



Best Practices for Optimizing **PFAS ANALYSIS**



Per- and polyfluoroalkyl substances (PFAS) are currently of great public health and environmental concern. Because ***PFAS are ubiquitous and commonly used in materials routinely employed for chemical analysis***, laboratories are in need of streamlined protocols to minimize background contamination from these chemicals and quickly generate accurate data. This ebook outlines best practices, from the field to the bench, for achieving those goals.

Collecting Samples

Personal Gear

Sampling for PFAS without contaminating the samples can be challenging due to the prevalence of these chemicals in many consumer products and standard sampling equipment. To avoid the possibility of cross-contamination, lab and field personnel should select field clothing and personal protective equipment (PPE) carefully when collecting or preparing samples for PFAS analysis.

Items to AVOID During Sampling	Items RECOMMENDED During Sampling
Water-resistant, waterproof or stain-treated clothing, boots and/or rain gear made from materials containing PFAS.	Rain gear made from polyurethane or wax-coated materials. Boots made with polyurethane and polyvinylchloride (PVC).
Clothing with fabric softener or suspected of containing PFAS. Some items labeled as “PFOA-free” contain replacement PFAS.	Cotton clothing is recommended and should be well washed before use due to possible contamination from PFAS-related treatments.
Sunscreens, moisturizers, hand cream or other related products.	Avoid using any personal care products.



During collection, well-washed cotton clothing and outer gear made from polyurethane or wax-coated materials is recommended.



Field Equipment and Sampling Bottles

Potential sources for PFAS cross-contamination include many items commonly found in the sampling equipment, such as items directly involved in the sample collection (e.g., automatic samplers, dippers and tubing) and other accessories. To ensure an accurate assessment of PFAS, sampling personnel should take precautions when collecting samples.

Due to potential adsorption of analytes onto glass, lab and field personnel should use polypropylene containers for all standard, sample and extraction preparations. Polypropylene bottles fitted with polypropylene screw caps allow for PFAS sampling without the risk of cross-contamination. Sample bottles must be discarded after use to prevent contamination from previous sampling procedures.

Items to AVOID During Sampling	Items RECOMMENDED During Sampling
Any items with a non-stick coating containing PFAS, including containers, tubing or any other waterproofed items (e.g., notebooks).	High-density polyethylene (HDPE) or polypropylene containers with HDPE or polypropylene caps.
Plastic materials potentially containing PFAS.	HDPE or silicone tubing materials.





Background Contamination

In order to check for residual PFAS on sampling equipment and overall contribution from different sources during the sampling event, equipment and field blanks should be collected prior to and during sampling. When collecting samples, personnel should use new nitrile gloves and replace them frequently to avoid cross-contamination.

Standard precautions for sample collection (e.g., bottle cap should not be placed on any other surface, avoid contact with inside of cap or bottle) should be strictly followed. After the sample is collected and capped, the sample bottle(s) should be placed in a resealable plastic bag separate from all other sample bottles.

Avoid reusing sampling equipment as previous uses may have involved PFAS-containing materials. Maintain separate supplies for PFAS sampling and for other contaminants. Before using new equipment, test for the presence of PFAS.

When reuse of materials and sampling equipment is necessary, lab and field personnel should follow standard decontamination procedures (as described later in this ebook) and confirm the absence of PFAS before reusing the equipment. It is also recommended to avoid the use of any materials listed on pages 3 and 4.

Lab Equipment Cleaning & Decontamination

PFAS can be present in the water and/or cleaning agents used in decontamination processes. When cleaning sampling equipment, lab personnel should avoid using decontamination soaps containing fluorosurfactants such as Decon 90. Water from an on-site well is also a potential source of contamination.

Alconox® and/or Liquinox® are recommended for decontamination processes as well as potable water from a municipal drinking water supply. Sampling equipment should be scrubbed using a polyethylene or PVC brush and flushed with water before the next use. Water should be always verified as “PFAS-free” before it is used for field and decontamination blanks and decontamination processes.

Food & Beverages

Standard safety protocols do not allow the presence of food and drinks in laboratories and areas where sampling is occurring. During the PFAS analysis, this safety protocol is even more relevant as food packaging, wrappers and containers may contain PFAS and can cause cross-contamination. Drinks and food should be kept nearby (e.g., staging area for sampling) to ensure personnel's safety.



