FINAL REPORT

Single-Laboratory Validation Study of PFAS by Isotope Dilution LC-MS/MS

JANUARY 2022

SERDP
DOD ▪ EPA ▪ DOE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
26/01/2022	Technical Report	

4. TITLE AND SUBTITLE

Single-Laboratory Validation Study of PFAS by Isotope Dilution LC-MS/MS

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

Janice Willey: Naval Sea Systems Command, U.S. Navy
Richard Anderson: Air Force Civil Engineering Center
Adrian Hanley: U.S. Environmental Protection Agency, Office of Water.
Marc Mills: U.S. Environmental Protection Agency, Office of Research and Development
Coreen Hamilton: SGS AXYS Analytical Services, Ltd.
Tim Thompson: Science and Engineering for the Environment, LLC
Andrea Leeson: SERDP & ESTCP

5d. PROJECT NUMBER
ER19-1409

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Strategic Environmental Research and Development Program
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-3605

8. PERFORMING ORGANIZATION REPORT NUMBER
ER19-1409

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-3605

10. SPONSOR/MONITOR'S ACRONYM(S)
SERDP

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
ER19-1409

12. DISTRIBUTION/AVAILABILITY STATEMENT

DISTRIBUTION STATEMENT A. Approved for public release: distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

This report presents the findings of a single-laboratory validation study (SLVS) of an analytical method for the preparation and analysis of per- and polyfluoroalkyl substances (PFAS) in environmental matrices. Conducted as a joint effort by the U.S. Department of Defense (DoD) and the Environmental Protection Agency (EPA), the study was designed to provide data on the accuracy and precision of the method in aqueous matrices (wastewater, surface waters, groundwaters, landfill leachate), solids (soil, sediment, biosolids), and fish and clam tissues. The laboratory standard operating procedure (SOP) used and the results of the study were the basis of the EPA's draft Office of Water Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS (EPA Method 1633).

15. SUBJECT TERMS

Single-Laboratory Validation Study, PFAS, Per- and Polyfluoroalkyl Substances, Isotope Dilution LC-MS/MS

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			Andrea Leeson
UNCLASS	UNCLASS	UNCLASS	UNCLASS	571	**19b. TELEPHONE NUMBER** *(Include area code)* 571-372-6565

Author Team

Principal Authors

Willey, Janice	Naval Sea Systems Command, U.S. Navy
Anderson, Richard	Air Force Civil Engineering Center
Hanley, Adrian	U.S. Environmental Protection Agency, Office of Water.
Mills, Marc	U.S. Environmental Protection Agency, Office of Research and Development
Hamilton, Coreen	SGS AXYS Analytical Services, Ltd.
Thompson, Tim	Science and Engineering for the Environment, LLC
Leeson, Andrea	SERDP & ESTCP

Other Contributing Authors

Chapter 8 – Holding Time Study

Burket, S. Bekah	U.S. Environmental Protection Agency, Office of Water: Office of Science and Technology
Strock, Troy	U.S. Environmental Protection Agency, Office of Land and Emergency Management
Alpizar, Mirna	General Dynamics Information Technology
McCarty, Harry	General Dynamics Information Technology

PFAS Single-Laboratory Validation Study Data Compilation Report

Buytendyk, Allyson	Institute for Defense Analyses
Runkel, Sara	Institute for Defense Analyses
Cazares, Shelly	Institute for Defense Analyses

This report should be cited as

Willey, J., R. Anderson, A. Hanley, M. Mills, C. Hamilton, T. Thompson, and A. Leeson. 2021. Report on the Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS. Strategic Environmental Research and Development Program (SERDP) Project ER19-1409.

Acknowledgments

This report was prepared under contract to the Department of Defense (DoD) Strategic Environmental Research and Development Program (SERDP). The helpful guidance and contributions by the Program Directors, Dr. Andrea Leeson and Dr. Herb Nelson, are gratefully acknowledged.

This study has been a joint effort between the DoD at SERDP and the U.S. Environmental Protection Agency (EPA). The assistance of Brian D'Amico, EPA Office of Water, Office of Science and Technology, Engineering and Analysis Division, and Robert Wood, EPA Office of Water, Office of Science and Technology, Engineering and Analysis Division, is also acknowledged.

