

# Expanding PFAS Analysis Beyond Environmental Matrices

## Webinar Q&A

### Q&A from Separation Science Webinar – November 14<sup>th</sup>, 2023

This document details questions submitted before and during the November 14th webinar, “Expanding PFAS Analysis Beyond Environmental Matrices.”

During the webinar, a panel of experts covered various topics extensively. However, due to limited time, we could not address all the questions we received.

To provide structure, we have organized the submitted questions into five sections below:

- PFAS Regulatory & Regional Questions
- General PFAS Chemistry Questions
- PFAS Analytical Questions
- PFAS Application Questions
- PFAS Treatment

The remainder of the document will present the questions submitted, along with responses from the panelists and our team of analytical testing experts. Some questions may have multiple responses, and some may only have one.

The experts answering your questions:



**Ruth Marfil-Vega,**  
Senior Market Manager,  
Environmental at Shimadzu  
Scientific Instruments



**Rock J Vitale,**  
Technical Director of Chemistry,  
Senior Principal at Environmental  
Standards, Inc.



**Olga Shimelis,**  
Analytical Chemistry R&D Manager  
Life Science business of Merck



## PFAS Regulatory & Regional Questions:

### 1. Technically speaking, what is the screening limit vs the regulatory limit?

Speaker	Response
Olga Shimelis	This will depend greatly on the regulatory bodies in question and the specific methods being used. Some methods are more sensitive than others.
Ruth Marfil-Vega	<p>According to the EPA's (Regional) Screening Limits are used to identify contaminated media (i.e., air, tap water, and soil) at a site that may need further investigation. Together with Regional Removal Management Levels, they are used to support the EPA's decision to undertake a removal action under CERCLA (<a href="https://www.epa.gov/newsreleases/epa-adds-five-pfas-chemicals-list-regional-screening-and-removal-management-levels">https://www.epa.gov/newsreleases/epa-adds-five-pfas-chemicals-list-regional-screening-and-removal-management-levels</a>). Currently, there are six PFAS with regional screening limits: PFBS, PFHxS, PFOS, PFOA, PFNA, HFPO-DA (GenX).</p> <p>Under the Safe Drinking Water Act (SDWA), the EPA is still working on establishing "regulatory limits" for six PFAS (the same as those mentioned above) in drinking water. These are legally enforceable levels known as Maximum Contaminant Levels (MCLs). The maximum contaminant level (MCL) that is permitted in the water that is supplied to all users of public water systems is known. The EPA is also working on establishing regulatory limits in other environmental matrices.</p> <p>Reference to levels:  <a href="https://www.epa.gov/newsreleases/epa-adds-five-pfas-chemicals-list-regional-screening-and-removal-management-levels">https://www.epa.gov/newsreleases/epa-adds-five-pfas-chemicals-list-regional-screening-and-removal-management-levels</a>  <a href="https://www.trccompanies.com/insights/five-new-pfas-added-to-epa-regional-screening-levels-rsls/">https://www.trccompanies.com/insights/five-new-pfas-added-to-epa-regional-screening-levels-rsls/</a>  <a href="https://www.epa.gov/sdwa/and-polyfluoroalkyl-substances-pfas">https://www.epa.gov/sdwa/and-polyfluoroalkyl-substances-pfas</a>.</p>

### 2. Are there any guidelines or regulations on PFAS exposure in food?

Speaker	Response
Olga Shimelis	There are several regions around the globe that are considering regulations regarding PFAS in food or food packaging. Some states within the United States have already passed laws that will go into effect in the next few years. To learn about state-level regulations in the U.S., you can visit this link: <a href="https://www.ncsl.org/environment-and-natural-resources/per-and-polyfluoroalkyl-substances#packaging">https://www.ncsl.org/environment-and-natural-resources/per-and-polyfluoroalkyl-substances#packaging</a> .
Ruth Marfil-Vega	<p>References:</p> <ul style="list-style-type: none"> <li>• <a href="#">USDA Farm Production and Conservation</a> <ul style="list-style-type: none"> <li>○ DoD is required to notify <b>agricultural</b> operations located within one mile downgradient of a military installation where PFAS has been detected in groundwater (PFOS and PFOA at 70 ppt and PFBS at 40 ppb).</li> </ul> </li> <li>• <a href="#">USDA PFAS in Blood and Tissue in Dairy Animals</a></li> <li>• <a href="#">USDA and DoD PFAS Memorandum – using EPA 1633 for fish under the tissue matrix: Establishing a Consistent Methodology for the Analysis of Per- and Polyfluoroalkyl Substances in Matrices Other than Drinking Water</a> <ul style="list-style-type: none"> <li>○ Methods other than Draft Method 1633 shall not be used to analyze samples for regulatory compliance, risk assessment, or comparison to a project screening.</li> <li>○ Other methods for analysis may be considered for screening samples to determine the presence or magnitude of PFAS concentration, but not to confirm its absence.</li> </ul> </li> <li>• From the FDA: <ul style="list-style-type: none"> <li>○ There are currently seven PFAS (PFOA, PFOS, PFNA, PFHxS, HFPO-DA [GenX], PFBS, and PFBA) from environmental contamination for which the FDA can assess the potential human health concern for levels found in food. <a href="#">FDA Shares Results on PFAS Testing in Seafood   FDA</a></li> </ul> </li> </ul>

