Designation: D7968 – 23

Standard Test Method for Determination of Polyfluorinated Compounds in Soil by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS)[1]

This standard is issued under the fixed designation D7968; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This procedure covers the determination of selected polyfluorinated alkyl substances (PFAS) in a soil matrix using solvent extraction, filtration, followed by liquid chromatography (LC) and detection with tandem mass spectrometry (MS/MS). These analytes are qualitatively and quantitatively determined by this method. This method adheres to multiple reaction monitoring (MRM) mass spectrometry. This procedure utilizes a quick extraction and is not intended to generate an exhaustive accounting of the content of PFAS in difficult soil matrices. An exhaustive extraction procedure for PFAS, such as published by Washington et al.,[2] for difficult matrices should be considered when analyzing PFAS. The approach from this standard was utilized to screen laboratory coats (textiles) to identify if PFAS would be leached from the materials.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 The method of detection limit[3] and reporting range[4] for the target analytes are listed in Table 1.

1.3.1 The reporting limit in this test method is the minimum value below which data are documented as non-detects. Analyte detections between the method detection limit and the reporting limit are estimated concentrations and are not reported following this test method. In most cases, the reporting limit is calculated from the concentration of the Level 1 calibration standard as shown in Table 2 for the PFAS after taking into account a 2 g sample weight and a final extract volume of 10 mL, 50 % water/50 % MeOH with 0.1 % acetic acid. The final extract volume is assumed to be 10 mL because 10 mL of 50 % water/50 % MeOH with 0.1 % acetic acid was added to each soil sample and only the liquid layer after extraction is filtered, leaving the solid and any residual solvent behind. It is raised above the Level 1 calibration concentration for PFOS, PFHxA, FHEA, and FOEA; these compounds can be identified at the Level 1 concentration but the standard deviation among replicates at this lower spike level resulted in a higher reporting limit.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[5]

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques

[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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[2] Washington, J. W., Naile, J. E., Jenkins, T. M., and Lynch, D. G., "Characterizing Fluorotelomer and Polyfluoroalkyl Substances in New and Aged Fluorotelomer-Based Polymers for Degradation Studies with GC/MS and LC/MS/MS," *Environmental Science and Technology*, Vol 48, 2014, pp. 5762–5769.

[3] The MDL is determined following the Code of Federal Regulations, 40 CFR Part 136, Appendix B utilizing solvent extraction of soil. A 2 g sample of Ottawa sand was utilized. A detailed process determining the MDL is explained in the reference and is beyond the scope of this standard to be explained here.

[4] Reporting range concentration is calculated from Table 2 concentrations assuming a 30 µL injection of the Level 1 calibration standard for the PFAS, and the highest level calibration standard with a 10 mL final extract volume of a 2 g soil sample. Volume variations will change the reporting limit and ranges.

[5] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Method Detection Limit and Reporting Range[A]

Analyte	MDL (ng/kg)	Reporting Limit (ng/kg)
PFTreA	6.76	25–1000
PFTriA	5.26	25–1000
PFDoA	3.56	25–1000
PFUnA	2.45	25–1000
PFDA	5.54	25–1000
PFOS	18.83	50–1000
PFNA	2.82	25–1000
PFecHS	2.41	25–1000
PFOA	6.24	25–1000
PFHxS	7.75	25–1000
PFHpA	5.80	25–1000
PFHxA	15.44	50–1000
PFBS	6.49	25–1000
PFPeA	20.93	125–5000
PFBA	22.01	125–5000
FHEA	199.04	600–20 000
FOEA	258.37	750–20 000
FDEA	137.46	500–20 000
FOUEA	4.85	25–1000
FhpPa	5.09	25–1000
FHUEA	3.50	25–1000

[A] Abbreviations are defined in 3.2.

2.2 *Other Documents:*[6]

EPA SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

40 CFR Part 136 Appendix B Definition and Procedure for the Determination of the Method Detection Limit

3. Terminology

3.1 *Definitions:*

3.1.1 *reporting limit, RL, n*—the minimum concentration below which data are documented as non-detects.