This report was a team effort. Special thanks go to the following individuals

U.S. Army Corps of Engineers
Melinda McClellan, Huntsville Environmental and Munitions Center of Expertise
Mike Malone, Huntsville Engineering and Support Center
Charles Coyle (retired)

SGS AXYS Analytical Services Ltd
Ivona Zysk
Ting Chen
Henry Huang

HydroGeoLogic, Inc
Joe Skibinski
John Powell

Noblis, Inc.
Cara Patton

BMT Designers and Planners Inc
Mathew Lloyd

Technical Editing
Phyllis Tower

Abstract

This report presents the findings of a single-laboratory validation study (SLVS) of an analytical method for the preparation and analysis of per- and polyfluoroalkyl substances (PFAS) in environmental matrices. Conducted as a joint effort by the U.S. Department of Defense (DoD) and the Environmental Protection Agency (EPA), the study was designed to provide data on the accuracy and precision of the method in aqueous matrices (wastewater, surface waters, groundwaters, landfill leachate), solids (soil, sediment, biosolids), and fish and clam tissues. The laboratory standard operating procedure (SOP) used and the results of the study were the basis of the EPA's draft Office of Water *Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS* (EPA Method 1633).

The analytical SOP used for this study was developed in cooperation with chemists at the EPA, the U.S. Navy, and a commercial analytical laboratory, SGS-AXYS in British Columbia, Canada. The method utilizes liquid chromatograph-tandem mass spectrometry (LC-MS/MS) in multiple reaction monitoring mode. The method included sample preparation and sample analysis procedures for aqueous, solid, and tissue matrices.

The objective of the study was to demonstrate the efficacy of the method using PFAS-spiked environmental samples. A Study Plan was developed that in addition to the method SOP, included interim quality assurance and quality control criteria. Extracts for aqueous matrices were prepared via solid-phase extraction (SPE) followed by carbon clean-up. Extracts for soil, sediment, biosolids, and tissue matrices were prepared via solvent extraction, followed by SPE and carbon clean up. Analyte concentrations were determined using either an isotope dilution or extracted internal standard (EIS) quantification schemes; both of which utilize isotopically labeled compounds that are added to the samples prior to extraction. Injection internal standards (IISs), referred to as non-extracted internal standards (NISs) in EPA Method 1633, also were used to determine EIS recoveries and provide a general indicator of overall analytical quality. The method includes 40 target analytes, 24 EIS compounds, and 7 IIS compounds. The isotope dilution and EIS quantification schemes correct the analyte results for the measured recovery. Analytes were quantified and reported as their acid form.

The efficacy of the method was evaluated using a mean matrix spike interim recovery criterion of 70–130% of the spike concentration after correcting for native sample concentrations. Mean matrix spike recoveries for groundwater and surface water, with the exception of NMeFOSAA in surface water, were within criterion. For wastewater, this criterion was met for 37 target analytes, the exceptions being PFDoS, NMeFOSAA, and NEtFOSAA. For landfill leachate, nine analytes did not meet the criterion: PFDoS, NMeFOSAA, ADONA, PFMBA, NFDHA, 9Cl-PF3ONS, PFEESA, and 5:3FTCA.

For soils and sediment, the criterion was met for all 40 analytes. For biosolids, four analytes did not meet the recovery criterion: PFDOS, PFMPA, NFDHA, and 3:3FTCA. For tissues, matrix spike recoveries for PFDOS, NMeFOSE, NEtFOSE, HFPO-DA, 5:3FTCA, and 7:3FTCA were outside the criterion.

The data generated during the study also demonstrated that the interim criteria for the mass calibration, mass calibration verification, initial calibration (ICAL), and initial demonstration of capability can be routinely achieved. The study method requirements for instrument blanks,

calibration verifications, instrument sensitivity checks (ISCs), and qualitative standard analyses were consistently met, with few exceptions. Failure to meet the 70–130% EIS recovery criteria proved to be inconsequential with respect to analyte recoveries. Because of this, it is recommended that the acceptance criteria for standards be adjusted accordingly. A criterion of 50–150% is recommended for EIS recoveries in instrument blanks, calibration verifications, ISCs, and qualitative standards.

The initial precision and recovery (IPR), method detection limit (MDL), lower limit of quantitation verification (LOQVER), ongoing precision and recovery (OPR), and matrix spike results were evaluated to determine if any target analytes should be eliminated for a particular matrix type. Tissue matrix sample results indicate that use of this method for the determination of NMeFOSE and NEtFOSE is inaccurate This is demonstrated through the consistently low recoveries of their associated EIS compounds and the low method analyte responses. These analytes in particular demonstrated that there is a limitation to the adjustments that can be made from the EIS recovery. When EIS recoveries fell below 10%, method analyte recoveries were over adjusted, resulting in recoveries well over 150%. This suggests that the lower limit of acceptable EIS recoveries should be set near or at 10%. With respect to IIS recoveries, study data indicate IIS recoveries routinely can meet a 50–150% criteria.