**3. What health effects does PFAS have on humans? Have there been laboratory trials or human studies?**

Speaker	Response
Olga Shimelis	PFAS refers to a very large group of substances. There are many studies investigating the toxicology of individual perfluorinated substances and groups of PFAS. Their chemistries and toxicological profiles vary greatly.
Ruth Marfil-Vega	Several studies on animals have examined the effects of contact, ingestion, and inhalation. The CDC report that compiled the information from different research sources is available here: <a href="#">PFBS, HFPO-DA (GenX) and PFBA reference doses</a> from the EPA and the <a href="#">ATSDR Perfluoroalkyls (PFAS) Tox Profile (cdc.gov)</a> . Further information is here: <a href="https://www.atsdr.cdc.gov/pfas/">https://www.atsdr.cdc.gov/pfas/</a> .

**4. How do you evaluate the possibility that PFAS reference materials—which might be banned in the EU or possibly globally—may no longer be available because the production and distribution of the substance are no longer possible or economical?**

Speaker	Response
Olga Shimelis	Reference materials are a critical aspect of understanding and tracking the development of global PFAS treatment efforts. There is historical precedent from other banned substances and abused drugs to permit certain institutions to keep producing the materials in small amounts for use as laboratory reference materials.
Rock J Vitale	I am confident that for the purposes of analytical testing, manufacturing reference materials will have special dispensation.
Ruth Marfil-Vega	It is a very unfortunate possibility, as it will limit the ability to properly identify and quantify specific PFAS and the validation of analytical methods to support monitoring of PFAS.

**5. My impression is that PFAS analysis is not yet common in Japan, and only a limited number of samples are analyzed by some analytical institutions using environmental water and soil as test samples. Recently, I heard that ballast water should also be subject to PFAS testing. How much is actually tested in other countries?**

Speaker	Response
Olga Shimelis	We don't have data on testing volumes of ballast water.
Ruth Marfil-Vega	Various countries are in the process of formulating regulations for PFAS across different sample types, including environmental, food, and materials. Signatories to the Stockholm Convention - the European Union, the United States, Australia, and Japan, among others - are at varying stages of this regulatory development. Hence, it is difficult to determine how much is tested in each country for compliance purposes. However, in the past 5-7 years, there has been an exponential increase in testing for R&D and "knowledge gathering" about PFAS.

## General PFAS Chemistry Questions

### 1. Approximately how many PFAS have been identified in the environment, and if there is an identified subset, which might be the most toxic or dangerous to wildlife and humans?

Speaker	Response
Olga Shimelis	The number of compounds that have been identified as PFAS varies depending on who you ask, and many of them lack reference materials that allow for precise quantification, making it impossible to say which have been found in the environment. Internally, we have chosen to focus on the compounds that are being regulated under the assumption that they have been identified as the most relevant for testing. The National Institute of Environmental Health Sciences ( <a href="https://www.niehs.nih.gov/">https://www.niehs.nih.gov/</a> ) is actively engaged in toxicology studies. We recommend reviewing their publications for more information.
Rock J Vitale	Within the US, up to 85 PFAS compounds are offered as reference materials. Many others are identified using non-target analysis, but these are not accurately quantitated with reference materials.
Ruth Marfil-Vega	According to the EPA's ComTox Database, PFAS are a group of nearly 15,000 synthetic chemicals ( <a href="https://www.niehs.nih.gov/health/topics/agents/pfc/index.cfm#:~:text=PFAS%20are%20a%20group%20of,Bioaccumulation">https://www.niehs.nih.gov/health/topics/agents/pfc/index.cfm#:~:text=PFAS%20are%20a%20group%20of,Bioaccumulation</a> ). From those, only a small fraction have been unequivocally identified in the environment (identification confirmed with an authentic standard). Knowledge about the toxicity of individual PFAS is still evolving. Currently, different agencies are focusing their efforts on mitigating the risk from PFAS exposure ( <a href="https://www.epa.gov/pfas/our-current-understanding-human-health-and-environmental-risks-pfas">https://www.epa.gov/pfas/our-current-understanding-human-health-and-environmental-risks-pfas</a> ).

### 2. If an organic molecule only contains one trifluoromethyl or difluoro methylene group, is that considered PFAS? Or does the molecule have to contain many repeating units to be PFAS?

