Guest Author: Grace Guo, Phenomenex Technical Specialist (find her on Live Chat!)

You wake up at 6 am and feel a little bit dehydrated. So you go to the kitchen, grab your mug, and get some fresh tap water. But wait, do you really trust you're drinking water? You might unknowingly intake something that will never come out of your body again. Per- and polyfluorinated alkyl substances (PFAS), which were thought to be inert and nontoxic, have found extensive usage in surfactants, firefighting foams, and commercial products like non-stick cooking pans, food packaging and outdoor clothing. During National Water Quality Month, learn about how the world of water analysis is evolving to keep up with the environmental impact of PFAS chemicals.

However, these chemicals are very persistent in the environment and do not break down in the body. They tend to bio-accumulate in organisms and have been linked to harmful health effects. And exposure to these human-made chemicals that you've never heard of until recently is more common than you may have thought. In fact, most or likely all of us have trace amounts in our blood streams. Eating the vegetables grown in PFAS-contained soil, cooking food with a nonstick pan, packing leftover with a grease-proof box are all sources of exposure.

These chemicals have actually been around since the 1930's,and have since spread in trace amounts throughout the globe. Drinking water sources are among the most critical, especially near contaminated sites with potentially the most abundant exposure, such that was featured in the movie, [Dark Waters](). The movie chronicles the cover-up and exposure of disposal of these chemicals in sources that reached the drinking water, leading to deaths in a West Virginia city from chronic exposure of high levels. I haven't seen the movie yet, but my environmental analytical chemist friends say it was pretty good – light on the science, but the science that was there was accurate.

Among the PFAS, perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA) are the most well studied compounds. But there are literally thousands of PFAS compounds, and their analytical characterization is important for toxicology, remediation, environmental pollution sources, and drinking water safety.

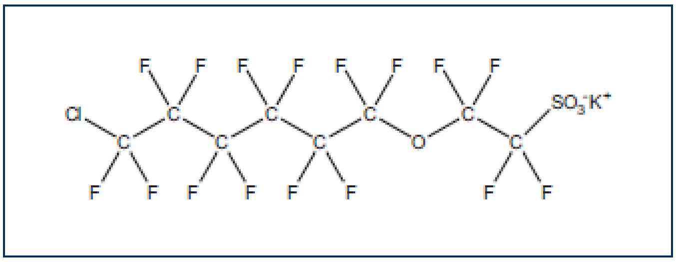
So, let’s take a closer look at some of the analytical techniques that scientists use to research them.

On December 19th, 2019, the EPA made official the most recent method for Per- and Polyfluoroalkyl Substances (PFAS) in drinking water, EPA Method 533.

EPA
United States
Environmental Protection
Agency

METHOD 533: DETERMINATION OF PER- AND POLYFLUOROALKYL SUBSTANCES IN DRINKING WATER BY ISOTOPE DILUTION ANION EXCHANGE SOLID PHASE EXTRACTION AND LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY

This newest addition focuses on shorter-chained PFAS and incorporates isotopic dilution to minimize sample background interference. It also includes compounds associated with newer replacement fluorinated material GenX, F-53B, and ADONA.



9Cl-PF3ONS (main component of F-53B)

Some of the routine analytical challenges with PFAS analytical methods are recovery and retention of some of the shorter chained acids are maintaining selectivity across prominent mid-chained and branched isomers of interest. Strata X-AW polymeric mixed-mode solid phase extraction cartridges (8B-S038-HCH) accomplish the former, and Gemini C18 HPLC columns (00B-4439-B0) manage the latter; both of which were relied on during the method development and multi-lab validation. The silica-polymer hybrid base of Gemini media results in a rugged, pH stable media that has high surface area for improved retention and sensitivity of the more polar short chained PFAS compounds like PFBA, and still maintains selectivity and resolution of the prominent branched isomers of PFHxS and PFOS. These front end techniques coupled to a SCIEX Triple Quad™ 5500 mass spectrometer have been relied on for trace quantitative analysis to ensure safe drinking water.

Since PFAS compounds have become ubiquitous, many components of lab instruments and solvents can contribute background PFAS that could potentially interfere with the trace sample quantification. As such, reducing background contamination is critical to obtaining low detection limits and accurate results. Besides using PEEK tubing and stainless-steel solvent filters to replace ethylene tetrafluoroethylene (ETFE) lines and polytetrafluoroethylene (PTFE) solvent filters, a delay column or "Trap" column can be placed between the pumps and autosampler to distinguish system related PFAS interferences.

Background PFAS will be slowed down on the delay column and come out separated just after the analytical peaks of interest. Normally the delay column should be more retentive or the same as the analytical column. A high surface area C18 column like Luna C18(2) is recommended as a delay column, which has become a simple, but elegant way to reduce background interference.

To sum up, PFAS analysis at low levels (ppt level) in drinking water matrix is not an easy task. However, with the help of analytical experts, critical environmental and safe drinking water techniques are routinely implemented in labs across the globe.

To learn more you can visit www.phenomenex.com/pfasresources or even chat with the author of this article, Grace Guo, through Live Chat today!

References:

- Method 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass

Spectrometry (LC/MS/MS): https://cfpub.epa.gov/si/si_public_record_Report.cfm?dirEntryId=343042&Lab=NERL)

- Method 533: Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry: https://www.epa.gov/dwanalyticalmethods/method-533-determination-and-polyfluoroalkyl-substances-drinking-water-isotope

- Quantitation of PFASs in Water Samples using LC-MS/MS Large Volume Direct Injection and Solid Phase Extraction: https://sciex.com/Documents/tech%20notes/PFAS-water-samples.pdf)

- Analysis of PFAS in drinking water with EPA Method 537.1 and the SCIEX QTRAP® 4500 System: https://sciex.com/Documents/tech%20notes/applications/environ/PFAS-drinking-water.pdf)

- Per- and Polyfluorinated Alkyl Substances (PFAS) from Milk, Eggs, Butter, Cheese, and Fish using QuEChERS, SPE, and LC-MS/MS: https://phenomenex.blob.core.windows.net/documents/a9406d77-c88e-49cb-9347-81efd828fc25.pdf)

- Rapid Analysis of 23 Per- and Poly-Fluorinated Alkyl Substances (PFASs) by UHPLC-MS/MS using Luna® Omega 1.6µm PS C18: https://az621941.vo.msecnd.net/documents/234361ad-5022-4cad-bb0a-5514059a1ba5.pdf