Given the success of the method in this SLVS, the EPA published the method as draft EPA Method 1633 in September 2021. The method is now being tested further in a multi-laboratory validation study that will be completed in 2022.

EXECUTIVE SUMMARY

E.S.1 INTRODUCTION

This report presents the results of a single-laboratory validation study (SLVS) of a method for the analysis of per- and polyfluoroalkyl substances (PFAS) in environmental matrices other than drinking water. The U.S. Environmental Protection Agency (EPA) previously published a method for select PFAS by solid-phase extraction (SPE) and liquid chromatograph-tandem mass spectrometry (LC-MS/MS) in drinking water (Methods 533 and 537.1). While many commercial analytical laboratories adapted the EPA drinking water methods to measure PFAS in other regulated environmental matrices, there remained a critical need to develop and validate methods (e.g., 40 CFR Part 136, SW-846) for complex water matrices, solids, and tissues.

The SLVS was undertaken through the U.S. Department of Defense (DoD) Strategic Environmental Research and Development Program (SERDP). Conducted as a joint effort by the DoD and the EPA, the study was designed to provide validating data for the method in critical regulated matrices in aqueous matrices (wastewater, surface waters, groundwaters, landfill leachate), solids (soil, sediment, biosolids) and tissues (fish and clam).

The data from the SLVS were used by the EPA Office of Water in developing the draft *Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS* (EPA Method 1633). EPA Method 1633 will subsequently be used for a multi-laboratory validation study (MLVS) needed to support a final promulgated EPA 1600-series method. These studies also are being conducted to include the information that the EPA Office of Land and Emergency Management (OLEM) needs to consider an EPA SW-846 method.

E.S.2 OBJECTIVES

There are two primary objectives to this overall project:

- Conduct a SLVS with a PFAS analytical method developed by EPA and a commercial analytical laboratory (SGS AXYS Analytical Services Ltd). The results from this laboratory study will be used to publish a draft method that will be used in a multi-laboratory validation study. The details of the method, SLVS design, and results are presented in this report.
- Conduct a MLVS in order to move towards the goal of establishing a promulgated analytical method for PFAS. The focus of the MLVS is to generate the necessary data to document the precision and accuracy of the analytical method for quantitation of PFAS in environmental matrices.

The main objective of this SLVS was to develop a laboratory analysis method that accomplished the following:

1. Identified and quantified PFAS (Table ES-1) in aqueous matrices (groundwater, surface water, landfill leachate, and wastewater), solids (soil, sediment, and biosolids), and tissues using the isotope dilution LC-MS/MS method.

2. Achieved a low parts per trillion (ppt) level of quantitation (LOQ) in aqueous matrices and parts per billion (ppb) in solids and tissues.
3. Capable of being implemented at a typical mid-sized, full-service, environmental laboratory.

E.S.3 METHOD DESCRIPTION

The study objectives and design are described in the *Single-Laboratory Validation Study Plan and Analytical Method Standard Operating Procedure*, which is included as Appendix A to the report. The study was designed to resemble what would be required in the *Protocol for Review and Validation of New Methods for Regulated Organic and Inorganic Analytes in Wastewater under EPA's Alternate Test Procedure Program* (EPA Office of Water, 2018). This is not an Alternate Test Procedure, but the number of matrices mirrors what would be required for an ATP for national use.

The method this study validated is based on a standard operating procedure (SOP) that was previously selected as the basis of an isotope dilution method that had previously been in development by the EPA's Office of Land and Emergency Management (OLEM). The SOP was developed by SGS AXYS Analytical Services Ltd (SGS AXYS) in Sydney, British Columbia, Canada. The project team made modifications to the method to ensure the quality control aspects of the method were consistent with those of the DoD and EPA. The complete method used for this study is provided in Appendix A to this report. The list of 40 PFAS that were included in this validation study are listed in Table ES-1.

Sample preparation and extraction methods are included in the method. Matrices evaluated by this study included wastewater, surface water, groundwater, landfill leachate, soil, sediment, biosolids, and clam and fish tissues. The aqueous matrices were prepared via SPE and carbon clean-up processes. Soil, sediment, biosolids, and tissue matrices were prepared via solvent extraction and SPE, and carbon clean up processes.