Speaker	Response
Olga Shimelis	This depends very much on the definitions being used under different regulations being developed in the US, EU, and elsewhere.
Rock J Vitale	It is my understanding - not that I agree - that any compound with at least one CF <sub>2</sub> or CF <sub>3</sub> is considered a PFAS.
Ruth Marfil-Vega	According to the OECD, "PFAS are defined as fluorinated substances that contain at least one fully fluorinated methyl or methylene carbon atom (without any H/Cl/Br/I atom attached to it), i.e., with a few noted exceptions, any chemical with at least a perfluorinated methyl group (-CF <sub>3</sub> ) or a perfluorinated methylene group (-CF <sub>2</sub> -) is a PFAS. <a href="https://www.oecd.org/chemicalsafety/portal-perfluorinated-chemicals/terminology-per-and-polyfluoroalkyl-substances.pdf">https://www.oecd.org/chemicalsafety/portal-perfluorinated-chemicals/terminology-per-and-polyfluoroalkyl-substances.pdf</a>

### 3. Have you done any testing of fluorotelomers in conjunction with PFAS testing to determine any strong correlations? I am wondering what precursors lead to PFAS and how you may have determined that.

Speaker	Response
Olga Shimelis	We have not been involved in any precursor research to date.

### 4. What do you perceive as the challenges associated with analyzing PFAS/microplastic systems?

Speaker	Response
Olga Shimelis	The two primary challenges for both applications are the accessibility / affordability of reference standards, as well as appropriately accounting for matrix interferences.
Rock J Vitale	Yes. Solvent extracting microplastic fluoropolymers will require research. I recently read that extracting with THF holds promise. Short of that, doing TF or TOF by CIC is becoming more commercially available.

## 5. How would you rate the ease of method development?

Speaker	Response
Olga Shimelis	Very challenging. There are many documented variables that can affect a lab's ability to get consistent and accurate data, even when using existing methods. Developing new methods compounds this problem due to the unknown issues that may arise.
Rock J Vitale	With good instruments and talented scientists, it typically takes 2-3 months to get methods up and running.
Ruth Marfil-Vega	Scientists have been working on the development and standardization of analytical methods for PFAS for several decades; hence, there is a wealth of knowledge available to ease the implementation of methods in different labs. Shimadzu developed method packages for EPA 537.1 and 533 ( <a href="https://www.ssi.shimadzu.com/products/liquid-chromatograph-mass-spectrometry/lc-ms-system/lcms-ms-ms-method-package-pfas/index.html">https://www.ssi.shimadzu.com/products/liquid-chromatograph-mass-spectrometry/lc-ms-system/lcms-ms-ms-method-package-pfas/index.html</a> ) to minimize the effort for method development. Information about other developed methods can be obtained from our <a href="#">team</a> .

## 6. How should dark matter or unknown unknowns be managed?

Speaker	Response
Olga Shimelis	High-res mass spectrometry is a powerful tool for analyzing unknowns.
Ruth Marfil-Vega	By using high-resolution QTOF instruments, like Shimadzu's QTOF LCMS-9050 (as shown in this <a href="https://www.ssi.shimadzu.com/products/liquid-chromatograph-mass-spectrometry/quadrupole-time-of-flight-lc-msms/index.html?utm_source=ssi-website&amp;utm_medium=lcms-index&amp;utm_campaign=prod-highlight">app note: https://www.ssi.shimadzu.com/products/liquid-chromatograph-mass-spectrometry/quadrupole-time-of-flight-lc-msms/index.html?utm_source=ssi-website&amp;utm_medium=lcms-index&amp;utm_campaign=prod-highlight</a> ).

## 7. Can firefighting foam be considered a full-part matrix, or should it be diluted before analysis?

Speaker	Response
Olga Shimelis	As with any sample, we recommend dilution / clean-up prior to injection into your instrument to prolong the life of your analytical columns.
Rock J Vitale	Dilution - perhaps substantial dilution - should be performed for speciation or TF/TOF.

## 8. Any advice on improving asymmetry?

Speaker	Response
Olga Shimelis	In general, it is our experience that iterating the chromatographic conditions can be used to improve chromatographic performance. Variables to consider would be pH, ionic strength of the mobile phase, and column chemistry.
Ruth Marfil-Vega	Using a column with a longer length, a wider inner diameter, and better axial diffusion improves symmetry at the column exit and improves peak shape. Also, co-injection, a feature of modern autosamplers that ensures the similarity between sample and mobile phase, helps in improving peak shape. This <a href="#">presentation</a> illustrates how to improve peak shape when analyzing PFAS using liquid injection after sample co-solvation (e.g., ASTM D8421).

## 9. Are there any specific models capable of simulating the migration of PFAC contaminants in groundwater?

Speaker	Response
Olga Shimelis	This is outside of our expertise. Our recommendation would be to check with the US EPA or USGS.

## PFAS Analytical Questions

### 1. High-level Analytical Questions

- a. **As PFAS analyses become more standardized, labs may choose to outsource analysis. What other analytical applications could utilize LC-MS/MS equipment previously used for PFAS?**

Speaker	Response
Olga Shimelis	The systems could be used for the analysis of pesticides not covered by GC methods, and for the analysis of pharma and personal care products (Method 1694). Also, any future contaminants (such as the next UCMR list) are not amenable to GC.
Rock J Vitale	Halogenated pesticides and PAHs.
Ruth Marfil-Vega	LC-MS/MS can be used for the analysis of a large list of organic chemicals. Some examples include <a href="#">pesticides</a> , <a href="#">mycotoxins</a> , <a href="#">veterinary drugs</a> , <a href="#">pharmaceuticals</a> , and <a href="#">other chemicals of toxicological relevance</a> ; <a href="#">restricted chemicals</a> ; <a href="#">6PPD and 6PPDQ</a> ; <a href="#">disinfection byproducts</a> ; and <a href="#">more</a> .

