios for quantifying PFAAs:

1. All LC peaks are co-eluted and quantified against a linear PFOS standard; this has a potential bias of 40 – 80%.

2. All LC peaks are co-eluted and quantified against a mixed standard (known amounts of linear and branched. This method has improved comparability and substantially less bias.

3. All LC peaks are resolved (baseline) and quantified against a technical mixture that has been characterised (commercially available).

   Individual calibration curves are developed for the linear isomer and each branched isomer based on standard values. This method has the least bias of the three scenarios.

## 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.