LABORATORY MATERIALS

Stock solutions should be prepared and stored in PFAS-free high-density polyethylene (HDPE) or polypropylene (PP) containers with lined or unlined HDPE or polypropylene caps. Do not store samples in containers made of glass or low-density polyethylene (LDPE) materials. PFAS can adsorb to glass, especially when the chemicals are stored in a glass container for long periods of time.

Stability of the standards solutions for a predetermined interval of time when stored under recommended conditions is a relevant parameter for ensuring the quality of the analysis. As shown in Figure 1 (see next page), 50% methanol in water (same mixture as that used in ASTM D7979) is the optimal solution for dissolving PFAS and maintaining them in solution.



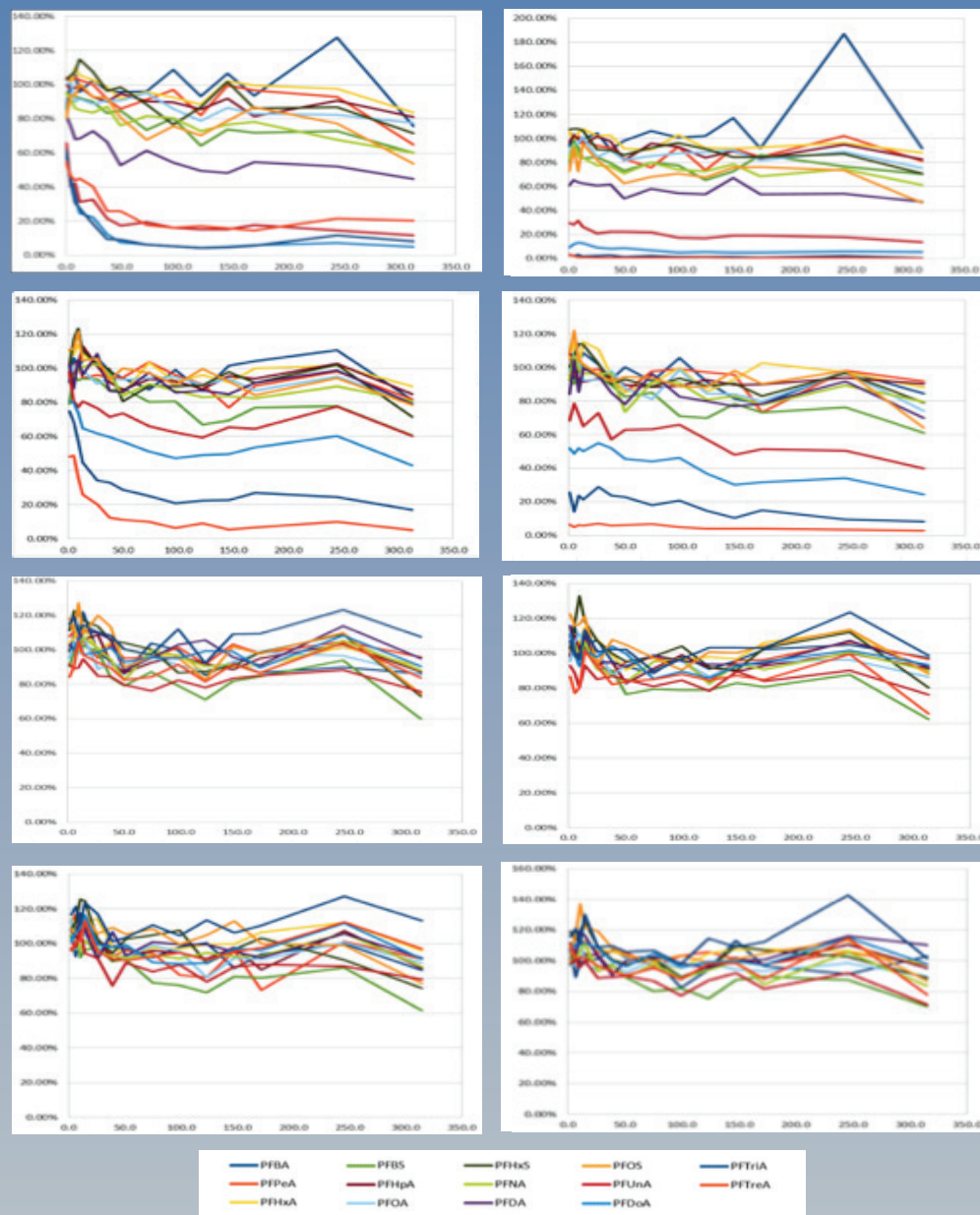
Figure 1: Potential adsorption of PFAS on the vial surface

Plots of PFAS recovery against shelf life (time/hour) for the various solvents in glass and polypropylene LC vials.

