## Method Summary

# TOP Assay in Soils by LC-MS/MS.

## Scope and Range

This procedure describes an assay by which samples can be oxidized prior to analysis, in an attempt to determine the concentration of total oxidizable precursors. It is appropriate for soils. The detection limit for this method is based on 1g of sample being used for the extraction; however, the detection limit will vary if limited sample is available for extraction or any dilutions are required.

Quantitation range: 1 to 200 µg/Kg or higher with dilutions

| **Perfluoroalkylcarboxylic Acids** | | **CAS** | **µg/Kg** |
|---|---|---|---|
| PFBA | perfluoro-n-butanoic acid | 375-22-4 | 3 |
| PFPA | perfluoro-n-pentanoic acid | 2706-90-3 | 3 |
| PFHxA | perfluoro-n-hexanoic acid | 307-24-4 | 2 |
| PFHpA | perfluoro-n-heptanoic acid | 375-85-9 | 1 |
| PFOA | perfluoro-n-octanoic acid | 335-67-1 | 1 |
| PFNA | perfluoro-n-nonanoic acid | 375-95-1 | 1 |
| PFDA | perfluoro-n-decanoic acid | 335-76-2 | 1 |
| PFUnA | perfluoro-n-undecanoic acid | 2058-94-8 | 1 |
| PFDoA | perfluoro-n-dodecanoic acid | 307-55-1 | 1 |
| **Perfluoroalkylsulfonates#** | | | |
| PFBS | perfuoro-1-butanesulfonate | 375-7-5 | 1 |
| PFPeS | perfluoro-1-pentanesulfonate | 2706-91-4 | 1 |
| PFHxS | perfluoro-1-hexanesulfonate | 355-46-4 | 1 |
| PFHpS | perfluoro-1-heptanesulfonate | 375-92-8 | 1 |
| Linear PFOS | perfluoro-1-octanesulfonate | 1763-23-1 | 1 |
| Branched PFOS | (mixture of isomers) | - | 1 |
| Total PFOS | (sum of linear and branched) | - | 1 |
| PFDS | perfluoro-1-decanesulfonate | 335-75-3 | 1 |
| **Perfluorooctanesulfonamides** | | | |
| PFOSA | perfluorooctanesulfonamide | 754-91-6 | 1 |
| **Fluorinated Telomer Sulfonates** | | | |
| 6:2FTS | 6:2 Fluorotelomer sulfonate | 27619-97-2 | 1 |

**Table 1 List of per- and polyfluorinated compounds contained within suite and associated limits of detection.**

# - The listed CAS numbers refer to the parent perfluoroalkylsulfonic acid. It should be noted that the method detects the pefluoroalkylsulfonate base anion which may derive from a range of substances, such as the parent acid and salts of the acid

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Agilent Application Note 5989-6577EN, Quantitative analysis of perfluorooctanoic acid by LC/MS/MS (2007).

DIN 38414-14 Determination of selected polyfluorinated compounds (PFC) in sludge, compost and soil – Method using high performance liquid chromatography and mass spectrometric detection (HPLC-MS/MS).

'Persistence of Perfluoroalkyl Acid Precursors in AFFF-Impacted Groundwater and Soil' Houtz E F, Higgins C P, Field J A, Sedlak D L *Environ. Sci. Technol.* 2013

## Principle

Samples are extracted using basic methanol and then oxidized. Once oxidized, they are then extracted using solid phase extraction (SPE) and analysed by liquid chromatography coupled with a triple quadrupole mass spectrometer (LC-MS/MS).

## Quality Assurance

A known amount of M6:2FTS is added to each sample prior to oxidation. Measurement of the amount remaining following oxidation is to check for incomplete oxidation. There should be a minimum of 90% removal for the process to be confirmed as satisfactory.

$[M6:2\ FTS]_{post\text{-}TOP} / [M6:2\ FTS]_{pre\text{-}TOP} = <10\%$

The total amount of perfluoroalkylcarboxylates in the sample post-oxidation would be expected to be greater than the amount present pre-oxidation.

$[Total\ perfluoroalkyl\ carboxylates]_{post\text{-}TOP} \geq [Total\ perfluoroalkyl\ carboxylates]_{pre\text{-}TOP}$

The total amount of perfluoroalkyl sulfonates in the sample post-oxidation would be expected to be similar to the amount present pre-oxidation.

$[Total\ perfluoroalkyl\ sulfonates]_{post\text{-}TOP} = [Total\ perfluoroalkyl\ sulfonates]_{pre\text{-}TOP}$

## Interferences

Extracted samples may contain interferences from other compounds contained within the sample matrix. Using the principles of MS/MS, many of these interferences will be eliminated. However, there may be occasions when interferences from non-target compounds arise from similar precursor and product ions. In these cases, reported limits of detection may be raised.