3.2 *Abbreviations:*

3.2.1 *CCC*—Continuing Calibration Check

3.2.2 *IC*—Initial Calibration

3.2.3 *ppt*—parts per trillion, ng/kg or ng/L

3.2.4 *LC*—Liquid Chromatography

3.2.5 *LCS/LCSD*—Laboratory Control Sample/Laboratory Control Sample Duplicate

3.2.6 *MDL*—Method Detection Limit

3.2.7 *MeOH*—Methanol

3.2.8 *mM*—millimolar, 1×10^{-3} moles/L

3.2.9 *MRM*—Multiple Reaction Monitoring

3.2.10 *MS/MSD*—Matrix Spike/Matrix Spike Duplicate

3.2.11 *NA*—Not available

3.2.12 *ND*—Non-detect

3.2.13 *P&A*—Precision and Accuracy

3.2.14 *PFAS*—Perfluoroalkyl substances

3.2.15 *PFBS*—Perfluorobutylsulfonate

3.2.16 *PFHxS*—Perfluorohexylsulfonate

3.2.17 *PFOS*—Perfluorooctylsulfonate

3.2.18 *PFecHS*—Decaluoro-4-(pentafluoroethyl)cyclohexanesulfonate

3.2.19 *PFAC*—Perfluoroalkyl Carboxylic Acid

3.2.20 *PFBA*—Perfluorobutanoate

3.2.21 *PFPeA*—Perfluoropentanoate

3.2.22 *PFHxA*—Perfluorohexanoate

3.2.23 *PFHpA*—Perfluoroheptanoate

3.2.24 *PFOA*—Perfluorooctanoate

3.2.25 *PFNA*—Perfluorononanoate

3.2.26 *PFDA*—Perfluorodecanoate

3.2.27 *PFUnA*—Perfluoroundecanoate

3.2.28 *PFTriA*—Perfluorotridecanoate

3.2.29 *PFTreA*—Perfluorotetradecanoate

3.2.30 *FTAs and FTUAs*—Fluorotelomer and Unsaturated Fluorotelomer Acids

3.2.31 *FHpPA—3*-perfluoropheptyl propanoic acid

3.2.32 *FOUEA—2H*-perfluoro-2-decenoic acid

3.2.33 *FDEA—2*-perfluorodecyl ethanoic acid

3.2.34 *FOEA—2*-perfluorooctyl ethanoic acid

3.2.35 *FHUEA—2H*-perfluoro-2-octenoic acid

3.2.36 *FHEA—2*-perfluorohexyl ethanoic acid

3.2.37 *MPFAS*—Isotopically labeled Perfluoroalkylsulfonates

3.2.38 *MPFHxS*—$^{18}O_2$-Perfluorohexylsulfonate

3.2.39 *MPFOS*—$^{13}C_4$-Perfluorooctylsulfonate

3.2.40 *MPFCA*—Isotopically labeled Perfluoroalkylcarboxylates

3.2.41 *MPFBA*—$^{13}C_4$-Perfluorobutanoate

3.2.42 *MPFHxA*—$^{13}C_2$-Perfluorohexanoate

3.2.43 *MPFOA*—$^{13}C_4$-Perfluorooctanoate

3.2.44 *MPFNA*—$^{13}C_5$-Perfluorononanoate

3.2.45 *MPFDA*—$^{13}C_2$-Perfluorodecanoate

3.2.46 *MPFUnA*—$^{13}C_2$-Perfluoroundecanoate

3.2.47 *MPFDoA*—$^{13}C_2$-Perfluorodecanoate

3.2.48 *QA*—Quality Assurance

3.2.49 *QC*—Quality Control

3.2.50 *RL*—Reporting Limit

3.2.51 *RLCS*—Reporting Limit Check Sample

3.2.52 *RSD*—Relative Standard Deviation

3.2.53 *RT*—Retention Time

3.2.54 *SRM*—Single Reaction Monitoring

3.2.55 *SS*—Surrogate Standard

3.2.56 *TC*—Target Compound

4. Summary of Test Method

4.1 The operating conditions presented in this test method have been successfully used in the determination of polyfluorinated compounds in soil; however, this test method is intended to be performance based and alternative operating

[6] Available from National Technical Information Service (NTIS), U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA, 22161, http://www.epa.gov/epawaste/hazard/testmethods/index.htm

TABLE 2 Concentrations of Calibration Standards (ng/L)

Analyte/Surrogate	LV1	LV2	LV3	LV4	LV5	LV6	LV7	LV8	LV9
PFPeA, PFBA	25	50	100	200	300	400	500	750	1000
PFTreA, PFTriA, PFDoA, PFUnA, PFDA, PFOS, PFNA, PFHxA, PFHpA, PFBS, PFechS, PFOA, PFHxS, FOUEA, FHUEA, FHpPA, MPFBS, MPFHxA, MPFUnA, MPFOA, MPFDA, MPFOS, MPFNA, MPFHxS, MPFBA	5	10	20	40	60	80	100	150	200
FHEA, FOEA, FDEA	100	200	400	800	1200	1600	2000	3000	4000

conditions can be used to perform this method provided data quality objectives are attained.

4.2 For PFAS analysis, samples are shipped to the lab on ice and analyzed within 28 days of collection. A sample (2 g) is transferred to a polypropylene tube, spiked with surrogates (all samples) and target PFAS (laboratory control and matrix spike samples). The analytes are tumbled for an hour with 10 mL of methanol:water (50:50) under basic condition (pH ~9 to 10 adjusted with ~20 µL ammonium hydroxide). The samples are centrifuged and the extract, leaving the solid behind, is filtered through a polypropylene filter unit. Acetic acid (~50 µL) is added to all the filtered samples to adjust the pH ~3 to 4 and then analyzed by LC/MS/MS.

4.3 Most of the PFAS target compounds are identified by comparing the single reaction monitoring (SRM) transition and its confirmatory SRM transition if correlated to the known standard SRM (Table 3) and quantitated utilizing an external calibration. The surrogates and some PFAS target analytes (PFPeA, PFBA, FOUEA, and FHUEA) only utilize one SRM transition due to a less sensitive or non-existent secondary

TABLE 3 Retention Times, SRM Ions, and Analyte-Specific Mass Spectrometer Parameters

Chemical	Primary/ Confirmatory	Retention Times (min)	Cone (V)	Collision (eV)	MRM Transition	Primary/ Confirmatory SRM Area Ratio
PFTreA	Primary	10.63	20	13	712.9→668.9	7.4
	Confirmatory		20	30	712.9→169	
PFTriA	Primary	10.17	25	12	662.9→618.9	7.4
	Confirmatory		25	28	662.9→169	
PFDoA	Primary	9.61	10	12	612.9→568.9	8.2
	Confirmatory		10	25	612.9→169	
PFUnA	Primary	9.05	15	10	562.9→519	7.2
	Confirmatory		15	18	562.9→269	
PFDA	Primary	8.45	20	10	512.9→468.9	6.5
	Confirmatory		20	16	512.9→219	
PFOS	Primary	8.78	10	42	498.9→80.1	1.3
	Confirmatory		10	40	498.9→99.1	
PFNA	Primary	7.78	20	10	462.9→418.9	4.9
	Confirmatory		20	16	462.9→219	
PFecHS	Primary	8.1	10	25	460.9→381	2.2
	Confirmatory		10	25	460.9→99.1	
PFOA	Primary	7.11	20	10	412.9→369	3.6
	Confirmatory		20	16	412.9→169	
PFHxS	Primary	7.39	15	32	398.9→80.1	1
	Confirmatory		15	32	398.9→99.1	
PFHpA	Primary	6.35	15	10	362.9→319	4.1
	Confirmatory		15	15	362.9→169	
PFHxA	Primary	5.54	15	8	312.9→269	24.1
	Confirmatory		15	18	312.9→119.1	
PFBS	Primary	5.66	10	30	298.9→80.1	1.6
	Confirmatory		10	25	298.9→99.1	
PFPeA	Primary	4.68	10	8	263→219	NA
PFBA	Primary	3.67	10	8	212.9→169	NA
FHEA	Primary	6.14	15	20	376.9→293	3.6
	Confirmatory		15	6	376.9→313	
FOEA	Primary	7.54	15	18	476.9→393	4.3
	Confirmatory		15	12	476.9→413	
FDEA	Primary	8.83	15	8	576.8→493	3.2
	Confirmatory		15	15	576.8→513	
FOUEA	Primary	7.54	20	12	456.9→392.9	NA
FHpPA	Primary	7.54	15	12	440.9→337	1.1
	Confirmatory		15	20	440.9→317	
FHUEA	Primary	6.08	10	12	357→293	NA
MPFBA	Primary	3.67	10	7	217→172.1	NA
MPFHxA	Primary	5.54	15	8	315→270	NA
MPFHxS	Primary	7.39	15	34	402.9→84.1	NA
MPFOA	Primary	7.11	15	10	417→372	NA
MPFNA	Primary	7.81	15	9	467.9→423	NA
MPFOS	Primary	8.78	15	40	502.9→80.1	NA
MPFDA	Primary	8.45	15	10	514.9→470	NA
MPFUnA	Primary	9.05	15	10	564.9→519.9	NA
MPFDoA	Primary	9.61	15	12	614.9→569.9	NA