Analyte concentrations were determined using either an isotope dilution or extracted internal standard (EIS) quantification scheme; both of which utilize isotopically labeled compounds that are added to the samples prior to extraction (EIS). At the time of validation, only twenty-four isotopically labeled analogs of the 40 target analytes were commercially available; therefore, only 24 target analytes could be quantified using isotope dilution quantitation. All other analytes are quantified using EIS quantitation using these isotopically labeled analogs. The recovery of both of these quantification schemes correct the analyte results. Analytes are quantified and reported as their acid form.

Seven injection internal standards (IIS), also known as non-extracted internal standards (NIS), are used to determine isotopically labeled compound (EIS) recoveries and provide a general indicator of overall analytical quality. The list of 40 target analytes, 24 EIS compounds, and 7 IIS compounds is provided in Table ES-1.

The study was conducted in three phases:

- Phase 1: Initial calibration (ICAL), initial demonstration of capability, method detection limit (MDL) study, and limit of detection (LOD) and LOQ verifications.

- Phase 2: Method evaluation in wastewaters, surface water, landfill leachate, and groundwater matrices.
- Phase 3: Method evaluation in biosolids, soil, sediment, and clam and fish tissue matrices.

The Study Plan required that Phase 1 include the verification of the LOD and LOQ in each matrices type (aqueous, solid, tissue) in accordance with DoD Quality Systems Manual version 5.3, Table B-15 (QSM 5.3) requirements. After the initial analyses, it was determined that the verification scheme required by QSM 5.3 artificially elevated the LOQ in some instances. As a result, the LOD verification was eliminated from the submittal, and the reporting scheme was adjusted to report detections above the MDL, not the LOD, and to qualify detections between the MDL and LOQ with a "J" qualifier. The LOQ verifications conducted in Phase 1 were intended to be carried forward and applied to the quality assurance/quality verification in Phases 2 and 3. Instead, SGS AXYS conducted LOQ verification samples with every 20 (or less) spiked matrix samples in Phases 2 and 3. Because these verifications met the QSM 5.3 requirements, they were used in lieu of the Phase 1 results. Evaluation of the LOQ verifications associated with study sample preparation batches provides greater insight into the performance of the method.

Phase 2 and Phase 3 matrix samples included seven wastewater samples, three groundwater samples, three surface water samples (two fresh and one marine), three landfill leachates, seven soils, three sediment (two fresh and one marine), three biosolid samples, and three tissue samples (two fish and one clam). Triplicate baseline or native PFAS concentrations were measured in all samples. Matrix spike concentrations for the matrices were first determined based on the LOQs established for the matrices and modified as necessary based on the measured native concentrations. Matrix spiking concentrations by matrices are given in Table ES-2.

For groundwater, the mean native concentration of perfluorooctanesulfonic acid (PFOS) in all three samples was greater than the matrix spike concentrations. The matrix spike evaluation rule used for this study considered samples with mean native concentrations that exceeded the spike concentration unusable, and thus those data could not be included in the mean and standard deviation spike recovery calculations. To provide useable data, SGS AXYS was instructed to dilute the original samples they received with PFAS-free water, after homogenization, to fill four 500-mL containers for each sample. One container was processed without the addition of target analytes, whereas the other three containers were spiked with high concentrations of target analytes. These matrix spikes are referred to as "High 2" throughout the report. The spiking concentrations were selected to ensure that the concentration of the spike was greater than the concentration of PFOS in the native sample.

In addition to the LOQ verification, the method included method blank analyses, ongoing precision and recovery (OPR) analyses, and an analysis of recoveries of the EIS and IIS spikes.

Raw data and reporting forms equivalent to a hardcopy data package were submitted for data reported for all phases of the study, and electronic data deliverables (EDDs) in Excel format were submitted for Phase 2 and 3 data. Multiple data packages and EDDs were submitted for each phase of the study, and all were reviewed for completeness and data quality. Data were evaluated based on the preliminary performance criteria described by the method and Study Plan (Appendix A). A total of 57,723 individual results were reviewed and validated in this study.