10% METHANOL
30% METHANOL
50% METHANOL
70% METHANOL

GLASS VIALS

POLYPROPYLENE VIALS



Mixtures with lower concentrations of methanol (10% and 30%) show larger losses of PFAS due to the insolubility of PFAS in the solvent used.

The recovery results for 90% methanol are similar to that of 70% methanol. However, the **higher methanol content evaporates faster and causes changes in the sample volume.**

The PFAS concentration in the vial may change after the vial cap is pierced as the organic solvent (e.g., methanol:water solution) and/or PFAS compound can be lost through the puncture. If calibration standards are to be used multiple times, it is recommended to use an amber glass vial with sealed replaceable caps. Sealing the vials immediately after injection may reduce the loss of PFAS.

The use of LC propylene vials is commonly recommended for the analysis of PFAS. Shimadzu scientists compared LC propylene vials to amber glass vials (used in the majority of general applications and more easily resealed) to determine the potential adsorption of PFAS on the vial surface. Similar recovery and quantitation were observed for both types of materials, as shown in Figure 1.

Sample Preparation and Injection

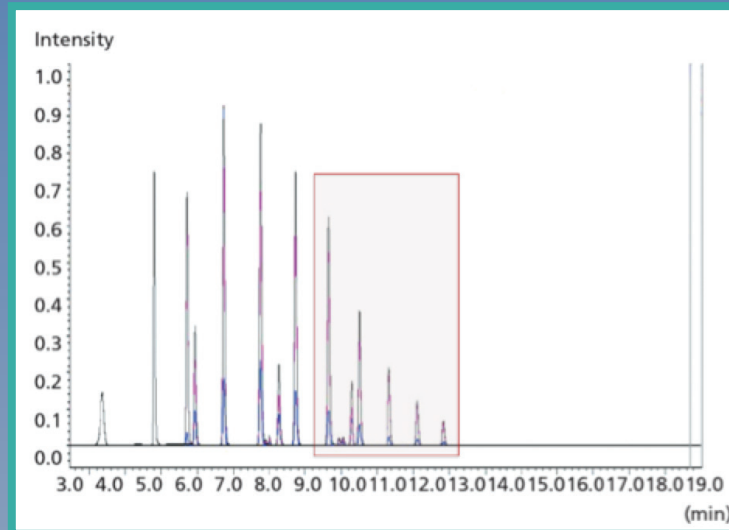
Some currently published methods (EPA 537, EPA 537.1) require a step of sample pre-concentration by solid phase extraction (SPE). Materials used in the manufacturing of supplies for preparing the samples by SPE may also contain PFAS. To avoid pre-concentrating the background PFAS during this step of the analysis, all new SPE cartridges, solvents and vials for collecting samples must be tested for PFAS prior to the first use.

PFAS-free tubing should be used for loading samples into the cartridges. If automatic sample extractors are employed for this step of the analysis, checking with the manufacturer is strongly recommended to identify all components made of PFTE and replace them when feasible.