- b. **Is there a better technology than mass spectrometry for positive PFAS identification and quantitation at minute concentrations?**

Speaker	Response
Olga Shimelis	Rather than "better," one could refer to complementary technologies that would provide structural information not possible with quadrupole mass spectrometry. Examples could be NMR, HR-MS, and MS <sup>n</sup> .
Rock J Vitale	I don't believe at ppt or ppq levels.
Ruth Marfil-Vega	Currently, mass spectrometry is the best technology for quantifying PFAS at trace concentrations.

- c. **From my own experience, I would say that analyzing PFAS is very expensive. As such, is there anything that is being done to reduce the costs involved in this analysis without compromising the end results?**

Speaker	Response
Olga Shimelis	Our scientists are actively pursuing ways to lower the cost of analysis by streamlining sample preparation workflows, reducing mobile phase consumption during analysis, and providing more options for reference materials. We recommend working with your account managers to identify areas where savings might be found.
Rock J Vitale	Interesting question, but basic economic theory applies here, such as supply vs. demand. Pricing will continue to increase for a while.
Ruth Marfil-Vega	As PFAS analysis becomes "mainstream," the cost per sample charged by commercial labs is decreasing. The cost of instrumentation has also decreased since commercial LC-MS/MS has become available. Unfortunately, the cost of labeled analytical standards is still expensive. For the most up-to-date information about return on investment for PFAS analysis by LC-MS/MS, I encourage you to reach out to Shimadzu's <a href="#">team</a> .

- d. **Do the current testing methods have sufficient sensitivity for short-chain PFAS? As far as I understand, long-chain PFAS are the ones of concern and therefore most targeted by monitoring, while short-chain PFAS are mostly undetected by these methods.**

Speaker	Response
Olga Shimelis	Not all methods are suitable for short-chain PFAS, but there are methods being developed specifically for them.
Rock J Vitale	The short-chain PFAS need alternate sampling and analytical/prep methodologies.

- e. **Water is the most popular matrix for testing, and many of our methods are for liquids. As PFAS are found in more consumer products, such as cosmetics, is there a focus on the analysis of PFAS in solids (other than CIC)?**

Speaker	Response
Olga Shimelis	Methods for PFAS in some solids (soil, food, and food packaging) have been developed already. If analysis is needed for other matrices, the methods can be adapted for the extraction of PFAS into the liquid phase and analysis.
Rock J Vitale	Yes, indeed. I co-chair the ASTM Subcommittee F15.81, and speciating PFAS in consumer product solids is in preparation. The first standard was recently balloted.
Ruth Marfil-Vega	Yes, PFAS are commonly analyzed in biosolids, textiles, foods, food packaging materials, and animal tissue by LC-MS/MS and GCMS.

- f. **We observed TOF analysis results that were lower than the fluoride ion concentration bound to PFAS compounds (fluorine ion bound to total detected PFAS compounds). How should we interpret these results? Are they due to the low sensitivity of TOF?**

Speaker	Response
Olga Shimelis	This question is outside our expertise.
Rock J Vitale	Without knowing how you did the TOF, that cannot be answered accurately.
Ruth Marfil-Vega	I am assuming that you calculated the concentration of fluorine bound to the total detected PFAS theoretically. If that is the case, your measurements of the total detected PFAS and TOF have an inherent analytical error associated with them. That could explain why the TOF is lower. Low sensitivity affects only the minimum concentration you can accurately quantify.

- g. **In 537.1, branched isomers have an observed lower response ratio than linear (partly because the MRM is optimized for linear). How worried are we about the low bias for branched isomers built into the method and ignoring branched isomers for most of the analytes?**

Speaker	Response
Olga Shimelis	This is a very interesting question. We recommend reaching out to method authors directly; they may have more insight to share on this.
Rock J Vitale	VERY worried. Each of those mono- and dimethyl-branched isomers has its own unique responses, and to assume they are all similar to the linear responses yields a gross estimation at best. We have an elegant method, but we lack quantitative branched isomer reference materials.

- h. **Have there been any new advancements observed in the analysis of PFAS for wastewater treatment plants?**

Speaker	Response
Olga Shimelis	Generally speaking, there is a lot of funding and investment in improving/simplifying the sample preparation steps around PFAS analysis.

- i. **Can we measure PFOA/PFOS in real-time? Are there instruments for this?**

Speaker	Response
Olga Shimelis	We have seen prototypes presented, but nothing is commercially available yet.
Rock J Vitale	Using Direct Aqueous Injection (DAI) may have application in semicontinuous monitoring.
Ruth Marfil-Vega	As published by C&EN in early 2023, there is <a href="#">a race on for low-cost PFAS sensors</a> .