Table ES-1. Names, Abbreviations, and CAS Registry Numbers for Target PFAS, EIS, and IIS

Analyte Name	Abbreviation	CAS Number
Perfluoroalkyl carboxylic acids		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluoroalkyl sulfonic acids		
Acid Form		
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentansulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
Fluorotelomer sulfonic acids		
1*H*,1*H*, 2*H*, 2*H*-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4
1*H*,1*H*, 2*H*, 2*H*-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2
1*H*,1*H*, 2*H*, 2*H*-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4
Perfluorooctane sulfonamides		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2
Perfluorooctane sulfonamidoacetic acids		
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9

Table ES-1. Names, Abbreviations, and CAS Registry Numbers for Target PFAS, EIS, and IIS

Analyte Name	Abbreviation	CAS Number
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
Perfluorooctane sulfonamide ethanols		
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2
Per- and Polyfluoroether carboxylic acids		
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
4,8-Dioxa-3*H*-perfluorononanoic acid	ADONA	919005-14-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Ether sulfonic acids		
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7
Fluorotelomer carboxylic acids		
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5
2*H*,2*H*,3*H*,3*H*-Perfluorooctanoic acid	5:3FTCA	914637-49-3
3-Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4
EIS Compounds		
Perfluoro-n-[$^{13}C_4$]butanoic acid	$^{13}C_4$-PFBA	NA
Perfluoro-n-[$^{13}C_5$]pentanoic acid	$^{13}C_5$-PFPeA	NA
Perfluoro-n-[1,2,3,4,6-$^{13}C_5$]hexanoic acid	$^{13}C_5$-PFHxA	NA
Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid	$^{13}C_4$-PFHpA	NA
Perfluoro-n-[$^{13}C_8$]octanoic acid	$^{13}C_8$-PFOA	NA
Perfluoro-n-[$^{13}C_9$]nonanoic acid	$^{13}C_9$-PFNA	NA
Perfluoro-n-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid	$^{13}C_6$-PFDA	NA
Perfluoro-n-[1,2,3,4,5,6,7-$^{13}C_7$]undecanoic acid	$^{13}C_7$-PFUnA	NA
Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid	$^{13}C_2$-PFDoA	NA
Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid	$^{13}C_2$-PFTeDA	NA
Perfluoro-1-[2,3,4-$^{13}C_3$]butanesulfonic acid	$^{13}C_3$-PFBS	NA
Perfluoro-1-[1,2,3-$^{13}C_3$]hexanesulfonic acid	$^{13}C_3$-PFHxS	NA
Perfluoro-1-[$^{13}C_8$]octanesulfonic acid	$^{13}C_8$-PFOS	NA

Table ES-1. Names, Abbreviations, and CAS Registry Numbers for Target PFAS, EIS, and IIS

Analyte Name	Abbreviation	CAS Number
Perfluoro-1-[$^{13}C_8$]octanesulfonamide	$^{13}C_8$-PFOSA	NA
N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	D_3-NMeFOSAA	NA
N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	D_5-NEtFOSAA	NA
1*H*,1*H*,2*H*,2*H*-Perfluoro-1-[1,2-$^{13}C_2$]hexanesulfonic acid	$^{13}C_2$-4:2FTS	NA
1*H*,1*H*,2*H*,2*H*-Perfluoro-1-[1,2-$^{13}C_2$]octanesulfonic acid	$^{13}C_2$-6:2FTS	NA
1*H*,1*H*,2*H*,2*H*-Perfluoro-1-[1,2-$^{13}C_2$]decanesulfonic acid	$^{13}C_2$-8:2FTS	NA
Tetrafluoro-2-heptafluoropropoxy-$^{13}C_3$-propanoic acid	$^{13}C_3$-HFPO-DA	NA
N-methyl-d_7-perfluorooctanesulfonamidoethanol	D_7-NMeFOSE	NA
N-ethyl-d_9-perfluorooctanesulfonamidoethanol	D_9-NEtFOSE	NA
N-ethyl-d_5-perfluoro-1-octanesulfonamide	D_5-NEtFOSA	NA
N-methyl-d_3-perfluoro-1-octanesulfonamide	D_3-NMeFOSA	NA
IIS Compounds		
Perfluoro-n-[2,3,4-$^{13}C_3$]butanoic acid	$^{13}C_3$-PFBA	NA
Perfluoro-n-[1,2,3,4-$^{13}C_4$]octanoic acid	$^{13}C_4$-PFOA	NA
Perfluoro-n-[1,2-$^{13}C_2$]decanoic acid	$^{13}C_2$-PFDA	NA
Perfluoro-n-[1,2,3,4-$^{13}C_4$]octanesulfonic acid	$^{13}C_4$-PFOS	NA
Perfluoro-n-[1,2,3,4,5-$^{13}C_5$] nonanoic acid	$^{13}C_5$-PFNA	NA
Perfluoro-n-[1,2-$^{13}C_2$]hexanoic acid	$^{13}C_2$-PFHxA	NA
Perfluoro-1-hexane[$^{18}O_2$]sulfonic acid	$^{18}O_2$-PFHxS	NA