SRM transition. As an additional quality control measure, isotopically labeled PFAS surrogate (listed in 12.4) recoveries are monitored. There is no correction to the data based upon surrogate recoveries. The final report issued for each sample lists the concentration of PFAS, if detected, or <RL, if not quantifiable, in ng/kg (dry weight basis) and the surrogate recoveries.

5. Significance and Use

5.1 This test method has been developed by the U.S. EPA Region 5 Chicago Regional Laboratory (CRL).

5.2 PFAS are widely used in various industrial and commercial products; they are persistent, bio-accumulative, and ubiquitous in the environment. PFAS have been reported to exhibit developmental toxicity, hepatotoxicity, immunotoxicity, and hormone disturbance. A draft Toxicological Profile for Perfluoroalkyls from the U.S. Department of Health and Human Services is available.[7] PFAS have been detected in soils, sludges, and surface and drinking waters. Hence, there is a need for a quick, easy, and robust method to determine these compounds at trace levels in various soil matrices for understanding of the sources and pathways of exposure.

5.3 This method has been used to determine selected PFAS in sand (Table 4) and four ASTM reference soils (Table 5).

6. Interferences

6.1 All glassware is washed in hot water with detergent and rinsed in hot water followed by distilled water. The glassware is then dried and heated in an oven at 250 °C for 15 to 30 min. All glassware is subsequently rinsed with methanol or acetonitrile.

6.2 All reagents and solvents should be pesticide residue purity or higher to minimize interference problems. The use of PFAS-containing caps must be avoided.

6.3 Matrix interferences may be caused by contaminants in the sample. The extent of matrix interferences can vary considerably depending on variations in the sample matrices.

6.4 Contaminants have been found in reagents, glassware, tubing, glass disposable pipettes, filters, degassers, and other apparatus that release polyfluorinated compounds. All of these materials and supplies are routinely demonstrated to be free

[7] A draft Toxicological Profile for Perfluroalkyls can be found at http://www.atsdr.cdc.gov/toxprofiles/tp.asp?id=1117&tid=237 (2014).

TABLE 4 Single-Laboratory Recovery Data in Ottawa Sand

Sample	Measured ng/kg from Ottawa Sand P&A Data (400 ng/kg spike for all PFAS except 2000 ng/kg for PFBA and PFPeA and 8000 ng/kg spike for FHEA, FDEA, and FOEA)										
	PFTreA	PFTriA	PFDoA	PFUnA	PFDA	PFNA	PFOA	PFHpA	PFHxA	PFPeA	PFBA
Unspiked 1	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL
Unspiked 2	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL
P&A 1	389.6	394.3	384.7	376.7	362.1	347.6	345.8	232.9	222.2	1614.9	1344.5
P&A 2	462.1	424.6	397.2	379.1	378.4	376.9	365.9	247.9	229.8	1710.1	1388
P&A 3	402.7	387.7	383.1	365.9	374.7	363.3	347.1	242.4	222.9	1658.9	1376
P&A 4	403.9	397.1	395.4	381.5	379	359.4	342.7	246.8	225.8	1693.6	1401.9
P&A 5	467.2	445.8	412.6	388.5	376.8	370.3	369.7	249.3	231.4	1716.5	1433.4
P&A 6	392.1	385.3	374.2	370.9	353.2	351.7	340.3	236.7	220.5	1659	1366.4
Mean Recovery (ng/kg)	419.6	405.8	391.2	377.1	370.7	361.5	351.9	242.7	225.4	1675.5	1385
% Mean Recovery	104.9	101.4	97.8	94.3	92.7	90.4	88	60.7	56.4	83.8	69.3
Standard Deviation	35.4	24.1	13.5	8	10.6	11.1	12.6	6.6	4.4	38.5	30.7
RSD (%)	8.4	5.9	3.5	2.1	2.9	3.1	3.6	11	1.9	2.3	2.2

Sample	PFBS	PFHxS	PFOS	PFechS	FOUEA	FHpPA	FHUEA	FHEA	FOEA	FDEA
Unspiked 1	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL
Unspiked 2	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL	<RL
P&A 1	337.4	349.1	340.3	342.8	389.5	371.3	372.5	7023.5	8202.6	8564.9
P&A 2	347.3	358.3	345.9	347.2	408.7	377.2	387.1	7346.1	8542.6	9308
P&A 3	366.3	330.1	331.7	345.4	401.5	361.4	379	6844.3	7402.4	8989.2
P&A 4	348.2	343.6	338.3	347.6	404.9	377.5	388.1	7258.2	7551.9	9173.4
P&A 5	351.8	361.7	365.6	362.6	417.5	395.1	391.8	7461.3	7821.2	9287.4
P&A 6	336.7	343.4	363.7	342.5	394.5	356.9	374.5	7559.3	8002.2	8367.1
Mean Recovery (ng/kg)	347.9	347.7	347.7	348	402.7	373.2	382.1	7248.8	7920.5	8948.3
% Mean Recovery	87	86.9	86.9	87	100.7	93.3	95.5	90.6	99	111.9
Standard Deviation	10.9	11.5	13.9	7.4	10	13.6	7.9	270.4	421.3	395.3
RSD (%)	3.1	3.3	4	2.1	2.5	3.6	2.1	3.7	5.3	4.4

