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TM441 - TOP Assay in Waters method summary

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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 ground water, effluents and similar water matrices. The detection limit for this method is based on 25ml 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: 2 to 400 ng/L or higher with dilutions

Perfluoroalkyl carboxylic Acids (PFCA)	CAS	LOD (ng/L)
PFBA	375-22-4	4
PFPA	2706-90-3	2
PFHxA	307-24-4	2
PFHpA	375-85-9	2
PFOA	335-67-1	2
PFNA	375-95-1	2
PFDA	335-76-2	4
PFUnA	2058-94-8	4
PFDoA	307-55-1	4
PFTrDA	72629-94-8	6
PFTeA	376-06-7	2
PFHxDA	67905-19-5	2
PFODA	16517-11-6	2
Perfluoroalkylsulfonic Acids (PFSA)		
PFBS	375-73-5	2
PFPeS	2706-91-4	2
PFHxS	355-46-4	2
PFHpS	375-92-8	2
Linear PFOS	1763-23-1	2
Branched PFOS	-	2
PFNS	68259-12-1	2
PFDS	335-77-3	4
PFUnDS	749786-16-1	4
PFDoS	79780-39-5	4
PFTrDS	791563-89-8	2
Fluorotelomer Sulfonates (X:2 FTS)		
4:2 FTS	757124-72-4	2
6:2 FTS	27619-97-2	2
8:2 FTS	39108-34-4	4
Perfluoroethylcyclohexanesulfonate (PFECHS)		
PFecHS	646-83-3	2

Perfluorooctanesulfonamides (FASA)	CAS	LOD (ng/L)
FBSA	30334-69-1	2
FHxSA	41997-13-1	2
PFOSA	754-91-6	4
N-MeFOSA	31506-32-8	2

N-EtFOSA	4151-50-2	2
Perfluoroalkanesulfonamidoethanols (FASE)		
MeFOSE	24448-09-7	20
EtFOSE	1691-99-2	20
Perfluorooctanesulfonamidoacetic Acids (FOSAA)		
MeFOSAA	2355-31-9	4
EtFOSAA	2991-50-6	4
Chloroperfluoroalkyl Ether Sulfonates (Cl-PFESA)		
9Cl-PF3ONS	756426-58-1	2
11Cl-PF3OUdS	763051-92-9	4
Fluorotelomer Carboxylic Acids (n:3 FTCA)		
3:3 FTCA	356-02-5	10
5:3 FTCA	914637-49-3	10
7:3 FTCA	812-70-4	10
Per- and Polyfluoroalkyl Ether Carboxylic Acids (PFECA)		
HFPO-DA	13252-13-6	4
HFPO-TA	13252-14-7	10
ADONA	919005-14-4	2
PFMOPrA	377-73-1	2
PFMOBA	863090-89-5	2
NFDHA	151772-58-6	6
Perfluoroalkyl Ether Sulfonates (PFESA)		
PFEESA	113507-82-7	2
Perfluoroalkyl Phosphate Di-esters (diPAP)		
Cationic/Zwitterionic PFAS		
N-CMAmP-6:2FOSA (6:2 FTAB)	34455-29-3	20

References

EPA Method 1633, Revision A – Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolid and Tissue Samples by LC-MS/MS.

Principle

Samples are first oxidized and 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 13C-6:2FTS 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.

$[\text{13C-6:2 FTS}]_{\text{post-TOP}} / [\text{13C-6:2 FTS}]_{\text{pre-TOP}} = <10\%$

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

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

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

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

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.