Table ES-2. Study Sample Spiking Levels

Matrices Type	Native Samples (*n* = 3)	Low-Level Spiked Samples (*n* = 3)	Mid-Level Spiked Samples (*n* = 3)	High-Level Spiked Samples (*n* = 3)
Aqueous[1]	NA	2.5 × LOQ[3]	5 × LOQ[3]	7.5 × LOQ[3]
Solids[2]	NA	4 × LOQ[4]	6 × LOQ[4]	10 × LOQ[4]

Notes:
1. Aqueous samples included wastewater, groundwater, surface water, and landfill leachate
2. Solids samples included soils, sediments, biosolids, and tissue
3. Aqueous LOQs range from 1.6 ng/L to 40 ng/L
4. Solid LOQs range from 0.32 ng/g to 10.0 ng/g

In addition, a holding time study was conducted using the matrices indicated for Phase 2 and 3, and is discussed separately in Section E.S.5.

E.S.4 PERFORMANCE EVALUATION

The data generated during the SLVS demonstrated that the interim criteria for the mass calibration, mass calibration verification, initial calibration, and initial demonstration of capability can be routinely achieved. The study method requirements for instrument blanks, calibration verifications, instrument sensitivity checks (ISCs), and qualitative standard analysis were consistently met with few exceptions. Failure to meet the 70–130% EIS recovery criteria proved to be inconsequential with respect to matrix spike analyte recoveries. Because of this, a conclusion from the report is that the acceptance criteria for standards be adjusted to a criterion of 50–150% for EIS recoveries in instrument blanks, calibration verifications, ISCs, and qualitative standards.

The initial precision and recovery (IPR), MDL, lower limit of quantitation verification (LOQVER), OPR, and native sample and matrix spike results were evaluated to determine if any target analytes should be eliminated for a particular matrix type. Tissue matrix sample results indicated that use of this method for the determination of NMeFOSE and NEtFOSE is not accurate. This is demonstrated through the consistently low recoveries of their associated EIS compounds and the low method analyte responses. These analytes in particular demonstrated that there is a limitation to the adjustment of analyte recoveries based on the measured EIS recovery. When EIS compound recoveries fell below 10%, method analyte recoveries were over adjusted, resulting in recoveries well over 150%. This suggests that the lower limit of acceptable EIS recoveries should be set near or at 10%. With respect to IIS recoveries, study data indicates IIS recoveries routinely can meet a 50–150% criteria.

Native PFAS were detected in all matrices samples. In groundwater and wastewater samples, PFBA, PFPeA PFHxA, PFOA, PFNA, PFDA, PFBS, PFPeS, PFHxS, PFHpS, and PFOS were measured in all samples. The fluorotelomer sulfates (FTS) and PFOSA also were measured, but not in all samples. Of the 40 analytes measured, the following compounds were not detected in any groundwater, wastewater, surface water, soil, sediment, or tissue samples: HFPO-DA, ADONA, PFMPA, PFMBA, PFMPA, NFDHA, 9Cl-PF3ONS, 11Cl-PF3OUdS, PFEESA, or 3:3FTCA. HFPO-DA, PFMBA, PFEESA and 3:3FTCA were reported in one of the three landfill leachate samples (Table C-15), but not in the other two. The only matrices reporting FTCA were the landfill leachates and the biosolids samples.

The process for calculating mean percent matrix spike recoveries included determining the mean spike concentration and mean percent recovery for the 40 PFAS analytes from three subsamples for each environmental sample. The mean percent recovery is the mean for all the samples, spike levels, and triplicate analysis for each of the 40 PFAS analytes. For example, the groundwater mean percent recovery is for the seven separate samples, the four (low, medium, high and “high 2”) spike levels, conducted in triplicate for the 40 PFAS analytes. Non-detected (U-flagged) and blank-contaminated (B-flagged) data were not included in these calculations. Additional data reported includes the lowest and highest reported percent recovery for the individual samples, and the relative standard deviation and the standard deviation for the matrix samples.