TABLE 5 Single-Laboratory Surrogate Recovery Data in Ottawa Sand

Sample	Measured ng/kg from Ottawa Sand – 400 ng/kg spike								
	MPFBA	MPFHxA	MPFHxS	MPFOA	MPFNA	MPFOS	MPFDA	MPFUnA	MPFDoA
Unspiked 1	420.0	433.5	431.8	428.0	439.4	429.2	442.6	443.3	447.7
Unspiked 2	366.5	396.8	378.5	384.9	389.8	373.6	404.9	400.8	425.8
P&A 1	361.1	364.3	356.3	377.0	376.6	354.4	384.9	391.3	409.3
P&A 2	383.6	378.4	357.3	389.4	379.7	375.7	395.7	399.2	412.2
P&A 3	374.5	378.5	375.4	390.5	378.6	372.4	382.5	386.9	402.2
P&A 4	370.1	384.4	366.1	396.3	384.4	374.2	397.8	406.2	420.5
P&A 5	370.1	386.8	372.0	395.7	381.1	372.8	394.4	399.9	421.5
P&A 6	363.6	384.8	356.1	397.9	384.9	368.6	389.5	392.3	402.9
Mean Recovery (ng/kg dry weight)	376.2	388.4	374.2	394.9	389.3	377.6	399.0	402.5	417.7
% Mean Recovery	94.0	97.1	93.5	98.7	97.3	94.4	99.8	100.6	104.4
Standard Deviation	19.0	20.4	24.9	15.0	20.7	21.9	19.0	17.6	14.9
RSD (%)	5.1	5.3	6.7	3.8	5.3	5.8	4.8	4.4	3.6

from interferences by analyzing laboratory reagent blanks under the same conditions as the samples. If found, measures should be taken to remove the contamination or data should be qualified; background subtraction of blank contamination is not allowed.

6.5 The liquid chromatography system used should consist, as much as practical, of sample solution or eluent contacting components free of PFAS target analytes of interest.

6.6 Polyethylene LC vial caps or any other target analyte-free vial caps should be used.

6.7 Polyethylene disposable pipettes or target analyte-free pipettes should be used. All disposable pipettes should be checked for release of target analytes of interest.

6.8 Degassers are important to continuous LC operation and most commonly are made of fluorinated polymers. To enable use, an isolator column should be placed after the degasser and prior to the sample injection valve to separate the PFAS in the sample from the PFAS in the LC system.

7. Apparatus

7.1 *LC/MS/MS System:*

7.1.1 *Liquid Chromatography System*—A complete LC system is required in order to analyze samples; this should include a sample injection system, a solvent pumping system capable of mixing solvents, a sample compartment capable of maintaining required temperature, and a temperature-controlled column compartment. An LC system that is capable of performing at the flows, pressures, controlled temperatures, sample volumes, and requirements of the standard must be used.

7.1.2 *Analytical Column*[8]—A reverse phase Charged Surface Hybrid Phenyl-Hexyl particle column was used to develop this test method. Any column that achieves adequate resolution may be used. The retention times and order of elution may change depending on the column used and need to be monitored.

7.1.3 *Isolator Column*—A reverse phase C18 column was used in this test method to separate the target analytes in the LC system and solvents from the target analytes in the analytical sample. This column was placed between the solvent mixing chamber and the injector sample loop.

7.2 *Tandem Mass Spectrometer System*—An MS/MS system capable of multiple reaction monitoring (MRM) analysis or any system that is capable of meeting the requirements in this test method must be used.

7.3 *Centrifuge*—A device to centrifuge the samples.

7.4 *Lab Rotator*[9]—A device to mix the samples by end-over-end rotation.

7.5 *Filtration Device:*

7.5.1 *Hypodermic Syringe*—A luer-lock tip glass syringe capable of holding a syringe-driven filter unit.

7.5.2 A 10 mL lock tip glass syringe size is recommended since a 10 mL sample size is used in this test method.

7.5.3 *Filter Unit*[10]—Polypropylene filter units were used to filter the samples.

8. Reagents and Materials

8.1 *Purity of Reagents*—High performance liquid chromatography (HPLC) pesticide residue analysis and spectrophotometry grade chemicals must be used in all tests. Unless indicated otherwise, it is intended that all reagents must conform to the Committee on Analytical Reagents of the American Chemical Society.[11] Other reagent grades may be

[8] A Waters Acquity UPLC CSH Phenyl-Hexyl, 2.1 × 100 mm and 1.7 µm particle size column, or equivalent, has been found suitable for use. It was used to develop this test method and generate the precision and bias data presented in Section 16. If you are aware of an alternative column that meets the performance of the standard, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at the meeting responsible technical committee,[1] which you may attend.

[9] A lab rotator, or equivalent, has been found suitable to mix samples.

[10] A 0.2 µm polypropylene membrane syringe-driven filter unit, or equivalent, has been found suitable for use.

[11] Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, EDH Ltd., Poole, Dorset, U.K. and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

used provided they are first determined to be of sufficiently high purity to permit their use without affecting the accuracy of the measurements.

8.2 *Purity of Water*—Unless otherwise indicated, references to water must be understood to mean reagent water conforming to Type I of Specification D1193. It must be demonstrated that this water does not contain contaminants at concentrations sufficient to interfere with the analysis.

8.3 *Gases*—Ultrapure nitrogen and argon.

8.4 *Vials*—2 mL amber glass or polypropylene autosampler vials or equivalent.

8.5 Polyethylene or any PFAS-free applicable autosampler vial caps.

8.6 *Syringe*—10 or 25 mL filter-adaptable glass syringe with luer lock.

8.7 pH paper (pH range 1 to 14).

8.8 *Polypropylene Tubes*—15 and 50 mL.

8.9 Class A volumetric glassware.

8.10 *Pipette Tips*—Polypropylene pipette tips free of release agents or low retention coating of various sizes.

8.11 Polyethylene disposable pipettes.

8.12 Acetonitrile (CAS No. 75-05-8).

8.13 Methanol (CAS No. 67-56-1).

8.14 Ammonium acetate (CAS No. 631-61-8).

8.15 Acetic acid (CAS No. 64-19-7).

8.16 2-Propanol (isopropyl alcohol, CAS No. 67-63-0).