## 2. Specific Analytical Questions

- a. **How much of the total analyzed fluorine do you assume to come from synthetic routes, e.g., PFAS? How much do you assume comes from a natural (mineral) source?**

Speaker	Response
Olga Shimelis	We will defer to our colleagues on this issue
Ruth Marfil-Vega	Most organic fluoride in the environment comes from synthetic sources.

- b. **How do you manage fully organic injection when analyzing food matrix coming from acetonitrile extraction? Are you able to run C4C18 in the same run?**

Speaker	Response
Olga Shimelis	Acetonitrile extract can be injected directly, and C4-C18 can be analyzed in the same run per FDA method C-010. The LC gradient can start at 10% methanol with 3 uL injections.
Ruth Marfil-Vega	I would suggest the use of co-injection (the same advice as for improving symmetry).

- c. **What is the main challenge that we face in PFAS analysis and the risk of analyzing PFAS?**

Speaker	Response
Olga Shimelis	From an analytical standpoint, the main challenges are background contamination and access to a comprehensive CRM library.
Ruth Marfil-Vega	Because PFAS is present in many analysis products, routine checks of background concentrations are required to ensure low reporting limits can be achieved.

- d. **What are the interferences when analyzing PFAS, and how do you resolve these interferences?**

Speaker	Response
Olga Shimelis	In our experience, background contamination and matrix interference pose the largest issues. Careful selection of consumables, the use of a delay column, and appropriate sample preparation are critical to resolving interferences.
Rock J Vitale	Considerations include handling analytes without a secondary transition, managing branched isomers, and, moreover, mitigating the impact of thousands of non-target PFAS compounds to prevent false positives and negative biases.

- e. **What is the biggest matrix issue facing PFAS analysis?**

Speaker	Response
Olga Shimelis	Any matrix that is not drinking water will generally require extensive sample preparation and clean-up. This issue is addressed in EPA Draft Method 1633 (4th draft), Section 4.4.

- f. **What is the best way to find the total PFAS contribution of a sample, and how do you account for inorganic fluorine in solids?**

Speaker	Response
Rock J Vitale	As a first screening, perform a total fluorine analysis. If the outcomes are within a reasonable range, no additional steps need to be taken. More investigation of solvent extraction techniques is needed to understand which techniques will dissolve all PFAS compounds, including fluoropolymers, while keeping inorganic fluoride out of the equation.



**g. What are the risks of false positives?**

Speaker	Response
Olga Shimelis	Every method has differing potential risks of false positives. This is why adequate QC checks are a crucial part of best practices.
Rock J Vitale	The more complex the sample, the greater the risk of false positives from non-target PFAS.

**h. How is ion mobility being used in the analysis?**

Speaker	Response
Olga Shimelis	We are not aware of any promulgated methods that utilize ion mobility as part of the analysis.

**i. How can PFAS contamination be eliminated from lab environments?**

Speaker	Response
Olga Shimelis	This is VERY challenging. Using consumables that have been validated to have low background contamination is a large step towards this, but there will always be some small sources of background contamination.

**j. Regarding PFAS, which are everywhere and in everything, it must be difficult to find a clean matrix to do blank analysis. How do we manage this aspect?**

Speaker	Response
Olga Shimelis	We have seen multiple PFAS compounds come from lab backgrounds. Of course, all chemicals need to be pre-tested before being used in analytical workflows. Clean water can be obtained from high-level water purification systems. Work areas need to be cleaned, and all glassware needs to be decontaminated on a regular basis.

**k. In contrast to US EPA 1633, where offline SPE is recommended, how do you see the potential to use online SPE on LC/MS/MS approaches instead?**

Speaker	Response
Olga Shimelis	There is definitely potential for online SPE.
Ruth Marfil-Vega	Online SPE is increasing in popularity but hasn't yet been implemented broadly. It automates the workflow, with all the associated advantages associated with it (time savings, error minimization). At the same time, it increases the complexity of hardware, method optimization, and troubleshooting. When it becomes accepted into standardized methods, labs will have to evaluate the pros and cons for their specific requirements.

**l. What is the method that you recommend for drinking water analysis evaluated with a detection limit of 1 ng/L?**

Speaker	Response
Olga Shimelis	Current methods, such as US EPA Method 533, have detection limits that are determined by the instrumentation's design and capability as well as other aspects of the method's execution, such as adequate control of the PFAS background and chromatographic conditions optimized to yield better S/N ratios.
Rock J Vitale	I would not recommend attempting this low of a limit. You cannot confidently assess this in anything short of an ultra-clean room research facility.

### 3. PFAS Consumables question

- a. **What are some hardware changes you have made to your instruments to reduce or remove PFAS contamination?**

Speaker	Response
Olga Shimelis	The use of delay columns is a must! Some instruments may benefit from replacing PTFE tubing if it is used.
Ruth Marfil-Vega	In this <a href="#">document</a> , we summarize the essential modifications of the instrument for minimizing the presence of PFAS in the background: a delay column.