Table ES-3 shows a summary for aqueous matrices of the mean matrix spike results. Generally, the matrix spike recoveries were robust for groundwater and surface water where all recoveries

were within 70–130%, with the exception of NMeFOSAA in surface water. That analyte appears problematic across all aqueous matrices. For wastewater, the analytical method was sufficiently robust for most compounds, with the exceptions of PFDoS, NMeFOSAA, and NEtFOSAA. For landfill leachate, there are nine analytes for which the matrix recoveries may be measured by this method with less accuracy than other analytes. Nine analytes had mean recoveries outside of 70–130%: two below 70% (PFDoS and NFDHA), and seven above 130% (NMeFOSAA, ADONA, PFMPA, PFMBA, 9Cl-PF3ONS, PFEESA, 5:3FTCA).

Table ES-4 presents the results for the solid matrices and tissues. For soils and sediments, the mean percent recoveries for all 40 target analytes were between 70–130%. For biosolids, there were four mean concentrations outside the recovery criterion: PFDoS, PFMPA, NFDHA, and 3:3FTCA.

Tissue matrix spike recoveries for the two fish and one clam tissue samples are also shown in Table ES-4. For these tissue samples, target analytes that were outside the range of the 70–130% criterion include NMeFOSE, NEtFOSE, 5:3FTCA, and 7:3FTCA.

An assessment of the accuracy and precision of the method is presented in this report. Matrix spike percent recoveries were statistically evaluated as the metric of method accuracy (Figure ES-1). As noted in Tables ES-3 and ES-4, There are several combinations of PFAS x Matrix outside the control limits (70% and 130% recovery); however, no statistical trend is indicated for increasing spike concentrations for all target analytes and all matrices. The relative standard deviation among laboratory subsamples were similarly evaluated as the metric of method precision (Figure ES-2). In this case, there is a significant decreasing trend apparent with increasing spike concentrations (Figure ES-2A). This indicates greater method precision at higher concentrations. As with the accuracy analysis, however, mean relative standard deviations for several combinations of PFAS x Matrix are outside of the control limit established at 20% (Figure ES-2B). All data used in the evaluation of method accuracy and precision were combined and evaluated collectively in terms of compliance with the control limits (Figure ES-3). This analysis demonstrated that the vast majority of the PFAS method analyte and matrix combinations (>93%) were within the accuracy and precision control limits.

E.S.5 HOLDING TIME STUDY

As part of the SLVS, a holding time study was designed and carried out in cooperation with EPA. The goal was to provide EPA with results to establish the holding times and preservation conditions for PFAS analytes that may be added to Table II at 40 CFR Part 136 in conjunction with the expected proposal and promulgation of EPA Method 1633.

SGS AXYS conducted the holding time study with the goal of assessing how long environmental samples and extracts of samples can be held, at various temperatures, before PFAS degrade, become unextractable, or otherwise change concentrations so that the samples are no longer representative of the matrices from which they are derived. This study included one groundwater, one soil, one sediment, and one biosolids sample matrix, as well as extracts of each of these matrices. The matrices were spiked at the mid-level concentrations and analyzed with three (3) replicate samples for each time increment. The samples were put into the appropriate sample containers as though they were field samples.

Table ES-3. Summary by Aqueous Matrices of Average Matrix Spike Results

Analyte	Aqueous Samples			
	Groundwater	Surface Water	Wastewater	Landfill Leachate
PFBA	+	+	+	+
PFPeA	+	+	+	+
PFHxA	+	+	+	+
PFHpA	+	+	+	+
PFOA	+	+	+	+
PFNA	+	+	+	+
PFDA	+	+	+	+
PFUnA	+	+	+	+
PFDoA	+	+	+	+
PFTrDA	+	+	+	+
PFTeDA	+	+	+	+
PFBS	+	+	+	+
PFPeS	+	+	+	+
PFHxS	+	+	+	+
PFHpS	+	+	+	+
PFOS	+	+	+	+
PFNS	+	+	+	+
PFDS	+	+	+	+
PFDoS	+	+	-	-
4:2 FTS	+	+	+	+
6:2 FTS	+	+	+	+
8:2 FTS	+	+	+	+
PFOSA	+	+	+	+
N-MeFOSA	+	+	+	+
N-EtFOSA	+	+	+	+
N-MeFOSAA	+	-	-	-
N-EtFOSAA	+	+	-	+
N-MeFOSE	+	+	+	+
N-EtFOSE	+	+	+	+
HFPO-DA	+	+	+	+
ADONA	+	+	+	-
PFMPA	+	+	+	+
PFMBA	+	+	+	-
NFDHA	+	+	+	-
9Cl-PF3ONS	+	+	+	-
11Cl-PF3OUdS	+	+	+	+
PFEESA	+	+	+	-
3:3 FTCA	+	+	+	+
5:3 FTCA	+	+	+	-
7:3 FTCA	+	+	+	+