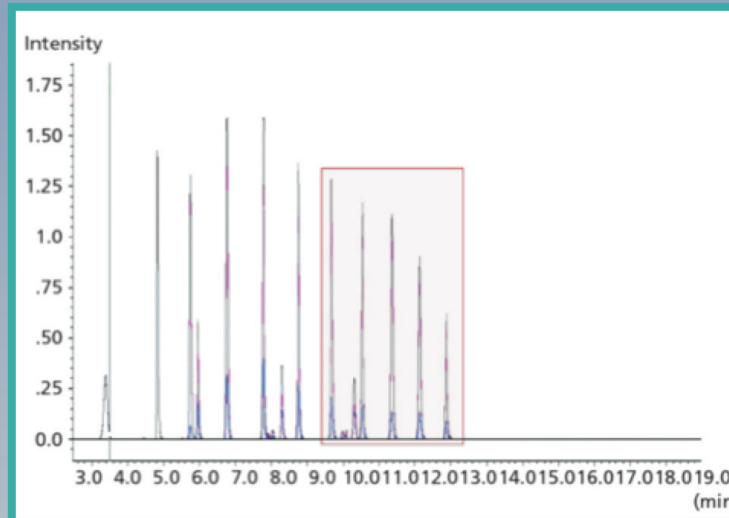
Once samples are pre-concentrated and ready for injection in the LC-MS/MS or samples are prepared accordingly to methods that allow for large volume injection (ASTM D7979), they may sit in the autosampler tray for extended periods of time. In these situations, some PFAS compounds may settle, precipitate or adsorb on the surface. It is important to remember to mix the extract/sample before (re)injection. Vortexing the solution before injection ensures a homogenous solution and optimum results. Figure 2 shows the chromatogram of the PFAS compounds before and after vortexing a 50 ng/L standard allowed to sit for 24 hours. The recovery of the long-chain PFAS is considerably lower before vortex.

Figure 2: PFAS compounds before and after vortexing a 50 ng/L standard allowed to sit for 24 hours

BEFORE VORTEX



AFTER VORTEX



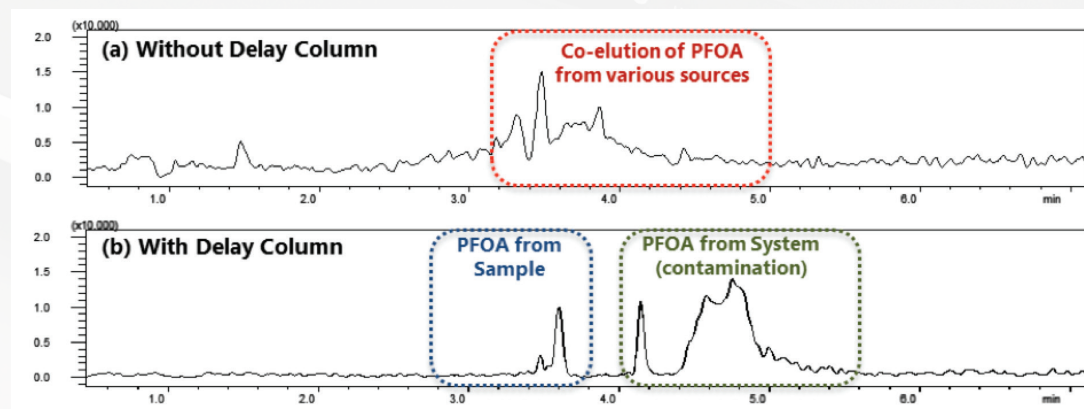


Instrumentation

It is recommended to use a solvent delay column (installed after the mixer and before the autosampler) to delay the elution of PFAS originating from solvent bottles and other parts of the liquid chromatography system (e.g., pumps and tubing). As shown in Figure 3 below, using the delay column enables the detection of PFOA originating solely from the sample.

Additionally, bypassing the degasser when possible is recommended as well as replacing any PTFE-containing tubing and parts in the LC.

Figure 3: Chromatogram of PFOA:
(a) without delay column and (b) with delay column



Shimadzu's team of service engineers can help you set up the exact LC configuration (including solvent lines, tubing, bypassing of solvent lines and more) that is proven to deliver contamination-free results. For more information, please contact a Shimadzu expert at **800-477-1227** or visit **www.OneLabOneEarth.com**.

In collaboration with EPA and ASTM International, Shimadzu is working to advance research and technical knowledge related to PFAS exposure and contamination. Using Shimadzu LC-MS/MS instruments, they have vetted standardized methods for analyzing PFAS compounds in a diverse type of samples. Designed with proprietary ultrafast technologies and patented ion focusing technology, Shimadzu's LC-MS/MS systems deliver fast, high-quality results for PFAS analysis.



To learn more about Shimadzu's solutions for PFAS analysis, visit **www.OneLabOneEarth.com**



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