8.17 Ammonium hydroxide (CAS No. 1336-21-6).

8.18 Ottawa sand (CAS No. 14808-60-7).

8.19 *PFAS Standards:*[12]

8.19.1 Perfluorobutylsulfonate (PFBS, CAS No. 29420-49-3).

8.19.2 Perfluorohexylsulfonate (PFHxS, CAS No. 3871-99-6).

8.19.3 Perfluorooctylsulfonate (PFOS, CAS No. 1763-23-1).

8.19.4 Perfluorobutanoate (PFBA, CAS No. 375-22-4).

8.19.5 Perfluoropentanoate (PFPeA, CAS No. 2706-90-3).

8.19.6 Perfluorohexanoate (PFHxA, CAS No. 307-24-4).

8.19.7 Perfluoroheptanoate (PFHpA, CAS No. 375-85-9).

8.19.8 Perfluorooctanoate (PFOA, CAS No. 335-67-1).

8.19.9 Perfluorononanoate (PFNA, CAS No. 375-95-1)

8.19.10 Perfluorodecanoate (PFDA, CAS No. 335-76-2).

8.19.11 Perfluoroundecanoate (PFUnA, CAS No. 2058-94-8).

8.19.12 Perfluorododecanoate (PFDoA, CAS No. 307-55-1).

8.19.13 Perfluorotridecanoate (PFTriA, CAS No. 72629-94-8).

8.19.14 Perfluorotetradecanoate (PFTreA, CAS No. 376-06-7).

8.19.15 Decafluoro-4-(pentafluoroethyl)cyclohexanesulfonate (PFechS, CAS No. 67584-42-3).

8.19.16 3-perfluoropheptyl propanoic acid (FHpPA, CAS No. 812-70-4).

8.19.17 2H-perfluoro-2-decenoic acid (FOUEA, CAS No. 70887-84-2).

8.19.18 2-perfluorodecyl ethanoic acid (FDEA, CAS number not available).

8.19.19 2-perfluorooctyl ethanoic acid (FOEA, CAS No. 27854-31-5).

8.19.20 2H-perfluoro-2-octenoic acid (FHUEA, CAS number not available).

8.19.21 2-perfluorohexyl ethanoic acid (FHEA, CAS No. 53826-12-3).

8.20 *PFAS Surrogates:*[13]

8.20.1 $^{18}O_2$-Perfluorohexylsulfonate (MPFHxS).

8.20.2 $^{13}C_4$-Perfluorooctylsulfonate (MPFOS).

8.20.3 $^{13}C_4$-Perfluorobutanoate (MPFBA).

8.20.4 $^{13}C_2$-Perfluorohexanoate (MPFHxA).

8.20.5 $^{13}C_4$-Perfluorooctanoate (MPFOA).

8.20.6 $^{13}C_5$-Perfluorononanoate (MPFNA).

8.20.7 $^{13}C_2$-Perfluorodecanoate (MPFDA).

8.20.8 $^{13}C_2$-Perfluoroundecanoate (MPFUnA).

8.20.9 $^{13}C_2$-Perfluorododecanoate (MPFDoA).

9. Hazards

9.1 Normal laboratory safety applies to this method. Analysts should wear safety glasses, gloves, and lab coats when working in the lab. Analysts should review the Safety Data Sheets (SDS) for all reagents used in this method.

10. Sampling

10.1 *Sampling and Preservation*—Grab samples are collected in glass or polypropylene containers. Sample containers and contact surfaces with PFAS must be avoided. As part of the overall quality assurance program for this test method, field blanks exposed to the same field conditions as samples are collected and analyzed according to this test method to assess the potential for field contamination. This test method is based on a 2 g sample size per analysis. If different sample sizes are used, spiking solution amounts may need to be modified. Conventional sampling practices should be followed with the caution that PFAS-containing products may be present in sampling equipment. All sampling equipment and supplies must be PFAS free in order to prevent contamination of the samples. EPA publication SW-846 may be used as a sampling guide. Samples must be shipped on ice with a trip blank. Once received, the sample temperature is taken and must be between freezing and 6 °C. If the receiving temperature is greater than 6 °C, the sample temperature is noted in the case narrative accompanying the data. Samples should be stored refrigerated between 0 and 6 °C from the time of collection until analysis.

[12] PFAS standards may be difficult to find; some sources of PFAS standards that have been found suitable for use were from Aldrich Chemical Company, Accustandard, Wellington Laboratories, Inc., and Wako Laboratory. Standards from other vendors may be used.

[13] PFAS surrogates from Wellington Laboratories Inc., or equivalent, have been found suitable for use.

The sample should be analyzed within 28 days of collection. No holding time study has been done on the various soil matrices tested in this test method. Holding time may vary depending on the matrix, and individual laboratories should determine the holding time in their matrix.[14]

11. Preparation of LC/MS/MS

11.1 *LC Chromatograph Operating Conditions:*

11.1.1 Injections of all standards and samples are made at a 30 µL volume. Other injection volumes may be used to optimize conditions. Standards and sample extracts must be in a 50:50 methanol:water solution containing 0.1 % acetic acid. In the case of extreme concentration differences amongst samples, it is wise to analyze a blank after a concentrated sample and before a dilute sample to minimize carryover of analytes from injection to injection. However, there should not be carryover between samples. The LC utilized to develop this test method has a flow-through LC needle design. The gradient conditions for liquid chromatography are shown in Table 6.

11.2 *LC Sample Manager Conditions:*

11.2.1 *Needle Wash Solvent*—60 % acetonitrile/40 % 2-propanol; Time: 5 min.