- b. **What are the best mobile phase modifiers to use for environmental samples as well as food samples of varying matrices?**

Speaker	Response
Olga Shimelis	Not necessarily a modifier, but we recommend using the highest-quality grade solvents and reagents possible.

- c. **I am analyzing PFAS in fish meal. My sample prep involves QuEChERS, followed by dSPE. My blanks have revealed that the dSPE kit contains a small quantity of PFOA. Is this a known and common issue?**

Speaker	Response
Olga Shimelis	We can only speak to our own dSPE products, which have not shown this issue. This is a great example of why all consumables should be tested prior to use.

- d. **Which column types of phases) should be used for PFAS analysis?**

Speaker	Response
Olga Shimelis	C18 and columns validated by the manufacturer for suitability in PFAS analysis. For example, we offer a column labeled specifically for PFAS analysis.

- e. **What are the column chemistries used for the delay columns?**

Speaker	Response
Ruth Marfil-Vega	Same as for the analytical column.

- f. **How long can a delay column be used?**

Speaker	Response
Olga Shimelis	Delay columns can be used until the background peak gets too close to the analytical peaks in your chromatogram.

- g. **Accuracy check: Are there suitable CRMs or standards in some relevant environmental matrices at suitable levels, e.g., at LOD or LOQ methods, over the analytical range, and/or at the levels typically present?**

Speaker	Response
Olga Shimelis	Not yet that we are aware of.
Rock J Vitale	CRMs and reference materials are severely lacking, and this needs attention from the manufacturers.
Ruth Marfil-Vega	NIST is a good source of CRM materials for PFAS testing.

- h. Is anyone aware of efforts to add more commercially available stable isotopes for some of the newer PFAS targets? If not, any ideas on what we can do to encourage this?**

Speaker	Response
Olga Shimelis	As a reference material provider, we are definitely aware of this situation and are working to address it.
Rock J Vitale	Isotope reference materials are severely lacking, and this needs attention from the manufacturers.

- i. What advancements are being looked at for analytical testing? Do you think that testing will eventually be a requirement for all consumables?**

Speaker	Response
Olga Shimelis	We see the most activity around sample prep and developing total fluorine methods.

#### **4. PFAS Waste**

- a. What is a good reference for best practices for disposing of PFAS waste?**

Speaker	Response
Olga Shimelis	We recommend working closely with your local regulators.
Ruth Marfil-Vega	Dispose of the waste according to the requirements for hazardous waste disposal in the lab.

- b. Is there any recycling program that can accept the vast amount of one-use plastic items used in PFAS analysis (e.g., sample bottles, tubes, vials, pipet tips, etc.)? It pains me that in attempting to address one environmental problem, we may be adversely impacting another one.**

Speaker	Response
Olga Shimelis	Not that we're aware of, but again, your local regulators may have more regional insight into this.
Ruth Marfil-Vega	I am not aware of any specific program for recycling consumables used for PFAS analysis. However, there are labs that dispose of and recycle them in accordance with their local regulations for lab materials.

## PFAS Application Questions

### 1. PFAS in Batteries and Electronics

#### a. Do you have any advice for the analysis of PFAS in Li-ion battery production and recycling?

Speaker	Response
Olga Shimelis	Methods such as ASTM 8535 or ISO 23702 that utilize solvent extraction from solids would be a good place to start in developing a method to test battery materials.

#### b. What types of PFAS are most likely to be present in lithium-ion batteries or formed when they are recycled?

Speaker	Response
Olga Shimelis	PFAS-containing materials/coatings can potentially be used as separators. Solvents and electrolyte solutions are the next most likely source of PFAS contamination, followed by contamination from packaging. We don't have any information on specific analytes, however.

### 2. PFAS in Human Matrices

#### a. Are there benefits to measuring linear and branched PFOS/PFOA in human matrices, or is a total level sufficient?

Speaker	Response
Olga Shimelis	This is outside our expertise.
Rock J Vitale	If you're not measuring branched compounds with quantitative standards, your total is an estimate at best.

#### b. Has there been any significant use of PFAS in plastic bags or plastic bottles containing blood, nutrient solutions, eye rinsing solutions, or other vital solutions used in medical treatment or in eye lenses?

Speaker	Response
Olga Shimelis	We do not have any specific data, but there is a lot of effort being applied in this industry to evaluate the components and materials used to replace any potential PFAS-containing materials.

