

TOP Assay SOP Addendum

SOP Title or Method Number: **SOP 23528**, Determination of Selected Perfluorinated Alkyl Substances by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry Isotope Dilution (LC/MS/MS)

Reference Documents: **EPA Method 537**, Version 1.1, September 2009, EPA Document #: EPA/600/R-08/09EPA; **Method 537.1**, Version 1, November 2018, EPA Document #: EPA/600/R-18/352; **Department of Defense, Quality Systems Manual for Environmental Laboratories**, Version 5.2, 2019

The following modifications to the referenced SOP are as follows:

Section 7: Water Bath – Capable of monitoring recording to .1 C maintaining 85 C.

Section 8.1

Sodium Hydroxide (NaOH, CAS#: 1310-73-2) – High purity, demonstrated to be free of analytes and interferences.

Potassium Persulfate (K₂S₂O₈, CAS#: 7727-21-1) - High purity, demonstrated to be free of analytes and interferences.

Hydrochloric Acid (HCl, CAS#: 7647-01-0) - High purity, demonstrated to be free of analytes and interferences.

Additional table to be added to Section 8

Table 1

Isotope Labeled Standard	Conc. Top Surr Stock (ng/mL)	Vol. of Top Surr Stock (mL)	Final Vol. of Top Surr PDS (mL)	Final Conc. of Top Surr PDS (ng/mL)
M3PFPeA	1000	1.0	2.0	500
M2PFHxA	1000	1.0	2.0	500
M4PFOA	1000	1.0	2.0	500
d3-N-MeFOSAA	1000	1.0	2.0	500
d5-N-EtFOSAA	1000	1.0	2.0	500
M2-4:2FTS	935	1.0	2.0	467.5
M2-6:2FTS	949	1.0	2.0	474.5
M2-8:2FTS	958	1.0	2.0	479

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Modified extracted internal standard list (Table 2) for post TOP extraction

Isotope Labeled Standard	Conc. of EIS Stock (ng/mL)	Vol. of EIS Stock (mL)	Final Vol. of EIS PDS (mL)	Final Conc. of EIS PDS (ng/mL)
M4PFBA	1000	1.0	2.0	500
M5PFPeA	1000	1.0	2.0	500
M5PFHxA	1000	1.0	2.0	500
M4PFHpA	1000	1.0	2.0	500
M8PFOA	1000	1.0	2.0	500
M9PFNA	1000	1.0	2.0	500
M6PFDA	1000	1.0	2.0	500
M7PFUdA	1000	1.0	2.0	500
MPFDoA	1000	1.0	2.0	500
M2PFTeDA	1000	1.0	2.0	500
M3PFBS	929	1.0	2.0	464.5
M3PFHxS	946	1.0	2.0	473
M8PFOS	957	1.0	2.0	478.5

Prior to Section 10.3, Cartridge SPE procedure, each sample treated with Potassium Persulfate until 60 mM (about 4 grams). Add Sodium Hydroxide until the sample is 125 mM (about 1.25 grams).

Sample should have a pH >12.

Fortify each sample with 20 µl of TOP pre-assay surrogate containing 5 negative control surrogates and 3 positive control surrogates from table 1.

Place sample in a water bath at 85 C for 6 hours.

Remove sample from water bath and adjust pH 6-8 with Hydrochloric Acid.

Following pre-treatment, extraction protocol unaltered.

Section 11, An additional calculation is added for the final reporting of the TOP assay results.

TOP Assay = Result of Post TOP assay extraction – Result of analysis from Pre-Top Assay extraction.