11.3 *Mass Spectrometer Parameters:*

11.3.1 To acquire the maximum number of data points per SRM channel while maintaining adequate sensitivity, the tune parameters may be optimized according to the instrument used. Each peak requires at least ten scans per peak for adequate quantitation. This test method contains nine surrogates, which are isotopically labeled PFAS, and 21 PFAS which are split up into 18 MRM acquisition functions to optimize sensitivity. Variable parameters regarding retention times, SRM transitions, and cone and collision energies are shown in Table 3. Mass spectrometer parameters used in the development of this method are listed below:

11.3.1.1 The instrument is set in the Electrospray negative source setting.

11.3.1.2 Capillary Voltage: 0.75 kV.

11.3.1.3 Cone: Variable depending on analyte.

11.3.1.4 Extractor: 2 Volts.

11.3.1.5 Source Temperature: 150 °C.

11.3.1.6 Desolvation Gas Temperature: 450 °C.

11.3.1.7 Desolvation Gas Flow: 800 L/h.

11.3.1.8 Cone Gas Flow: 200 L/h.

11.3.1.9 Collision Gas Flow: 0.15 mL/min.

11.3.1.10 Low Mass Resolution 1: 2.6.

11.3.1.11 High Mass Resolution 1: 14.

11.3.1.12 Ion Energy 1: 1.

11.3.1.13 Entrance Energy: 1.

11.3.1.14 Collision Energy: Variable depending on analyte.

11.3.1.15 Exit Energy: 1.

11.3.1.16 Low Mass Resolution 2: 2.5.

11.3.1.17 High Mass Resolution 2: 14.

11.3.1.18 Ion Energy 2: 3.

11.3.1.19 Gain: 1.0.

11.3.1.20 Multiplier: 511.1.

11.3.1.21 Inter-Scan Delay: 0.004 s.

[14] A guide to help and determine sample holding times can be found at http://www.epa.gov/esd/cmb/research/bs_033cmb06.pdf (2014).

TABLE 6 Gradient Conditions for Liquid Chromatography

Time (min)	Flow (mL/min)	Percent 95 % Water: 5 % Acetonitrile	Percent Acetonitrile	Percent 95 % Water: 5 % Acetonitrile, 400 mM Ammonium Acetate
0	0.3	95	0	5
1	0.3	75	20	5
6	0.3	50	45	5
13	0.3	15	80	5
14	0.4	0	95	5
17	0.4	0	95	5
18	0.4	95	0	5
21	0.4	95	0	5

12. Calibration and Standardization

12.1 The mass spectrometer must be calibrated as per manufacturer's specifications before analysis. Analytical values satisfying test method criteria have been achieved using the following procedures. Prepare all solutions in the lab using Class A volumetric glassware.

12.2 *Calibration and Standardization*—To calibrate the instrument, analyze nine calibration standards of the polyfluorinated compounds prior to sample analysis as shown in Table 2. Calibration stock standard solution is prepared from the target and surrogate spike solutions directly to ensure consistency. Stock standard Solution A containing the polyfluorinated compounds and surrogates is prepared at Level 9 concentration and aliquots of that solution are diluted to prepare Levels 1 through 8. The following steps will produce standards with the concentration values shown in Table 2. The analyst is responsible for recording initial component weights carefully when working with pure materials and correctly carrying the weights through the dilution calculations. At a minimum five calibration levels are required when using a linear calibration curve and six calibration levels are required when using a quadratic calibration curve. An initial nine-point curve may be used to allow for the dropping of the lower level calibration points if the individual laboratory's instrument cannot achieve low detection limits on certain PFAS. This should allow for at least a five or six-point calibration curve to be obtained. No problems were encountered while using the nine-point calibration curve in developing this test method.

12.2.1 Calibration stock standard Solution A (Level 9, Table 2) is prepared from the target and surrogate spike solutions directly to ensure consistency. 500 µL of surrogate spike (20 µg⁄L), 500 µL Target Spike I, and 500 µL of PFAS Target Spike II (refer to Table 7) is added to a 50 mL volumetric flask and diluted to 50 mL with 50:50 methanol:water containing 0.1 % acetic acid. The preparation of the Level 9 standard can be accomplished using appropriate volumes and concentrations of stock solutions as per a particular laboratory's standard procedure. It is critical to ensure that analytes are solubilized in the Level 9 standard.

12.2.2 Aliquots of Solution A are then diluted with 50:50 methanol:water containing 0.1 % acetic acid to prepare the

TABLE 7 PFAS Target Spike Solutions (PPB)

Analyte	Concentration of Analyte in PFAS Target Spike Solutions		
	PFAS High Target Spike Solutions		PFAS Reporting Limit Spike Solution
	Target Spike I	Target Spike II	
PFTreA, PFTriA, PFDoA, PFUnA, PFDA, PFOS, PFNA, PFHxA, PFHpA, PFBS, PFechS, PFOA, PFHxS	20 µg/L	—	2 µg/L
PFBA, PFPeA	100 µg/L	—	10 µg/L
FOUEA, FHUEA, FHpPA	—	20 µg/L	2 µg/L
FHEA, FOEA, FDEA	—	400 µg/L	40 µg/L

desired calibration levels in 2 mL LC vials. The calibration vials must be used within 24 h to ensure optimum results. The end calibration check must be prepared in a separate LC vial near the mid-level. All calibration standards should only be used once. The analyte concentration in the vial may change after the vial cap is pierced because the vial caps may not reseal after puncture; if the cap reseals it may be used over again. Changing the caps immediately after the injection should alleviate this problem. Calibration standards are not filtered.

12.2.3 Inject each standard and obtain its chromatogram. An external calibration technique is used to monitor the primary and confirmatory SRM transitions of each analyte. Calibration software is utilized to conduct the quantitation of the target analytes and surrogates using the primary SRM transition. The ratios of the primary/confirmatory SRM transition area counts are given in Table 3 and will vary depending on the individual tuning conditions. The primary/confirmatory SRM transition area ratio must be within 35 % of the individual lab's accepted primary/confirmatory SRM transition area ratio. The primary SRM transition of each analyte is used for quantitation of and the confirmatory SRM transition for confirmation. This gives added confirmation by isolating the parent ion, forming two product ions via fragmentation, and relating it to the retention time in the calibration standard.

12.2.4 Depending on sensitivity and matrix interference issues dependent on sample type, the confirmatory SRM transition can be used as the primary SRM transition for quantitation during analysis. This must be explained in a narrative accompanying the data. A new primary/confirmatory ion ratio will then be determined if switching the SRM transitions used to quantitate and confirm. The new primary/confirmatory SRM transition area ratio is required to be within 35 % of the individual lab's new primary/confirmatory SRM transition area ratio.