### 3. PFAS in Food

#### a. Where can we analyze PFAS in food matrices? What are the food safety and health impacts of PFAS on humans?

Speaker	Response
Olga Shimelis	Local government labs or independent testing labs should be able to help provide testing services. The US FDA has published a website addressing PFAS in food concerns here: <a href="https://www.fda.gov/food/process-contaminants-food/questions-and-answers-pfas-food">https://www.fda.gov/food/process-contaminants-food/questions-and-answers-pfas-food</a>
Ruth Marfil-Vega	PFAS bioaccumulates in the environment, so the most contaminated food sources will come from contaminated environments. This makes seafood particularly susceptible.  <i>Note from the FDA:</i> No PFAS have been detected in over 97% (701 out of 718) of the fresh and processed foods tested in the TDS ( <a href="#">Total Diet Study</a> ). At least one type of PFAS was detected in 44% (14 out of 32) of the TDS seafood samples and in 74% (60 out of 81) of the samples from our 2022 targeted seafood survey.  There are currently seven PFAS (PFOA, PFOS, PFNA, PFHxS, HFPO-DA [GenX], PFBS, and PFBA) that have sources for toxicological reference values. These are being studied and monitored with first priority as the list is expanded.

**b. How do high PFAS levels in water sources impact the manufacturing of food, and are they monitored today?**

Speaker	Response
Olga Shimelis	PFAS in feed water sources can impact manufacturing facilities if there are no mitigation efforts in place. The extent of monitoring and treatment is unknown to us.
Ruth Marfil-Vega	Water is needed throughout the food chain; contaminated water will impact crops and animals to which it is exposed. The <a href="#">USDA Farm Production and Conservation Act</a> requires <b>agricultural</b> operations located within one mile downgradient of a military installation where PFAS has been detected in groundwater (PFOS and PFOA at 70 ppt, PFBS at 40 ppb) to be notified of possible contamination.

**c. What is the best method to determine total organic fluorine in food packaging material?**

Speaker	Response
Olga Shimelis	Solvent extraction methods are a good place to start?
Rock J Vitale	As a first screening, perform a total fluorine analysis. If the outcomes are within a reasonable range, no additional steps need to be taken. More investigation of solvent extraction techniques is needed to understand which techniques will dissolve all PFAS compounds, including fluoropolymers, while keeping inorganic fluoride out of the equation.

**d. I am interested in the analysis of a variety of beverages. Are there any issues or points to keep in mind?**

Speaker	Response
Olga Shimelis	The methods used for water are a good place to start. Additional sample clean-up is likely to be necessary, though, and care should be taken that the additional sample clean-up doesn't absorb PFAS from the sample or introduce additional background contamination.
Ruth Marfil-Vega	There are three main areas to keep in mind: <ol style="list-style-type: none"> <li>1. Minimize the presence of PFAS coming from the instrument used. You can learn about the essentials of PFAS analysis by LC-MS/MS <a href="#">in this report</a>.</li> <li>2. Eliminate from your workflow those consumables that may contain PFAS. If this is not possible, make sure you check every batch of each consumable you use in your analysis.</li> <li>3. Use the same good laboratory practices and sample preparation techniques you normally use for the analysis of other trace organics in the beverages of interest.</li> </ol>

**e. What methodology might be used to measure the transfer of PFAS from food containers to the food itself?**

Speaker	Response
Olga Shimelis	This is outside our area of expertise.
Ruth Marfil-Vega	Extraction of the food container or packaging using similar conditions to the regular storage or usage of the packaging will provide the most real-world sample. Sampling of the packaging before and after such stress testing of the material would provide a way to measure loss. Measurement of the medium used for extraction (either aqueous or lipid-based) could provide direct measurement.  In the upcoming new year, five states are banning the use of added PFAS in food packaging, with other states imposing strict limits: <a href="#">American Bar Association</a> .  Check out one of our <a href="#">magazine issues that discusses food packaging testing</a> .

**f. Food packaging is regulated at 60-100 ppm as measured in total organic fluorine. What's the best method to determine total organic fluorine?**

Speaker	Response
Olga Shimelis	No methods have been promulgated yet to our knowledge, but a solvent extraction method would be a good place to start.
Rock J Vitale	See "C" above

**g. In the case of food packaging, should the analysis focus on PFAS transferred to the food rather than TOF, and how would you go about that?**

Speaker	Response
Olga Shimelis	This is outside our area of expertise. For an overview of this topic, the FDA has a website addressing food contact: <a href="https://www.fda.gov/food/process-contaminants-food/authorized-uses-pfas-food-contact-applications">https://www.fda.gov/food/process-contaminants-food/authorized-uses-pfas-food-contact-applications</a>
Ruth Marfil-Vega	Deciding which parameter, specifically PFAS or TOF, will depend on the question that needs to be answered. TOF is a sum parameter, similar to TOC (Total Organic Carbon) or TN (Total Nitrogen), that provides the amount of organic fluoride in the sample. If end user wants to quantify the concentration of a specific PFAS (e.g., the seven priority PFAS for the FDA), TOF won't be suitable for this purpose

**4. PFAS in Pharmaceuticals**

**a. What is known about the types and quantities of PFAS in pharmaceuticals, and what role do they play in these products? Is it considered an 'essential use'?**

Speaker	Response
Olga Shimelis	We do not have any data on this topic.

