FACT SHEET: AUGUST 2020

# A SHORT GUIDE TO COMMON TESTING METHODS

## FOR PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS)

A challenge to eliminating the use and exposure to per- and polyfluoroalkyl substances (PFAS) is knowing where these chemicals are used and found. Testing, be it of products, water, or food, is critical to ascertain whether PFAS are present and at what levels. But what test methods are appropriate in which application? This guide to PFAS test methods is to help manufacturers, researchers, government agencies, and NGOs understand the different types of PFAS testing techniques available to support their work in knowing where PFAS are found and at what level.

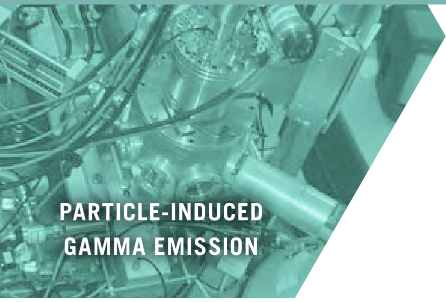
## PFAS BACKGROUND

Per- and polyfluoroalkyl substances (PFAS) are a class of fluorinated organic chemicals that contain at least one fully fluorinated carbon atom, with over 5,000 chemicals identified in the class. PFAS are used in a wide variety of products, from non-stick cookware and textiles to firefighting foams and food packaging. One of the main concerns surrounding PFAS is their high potential to persist in the environment. Given their ubiquitous use and persistence, testing for PFAS is an important part of monitoring and limiting their continued use in supply chains.

This fact sheet focuses on testing techniques and methods that can be used to identify PFAS in food packaging, firefighting foams, and drinking water. While some test methods apply to multiple products and drinking water, there are few standardized PFAS test methods, which makes comparing results between different laboratories and studies difficult.

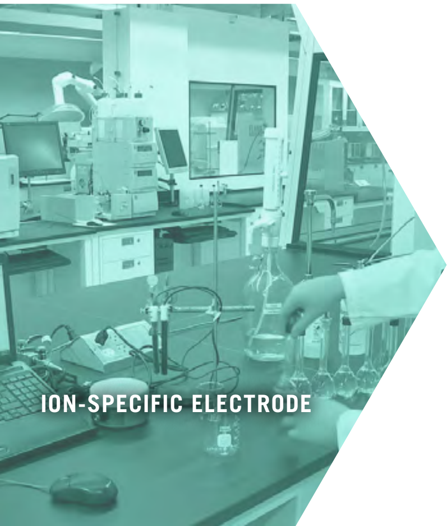
## TESTING TECHNIQUES

PFAS analytical testing techniques can be divided into two categories: (1) targeted testing, and (2) total fluorine test methods. Targeted testing techniques measure a subset of PFAS (e.g., 30 of the 5,000 PFAS chemicals), while total fluorine tests are indirect methods designed to measure a representative element indicative of PFAS.


PARTICLE-INDUCED GAMMA EMISSION


COMBUSTION ION CHROMATOGRAPHY


INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS


ION-SPECIFIC ELECTRODE

## TOTAL FLUORINE METHODS

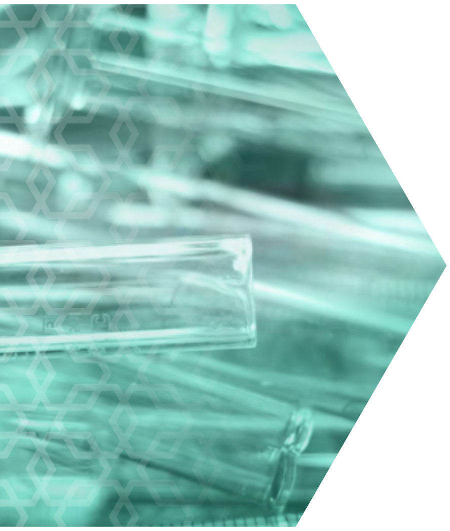
Total fluorine techniques measure either total organic fluorine or total fluorine. These techniques are efficient ways to identify whether PFAS are likely present. They can be used for screening as well as in a tiered approach to quantify PFAS in a product.

Total fluorine methods include: (1) particle-induced gamma emission (PIGE), (2) combustion ion chromatography (CIC), (3) instrumental neutron activation analysis (INAA), (4) ion-specific electrode (ISE), and (5) x-ray photoelectron spectroscopy (XPS). CIC can be used to quantify total organic fluorine or total fluorine. PIGE can be used to quantify total fluorine. PIGE, XPS, and INAA are non-destructive, meaning a single sample can be analyzed multiple times, which is beneficial when using more than one technique. PIGE and XPS are surface measurements, providing information on fluorine present on the surface rather than the concentration in the entire sample. CIC and ISE are both destructive, requiring combustion of the product sample, and measure fluorine in the entire sample. Table A includes more specific information on these methods.

## TARGETED TESTING

Targeted testing techniques identify and quantify the presence of a specific set of PFAS. The chemicals of interest being identified and quantified are called analytes. The majority of targeted test methods use chromatography, with most relying on a combination of gas or liquid chromatography (GC or LC) and mass spectrometry (MS) analysis. GC/MS and LC/MS methods are used to both identify and quantify different types of specific chemical analytes.

Standards are required to identify and quantify an analyte and, to date, less than 100 of the 5,000 PFAS can be identified and quantified using targeted testing. Targeted testing techniques have the advantage of positively identifying the specific chemical as well as the concentration of that chemical in the sample. In addition, there are standardized and validated test methods in this category, such as those for drinking water. The primary disadvantage is that in most cases these methods measure only a small fraction of the PFAS that may be present in the

sample, missing those present in polymers and others not on the list of target analytes. The table includes more specific information on these techniques.