12.2.5 The calibration software manual should be consulted to use the software correctly. The quantitation method is set as an external calibration using the peak areas in ppt units. Concentrations may be calculated using the data system software to generate linear regression or quadratic calibration curves. Forcing the calibration curve through the origin (X = 0, Y = 0) is not recommended. Curves should be evaluated using relative error or relative standard error.[15]

12.2.6 Linear calibration may be used if the point of origin is excluded and a fit weighting 1/X is used in order to give more emphasis to the lower concentrations. Each calibration point used to generate the curve must have a calculated percent deviation less than 25 % from the generated curve.

12.2.7 Quadratic calibration may be used if the point of origin is excluded, and a fit weighting of 1/X is used in order to give more emphasis to the lower concentrations. Each calibration point used to generate the curve must have a calculated percent deviation less than 25 % from the generated curve.

12.2.8 The retention time window of the SRM transitions must be within 5 % of the retention time of the analyte in a midpoint calibration standard. If this is not the case, re-analyze the calibration curve to determine if there was a shift in retention time during the analysis and the sample needs to be re-injected. If the retention time is still incorrect in the sample, refer to the analyte as an unknown.

12.2.9 A midpoint calibration check standard must be analyzed at the end of each batch of 30 samples or within 24 h after the initial calibration curve was generated; the criteria in the individual lab's quality system may be more restrictive pertaining to the number of samples. This end calibration check, a new not pierced sealed vial, should come from the same calibration standard solution that was used to generate the initial curve. The results from the end calibration check standard must have a percent deviation less than 30 % from the calculated concentration for the target analytes and surrogates. If the results are not within these criteria, corrective action including reoccurrence minimization is performed and either all samples in the batch are re-analyzed against a new calibration curve or the affected results are qualified with an indication that they do not fall within the performance criteria of the test method. If the analyst inspects the vial containing the end calibration check standard and notices that the sample evaporated affecting the concentration or other anomaly, a new end calibration check standard may be made and analyzed. If this new end calibration check standard has a percent deviation less than 30 % from the calculated concentration for the target analytes and surrogates, the results may be reported unqualified.

12.3 If a laboratory has not performed the test before or if there has been a major change in the measurement system, for example, new analyst, new instrument, etc., an instrument qualification study including method detection limit (MDL),

[15] Management and Technical Requirements for Laboratories Performing Environmental Analysis, Module 4: Quality Systems for Chemical Testing; The NELAC Institute, 2017.

calibration range determination, and precision and bias determination must be performed to demonstrate laboratory capability.

12.3.1 Analyze at least four replicates of a spiked sand sample containing the PFAS and surrogates at an extract concentration in the calibration range of Levels 4 through 7. The Level 6 concentration of the nine-point calibration curve was used to set the QC acceptance criteria in this method. The matrix and chemistry should be similar to the matrix used in this test method. Each replicate must be taken through the complete analytical test method including any sample manipulation and extraction steps.

12.3.2 Calculate the mean (average) percent recovery and relative standard deviation (RSD) of the four values and compare to the acceptable ranges of the QC acceptance criteria for the Initial Demonstration of Performance in Table 8.

12.3.3 This study should be repeated until the single-operator precision and mean recovery are within the limits in Table 8. If a concentration other than the recommended concentration is used, refer to Practice D5847 for information on applying the *F* test and *t* test in evaluating the acceptability of the mean and standard deviation.

12.3.3.1 The QC acceptance criteria for the Initial Demonstration of Performance in Table 8 were generated from the single-laboratory data shown in Section 16. Data from Ottawa sand and four ASTM soil matrices are shown in Section 16. It is recommended that each laboratory determine in-house QC acceptance criteria which meet or exceed the criteria in this test method. References generating QC acceptance criteria are ASTM Practices D2777, D5847, E2554, or Method 8000 in EPA publication SW-846.

12.4 *Surrogate Spiking Solution:*

12.4.1 A surrogate spiking solution containing nine isotopically labeled PFAS—MPFBA, MPFHxA, MPFHxS, MPFDA, MPFOA, MPFOS, MPFNA, MPFUnA, and MPFDoA—is added to all samples, method blanks, duplicates, laboratory control samples, matrix spikes, and reporting limit checks. A stock surrogate spiking solution is prepared at 20 µg/L in 95 % acetonitrile: 5 % water. Spiking 40 µL of this spiking solution into a 2 g soil sample results in a concentration of 400 ng/kg of the surrogate in the sample. The results obtained for the surrogate recoveries must fall within the limits of Table 8. If the limits are not met, the affected results must be qualified with an indication that they do not fall within the performance criteria of the test method.

12.4.1.1 The surrogate spiking solution was prepared by adding 500 µL of a 2 mg/L PFAS surrogate mix[16] in a 50 mL volumetric and diluted to 50 mL with 95 % acetonitrile: 5 % water. Surrogate spiking solutions are routinely replaced every year if not previously discarded for quality control failure.

12.5 *Method Blank:*

[16] Surrogate mix from Wellington Laboratories, Inc. has been found suitable for use.

TABLE 8 QC Acceptance Criteria

NOTE 1—Table 8 data is preliminary until a multi-lab validation study is completed.

Analyte/Surrogate	Spike Conc. ng/kg	Initial Demonstration of Performance			Laboratory Control Sample	
		Recovery (%)		Precision	Recovery (%)	
		Lower Limit	Upper Limit	Maximum % RSD	Lower Control Limit (LCL) %	Upper Control Limit (UCL) %
PFTreA	400	70	130	30	70	130
PFTriA	400	70	130	30	70	130
PFDoA	400	70	130	30	70	130
PFUnA	400	70	130	30	70	130
PFDA	400	70	130	30	70	130
PFOS	400	70	130	30	70	130
PFNA	400	70	130	30	70	130
PFecHS	400	70	130	30	70	130
PFOA	400	70	130	30	70	130
PFHxS	400	70	130	30	70	130
PFHpA	400	50	130	30	50	130
PFHxA	400	50	130	30	50	130
PFBS	400	70	130	30	70	130
PFPeA	2000	70	130	30	70	130
PFBA	2000	50	130	30	50	130
FHEA	8000	70	130	30	70	130
FOEA	8000	70	130	30	70	130
FDEA	8000	70	130	30	70	130
FOUEA	400	70	130	30	70	130
FHpPA	400	70	130	30	70	130
FHUEA	400	70	130	30	70	130
MPFBA	400	70	130	30	70	130
MPFHxA	400	70	130	30	70	130
MPFHxS	400	70	130	30	70	130
MPFOA	400	70	130	30	70	130
MPFNA	400	70	130	30	70	130
MPFOS	400	70	130	30	70	130
MPFDA	400	70	130	30	70	130
MPFUnA	400	70	130	30	70	130
MPFDoA	400	70	130	30	70	130

