Per- and Polyfluorinated Alkyl Substances (PFAS): *The Determination of Total Oxidizable Precursors (TOPS)*

TECHNICAL BULLETIN

Perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA), as well as related per- and polyfluorinated alkyl substances (PFAS) are man-made chemicals that, because of their stability under extreme heat and chemical stress, as well as their surfactant properties, have been used in a variety of applications including industrial polymers (Teflon™), Stain repellants (Scotch Guard™), Aqueous film forming foams (AFFF or firefighting foams).

PFAS have received a substantial amount of attention not only because they are recognized as ubiquitous environmental contaminants, but because these compounds persist, bioaccumulate and have been associated with toxicity in some animal studies. PFAS have been identified as compounds of environmental concern at numerous sites across North America and around the world.

Problem Statement

While PFAS compounds can be released into the environment directly, there is evidence[1] that some may also be formed in situ from the conversion of so-called "precursor" compounds that either already exist in the environment as contaminants, or are co-released with the target PFAS.

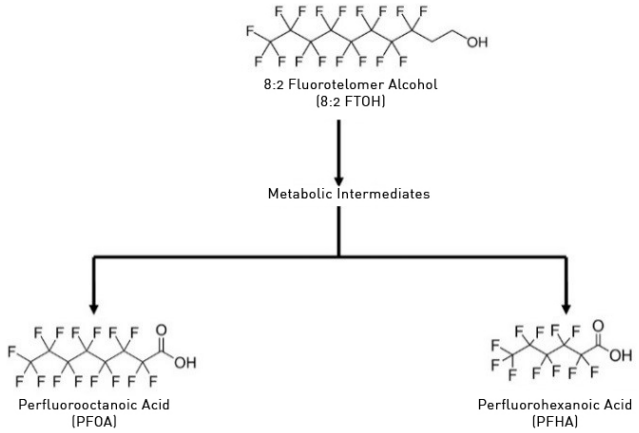

Figure 1: Transformation of 8:2 Fluorotelomer Alcohol (8:2 FTOH) ("precursor") to Perfluorooctanoic Acid (PFOA) and Perfluorohexanoic Acid (PFHxA) ("end products").

Precursor transformation to PFAS "end products" has important implications for PFAS remediation efforts. By solely focusing on target PFAS removal, without consideration of the total precursor pool, an unanticipated increase in the concentrations of target PFAS may occur over time, resulting in potential future liability.

Total Oxidizable Precursors (TOPs) Assay

Maxxam currently provides analyses of up to twenty five (25) PFAS (including PFOS and PFOA), nine (9) of which are known precursor compounds, in a diverse range of environmental matrices.

However, the results from these analyses only represent the concentrations of these twenty-five (25) PFAS at the time the sample was collected. They do not measure the potential for PFAS formation due to transformation of precursor compounds over time.

One approach to measure the potential for PFAS formation in water and soil is to apply the total oxidizable precursors (TOPs) assay[2].

[1] Wang, et. al. 2005 Environ. Sci. Technol., 39, 7516-7528.

[2] Houtz, E.F. and Sedlak, D.L. (2012), *Environ Sci. Technol.*, 46, 9342-9349

Analytical Method Summary

Maxxam offers the TOPs assay on a variety of matrices, including: surface water; groundwater; wastewater; drinking water; and soils.

The TOPs assay requires that samples be collected in duplicate.

1. One part of the duplicate sample is analysed as received, for the 25 measured PFAS to establish the initial concentrations of the PFAS end products.
2. The second part of the duplicate sample is heated with an oxidizing agent under alkaline conditions to transform potential precursor compounds, both known (the nine that are monitored by the PFAS Test) and unknown, into PFAS “end products."
3. The oxidized sample is then submitted for analysis of the 25 PFAS to determine any increase in PFAS end product concentrations.
4. The difference between the initial and final concentrations of the PFAS end products represents the presence of per- and polyfluorinated precursor compounds.

Quantitative analyses of the target list PFAS and precursor compounds is performed by using isotope dilution liquid chromatography coupled with tandem mass spectrometry (LC/MS/MS).

Laboratory Accreditation

Maxxam is accredited by the Standards Council of Canada (SCC), the US National Environmental Laboratory Accreditation Program (NELAP) and the US Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP) for the analysis of PFAS in environmental matrices.

Reported Parameters

For the TOPs assay Maxxam reports initial and oxidized concentrations for the twenty-five (25) PFAS as well as the calculated “difference”.

Sample Containers/Hold Times

Water

Samples should be collected in high density polyethylene (HDPE) bottles, provided by the laboratory, and fitted with an unlined (Teflon-free) polypropylene screw cap.

A minimum of 250 mL (2 x 125 mL) of sample is required. The sample hold time is 14 days with proper storage (1-6° C, minimum exposure to light).

Soil

Samples should be collected in high density polyethylene (HDPE) wide-mouth bottles, provided by the laboratory and fitted with an unlined (Teflon-free), polypropylene screw cap. A minimum of 50 g of sample is required. In the absence of any regulated sample hold time, Maxxam adheres to a 28 day hold time for solids with proper storage (1-6° C, minimum exposure to light).

Because of the ubiquitous nature of PFAS compounds in many modern materials, all batches (lots) of sample containers provided by Maxxam, used for collecting samples for PFAS determinations, are “proofed” by the laboratory to demonstrate that they are PFAS-free. Similarly, water used in the field to generate quality control (QC) samples should be PFAS-free.

For a nominal fee, Maxxam will provide PFAS-free water which has been “proofed” by the laboratory.

Analytical Turnaround Time (TAT)

Standard TAT: 15 business days

Priority TAT: By pre-arrangement only.

Pricing

Water	Soil
$1,200/sample	$1,200/sample

Analytical Limitations

The results of the TOPs assay provide an indication of per- and polyfluorinated materials in a sample that may transform to the more persistent end products. However, it is not without limitations that need to be considered when interpreting the results:

- The number of potential precursor compounds in a sample may be significantly higher than the nine (9) PFAS compounds routinely measured as part of the test.

Therefore, not all of the PFAS end products produced by the TOPs assay necessarily result from the oxidation of the nine precursors measured as part of the analysis. Moreover, calculating the increase in end product concentrations may only represent part of the full scope of the precursors present in the sample since the assay only applies to compounds that are oxidizable via this method.

- The oxidation of precursor compounds (known and unknown) may result in intermediate fluorinated compounds or other PFAS that are not measured as part of the analysis. Thus measuring increases or decreases in only the 16 routine PFAS may only represent a portion of the potentially transformable precursors in the sample.
- Complete oxidation cannot be confirmed beyond the nine (9) precursor compounds measured as part of Maxxam's routine target compound list. Therefore, the TOPs assay may only represent the minimum potential PFAS transformation.

About Us

Maxxam is a leading North American provider of analytical services and solutions to the energy, environmental, food, Industrial Hygiene and DNA industries. We are a member of the Bureau Veritas Group of companies – a world leader in testing, inspection and certification services. We support critical decisions made by our customers through the application of rigorous science and the knowledge and expertise of over 2,500 employees.

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