Two other methods incorporate LC/MS, namely quadrupole time-of-flight mass spec (QTOF-MS) and total oxidizable precursors (TOP) assay. QTOF-MS can determine a wide range of compounds in the PFAS family. TOP assay is used to convert precursors to PFAS that can be quantified using standard methods.

## STANDARDIZED AND VALIDATED TEST METHODS

Most of the standardized and validated test methods are written for drinking water, groundwater, and open waterways. They are all chromatography-based methods, with differences mainly appearing in the sample preparation step. The complex nature of samples containing multiple materials, such as packaging, require significantly more sample preparation and clean-up when compared to drinking water. The number of analytes that can be determined varies depending on which method is employed and ranges from 11 to 40. The US Environmental Protection Agency (EPA) test method 537.1 is commonly referenced as it is a multi-laboratory validated test method.

PFAS testing is an evolving science that will benefit from standardized test methods for a wider variety of matrices and analytical techniques. Standardization allows for better reproducibility and comparison of PFAS determination, which will become more important as governments, agencies, and supply chains aim to reduce and eliminate the use of PFAS.

| ANALYTICAL TECHNIQUES | APPLICATIONS | TARGET SUBSTANCE(S) | ADDITIONAL INFORMATION |
|---|---|---|---|
| COMBUSTION ION CHROMATOGRAPHY (CIC) | Food packaging<br>Firefighting foams<br>Water samples | Total fluorine or total organic fluorine | • Offers possibility of fast, accurate analysis<br>• Destructive to sample<br>• Direct combustion of the sample measures the total fluorine of the entire sample, independent of thickness. Direct combustion can be combined with a separate measurement of total inorganic fluorine through water extraction of the sample followed by combustion to determine total, inorganic, and organic fluorine.<br>• Instead of directly combusting the sample, the organic fluorine can be either extracted or adsorbed and then combusted to measure total organic fluorine (either extractable or adsorbable). These techniques result in lower limits of reporting than direct combustion. These techniques are known as Extractable Organic Fluorine (EOF) and Adsorbable Organic Fluorine (used for waters/ wastewater) methods.<br>• Technique referenced in Clean Production Action's firefighting foam standard - 1 ppm total organic fluorine threshold requirement for certification<br>• Commercially available |
| INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS (INAA) | Food packaging<br>Textiles<br>Other organic materials | Total fluorine | • Measures total of entire sample, independent of thickness<br>• Non-destructive and rapid<br>• Since technique relies on nuclear, rather than chemical reaction, samples may be analyzed without dissolution or decomposition<br>• No chemical prep needed<br>• Samples are irradiated followed by a decay period, emitting gamma rays, and target nuclide identified via gamma ray spectroscopy. Quantification accomplished by comparison with standards.<br>• Not commercially available |
| ION-SPECIFIC ELECTRODE (ISE) | Food packaging | Total fluorine | • Combustion with known amount of buffer solution, then analyzed with fluoride-specific electrode<br>• Destructive method, low-cost<br>• Commercially available |
| PARTICLE-INDUCED GAMMA EMISSION (PIGE) | Food packaging<br>Firefighting foam | Total fluorine | • Surface measurement, so results dependent on sample thickness<br>• Good result accuracy, well-used, cost-effective<br>• Not commercially available |
| QUADRUPOLE TIME-OF-FLIGHT-MASS SPEC (QTOF-MS) | Water samples<br>Materials | Full range of potential compounds in the PFAS family | • QTOF-MS combines TOF and quadrupole instruments, a pairing that results in high mass accuracy; speed and sensitivity are benefits of the QTOF<br>• Coupled with LC or GC for analyte determination<br>• Expensive and time consuming |
| TOTAL OXIDIZABLE PRECURSORS (TOP) ASSAY | Foam products<br>Textiles<br>Water samples | Quantifies total amount of chemical precursors to perfluoroalkyl acids (PFAAs) | • Selective PFAS method (only those that can be oxidized to form targeted PFAAs)<br>• Destructive, relatively rapid, low cost<br>• Sample treated so precursor substances contained within the sample are oxidized, then PFAS determination done using methods like LC-MS/MS |

| STANDARDIZED TEST METHODS | APPLICATIONS | TARGET SUBSTANCE(S) | ADDITIONAL INFORMATION |
|---|---|---|---|
| DIN EN ISO 10304-1 | Water samples | Total organic fluorine | • Validated standardized test method is for water samples |
| ASTM D7979 | Water samples | Individual PFAS (39 analytes) | • Direct injection method, LC-MS/MS analysis<br>• Performance-based method (i.e., can adjust measurement sensitivity), wider range of analytes |
| ISO METHOD 25101 | Unfiltered drinking water | Determination of linear isomers of PFOS and PFOA | • Solid phase extraction (SPE), high performance mass spec (HPLC-MS/MS) technique |
| USEPA METHOD 533 | Drinking water | Individual PFAS (25 analytes) | • Isotope dilution anion exchange, solid phase extraction (SPE), and liquid chromatography/ mass spectrometry (LCMS/MS) techniques |
| USEPA METHOD 537 | Drinking water | Selected PFAAs | • Solid phase extraction (SPE) liquid chromatography/tandem mass spectrometry (LCMS/MS) techniques utilized |
| USEPA METHOD 537.1 | Drinking water | Individual PFAS (18 analytes) | • Multi-laboratory validated<br>• Solid phase extraction (SPE), liquid chromatography/tandem mass spectrometry (LCMS/MS) techniques utilized |

## CFE GUIDING STAR

*Within our generation, we will lift the burden of cancers and other diseases by driving a dramatic & equitable transition from toxic substances in our lives, communities and economy to safe and healthy alternatives for all.*

## FOR MORE INFORMATION

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