12.5.1 At least two method blanks for every 30 samples are prepared in 2 g of Ottawa sand to investigate for contamination during sample preparation and extraction. The concentration of target analytes in either/both blank(s) must be at less than half the reporting limit or the data must be qualified as having a blank issue and the reporting limit must be raised to at least three times above the blank contamination concentration. PFAS are common in the environment and laboratories requiring an additional method blank sample.

12.6 *Reporting Limit Check Sample (RLCS):*

12.6.1 Each batch or within the 24 h analysis window a reporting limit check sample must be analyzed. The reporting limit check sample is processed like a laboratory control sample just spiked at or near (one to two times) the reporting limit. The concentration of the RLCS may be reported below the reporting limit since the spike is at or near the reporting limit. This sample is to check if the analytes were present at the reporting limit, they would be identified. The recovery limits for the RLCS are 35 to 150 %, if any analytes are outside of these limits the QC failure is explained in a narrative accompanying the data.

12.6.2 Two grams of Ottawa sand is added to a 15 mL polypropylene centrifuge tube. The sample is spiked with 40 µL of PFAS surrogate spiking solution and 25 µL of PFAS reporting limit check solution (Table 7) and then taken through the sample preparation and analyzed.

12.7 *Laboratory Control Sample (LCS):*

12.7.1 Analyze at least one LCS with the PFAS at a mid-level extract concentration. A concentration at the Level 6 concentration was used in this test method; any mid-level (Levels 4 through 7) concentration may be chosen using this test method. The LCS is prepared following the analytical method and analyzed with each batch of 30 samples or less. Prepare a stock matrix spiking solution—Target Spike I and II in 95 % acetonitrile: 5 % water containing the 21 PFAS at concentrations listed in Table 7. Spike 40 µL each of Target Spike I and Target Spike II into 2 g of Ottawa sand to yield a concentration of 2000 ng/kg (PFBA and PFPeA), 8000 ng/kg (FHEA, FDEA, and FOEA), and 400 ng/kg of remaining 16 PFAS (PFTreA, PFTriA, PFDoA, PFUnA, PFDA, PFOS, PFNA, PFHxA, PFHpA, PFBS, PFechS, PFOA, PFHxS, FOUEA, FHUEA, and FHpPA) in the sample. The result obtained for the LCS must fall within the limits in Table 8. Spiking solutions are routinely replaced every year if not previously discarded for quality control failure.

12.7.2 If the result is not within these limits, sample analysis is halted until corrective action resolving the problem has been performed. Impacted samples in the batch are either re-analyzed or the results are flagged with a qualifier stating that they do not fall within the performance criteria of the test method.

12.8 *Matrix Spike (MS):*

12.8.1 To check for interferences in the specific matrix being tested, perform an MS on at least one sample from each batch of 30 or fewer samples by spiking the sample with a known concentration of PFAS and following the analytical method. Prepare a stock matrix spiking solution—Target Spike I and II in 95 % acetonitrile: 5 % water containing the 21 PFAS at concentrations listed in Table 7. Spike 40 µL of these stock solutions into 2 g of the site sample to yield a concentration of 2000 ng/kg (PFBA and PFPeA), 8000 ng/kg (FHEA, FDEA, and FOEA), and 400 ng/kg of remaining 16 PFAS (PFTreA, PFTriA, PFDoA, PFUnA, PFDA, PFOS, PFNA, PFHxA, PFHpA, PFBS, PFechS, PFOA, PFHxS, FOUEA, FHUEA, AND FHpPA) in the sample.

12.8.2 If the spiked concentration plus the background concentration exceeds that of the Level 9 calibration standard, the sample must be diluted (using 50 % Methanol/50 % Water with 0.1 % acetic acid) to a level near the midpoint of the calibration curve.

12.8.3 Calculate the percent recovery of the spike (P) using Eq 1:

$$P = 100\frac{|A(V_s + V) - BV_s|}{CV} \quad (1)$$

where:

A = concentration found in spiked sample,
B = concentration found in unspiked sample,
C = concentration of analyte in spiking solution,
V_s = volume of sample used,
V = volume of spiking solution added, and
P = percent recovery.

12.8.4 The percent recovery of the spike must fall within the limits in Table 9. If the percent recovery is not within these limits, a matrix interference may be present. Under these

TABLE 9 MS/MSD QC Acceptance Criteria

NOTE 1—Table 9 data is preliminary until a multi-lab validation study is completed.

Analyte	Spike Conc. ng/L	MS/MSD Recovery (%)		Precision RPD (%)
		Lower Limit	Upper Limit	
PFTreA	400	70	130	30
PFTriA	400	70	130	30
PFDoA	400	70	130	30
PFUnA	400	70	130	30
PFDA	400	70	130	30
PFOS	400	70	130	30
PFNA	400	70	130	30
PFecHS	400	70	130	30
PFOA	400	70	130	30
PFHxS	400	70	130	30
PFHpA	400	50	130	30
PFHxA	400	50	130	30
PFBS	400	70	130	30
PFPeA	2000	70	130	30
PFBA	2000	50	130	30
FHEA	8000	70	130	30
FOEA	8000	70	130	30
FDEA	8000	70	130	30
FOUEA	400	70	130	30
FHpPA	400	70	130	30
FHUEA	400	70	130	30
MPFBA	400	70	130	30
MPFHxA	400	70	130	30
MPFHxS	400	70	130	30
MPFOA	400	70	130	30
MPFNA	400	70	130	30
MPFOS	400	70	130	30
MPFDA	400	70	130	30
MPFUnA	400	70	130	30
MPFDoA	400	70	130	30