**b. What is your view on the PFAS monitoring of pharmaceutical products as potential leachables from packaging?**

Speaker	Response
Olga Shimelis	This is outside our area of expertise. Sources such as <a href="https://www.elsiedata.org/">https://www.elsiedata.org/</a> or <a href="https://niimbl.my.site.com/s/">https://niimbl.my.site.com/s/</a> would have more information on this topic.

**c. Will PFAS ever be established as a quality requirement for pharmaceutical manufacturing?**

Speaker	Response
Olga Shimelis	This is outside our expertise. Monitoring the activities of your local regulatory agencies is the best way to stay informed on any such future requirements.

**d. I have recently been tasked with setting up a testing platform for PFAS in medical devices and pharmaceuticals. I am interested in the target compounds that the regulatory agencies are focusing on and the levels of detection being applied in the medical device, pharmaceutical, and personal care product industries.**

Speaker	Response
Olga Shimelis	See our answer above.

## 5. PFAS in Air

### a. I am wondering if there is any analytical method that can detect PFAS in vapors, aerosols, or air?

Speaker	Response
Olga Shimelis	OTM 45 and OTM 50 (Draft) are the only published methods we're aware of. Academic researchers, however, have published numerous studies using internally developed methods for PFAS in air.
Rock J Vitale	Sample using OTM 45, then extract and shoot onto an LC/MS/MS.
Ruth Marfil-Vega	Yes, there are analytical methods that can be used to analyze specific classes of PFAS in the types of samples mentioned above. They are normally based on the use of GCMS in combination with different sampling techniques. An example of a GCMS method can be found <a href="#">here</a> . Also, PFAS can be sorbed onto different SPE materials and then eluted for conventional LCMS analysis.

### b. What is the status of ambient air testing methods and regulations? What is the status of testing methods and regulations for occupational exposure?

Speaker	Response
Olga Shimelis	We are not aware of any regulations or promulgated methods for PFAS occupational exposure.
Ruth Marfil-Vega	In the United States, the regulations and testing methods are still being developed. For now, the EPA has published methods OTM-45 and Modified TO-15 for the analysis of PFAS; however, additional methods are being developed ( <a href="https://www.epa.gov/water-research/pfas-analytical-methods-development-and-sampling-research">https://www.epa.gov/water-research/pfas-analytical-methods-development-and-sampling-research</a> ).

## 6. PFAS in Hydrocarbons

### a. Is there any validated method for PFAS substance determination for polymeric materials?

Speaker	Response
Olga Shimelis	Not of which we are aware.

### b. With respect to the analysis of PFAS in other matrices, in fields like petrochemicals and plastic pyrolysis oils, what would be a good approach to start looking for PFAS in such complex matrices?

Speaker	Response
Olga Shimelis	Extraction and sample clean-up will be your largest challenges. Consult your lab materials provider; they may have specific products to suggest as you start your experimentation.

### c. Can anyone comment on sample preparation and analytical techniques for hydrocarbon matrices?

Speaker	Response
Olga Shimelis	XAD2 has been used for gaseous hydrocarbon streams, i.e., emissions testing. This is the only application we are aware of currently.

### d. I should have expanded my original question beyond SPE. Do you have recommended sample prep techniques and/or analytical tools for hydrocarbon matrices?

Speaker	Response
Olga Shimelis	This will depend heavily on the analytes and matrix chemistries/interactions. Consulting various vendors may help identify potential solutions, but we are not aware of any published data for this application.

### e. What adsorbents or SPE cartridges are recommended for PFAS in hydrocarbon streams?

Speaker	Response
Olga Shimelis	See the response above.

## PFAS Treatment

### 1. What are the most promising and effective strategies for the remediation and management of PFAS contamination in the environment, and how can we strike a balance between addressing the existing contamination and preventing future PFAS pollution?

Speaker	Response
Olga Shimelis	There are many start-ups and academic groups developing novel solutions for PFAS remediation and destruction. Monitoring trade publications and scientific journals is a useful source of information for these developments.

### 2. What protocol should be used to clean contaminated soil when PFAS-containing fire extinguishers have been extensively used in military and firefighter training?

Speaker	Response
Olga Shimelis	We recommend contacting a local engineer or contractor. Local regulations and permitting will be the primary considerations in developing protocols.

### 3. Is adsorption an adequate technology to remove PFAS?

Speaker	Response
Olga Shimelis	Adsorption has been used successfully for many years as the primary solution to PFAS in drinking water.

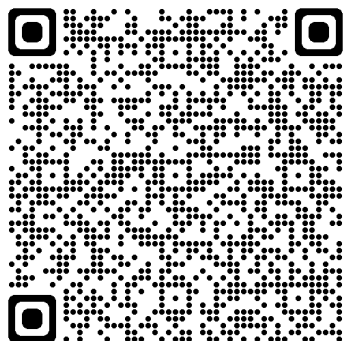
### 4. Do you see any benefits for the removal of PFAS from leachate at a landfill?

Speaker	Response
Olga Shimelis	Yes. Landfill leachate is an easily monitored and treated source. After the leachate is discharged, removal and remediation become much more difficult.



Learn more or contact us at

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