



**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

<b>Perfluoroalkylcarboxylic Acids</b>		<b>CAS</b>	<b>µg/Kg</b>
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
<b>Perfluoroalkylsulfonates#</b>			
PFBS	perfluoro-1-butanefluorosulfonate	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
<b>Perfluorooctanesulfonamides</b>			
PFOSA	perfluorooctanesulfonamide	754-91-6	1
<b>Fluorinated Telomer Sulfonates</b>			
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 perfluoroalkylsulfonate base anion which may derive from a range of substances, such as the parent acid and salts of the acid

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Page 2 of 2



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**References**

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).

**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.