+	Average matrix spike recoveries between 70–130%
-	Average matrix spike recoveries outside of 70–130%

Table ES-4. Summary by Matrix of Average Matrix Spike Results

Analyte	Solids			Tissue
	Soils	Sediment	Biosolids	
PFBA	+	+	+	+
PFPeA	+	+	+	+
PFHxA	+	+	+	+
PFHpA	+	+	+	+
PFOA	+	+	+	+
PFNA	+	+	+	+
PFDA	+	+	+	+
PFUnA	+	+	+	+
PFDoA	+	+	+	+
PFTrDA	+	+	+	+
PFTeDA	+	+	+	+
PFBS	+	+	+	+
PFPeS	+	+	+	+
PFHxS	+	+	+	+
PFHpS	+	+	+	+
PFOS	+	+	+	+
PFNS	+	+	+	+
PFDS	+	+	+	+
PFDoS	+	+	-	-
4:2 FTS	+	+	+	+
6:2 FTS	+	+	+	+
8:2 FTS	+	+	+	+
PFOSA	+	+	+	+
N-MeFOSA	+	+	+	+
N-EtFOSA	+	+	+	+
N-MeFOSAA	+	+	+	+
N-EtFOSAA	+	+	+	+
N-MeFOSE	+	+	+	-
N-EtFOSE	+	+	+	-
HFPO-DA	+	+	+	-
ADONA	+	+	+	+
PFMPA	+	+	-	+
PFMBA	+	+	+	+
NFDHA	+	+	-	+
9Cl-PF3ONS	+	+	+	+
11Cl-PF3OUdS	+	+	+	+
PFEESA	+	+	+	+
3:3 FTCA	+	+	-	+
5:3 FTCA	+	+	+	-
7:3 FTCA	+	+	+	-

+	Average matrix spike recoveries between 70–130%
-	Average matrix spike recoveries outside of 70–130%

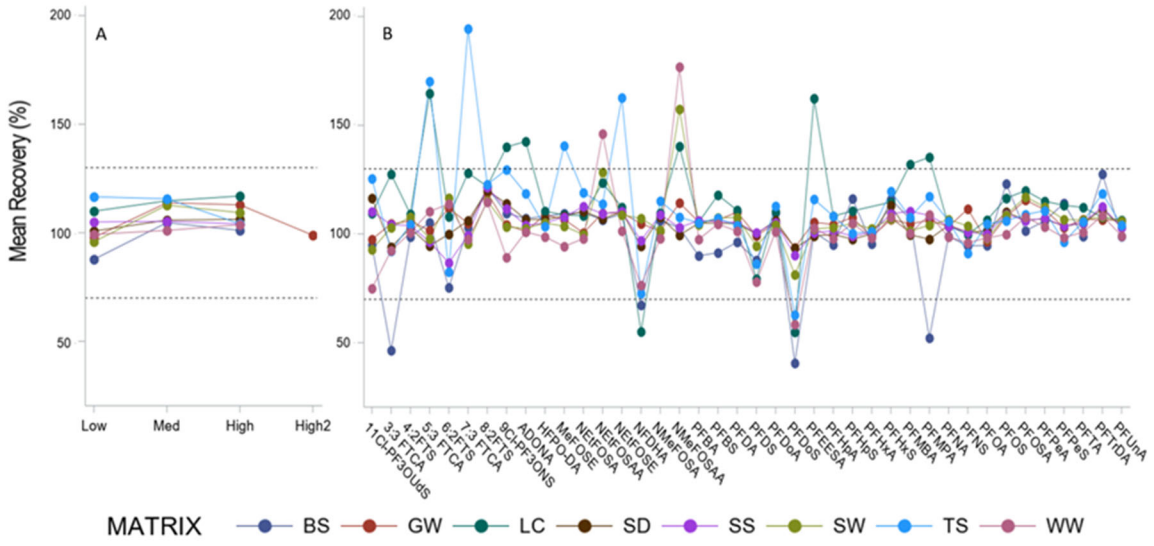


Figure ES-1. Accuracy Analysis

Significant two-way interactions from ANOVA are presented in the report. Panel A represents mean spike recoveries for each matrix and spike category combination (the High 2 spike category is specific to the groundwater matrix) and means over all PFAS target analytes. Similarly, Panel B represents each PFAS method analyte and matrix combination and means over spike category. Horizontal reference lines represent 70% and 130% control limits.

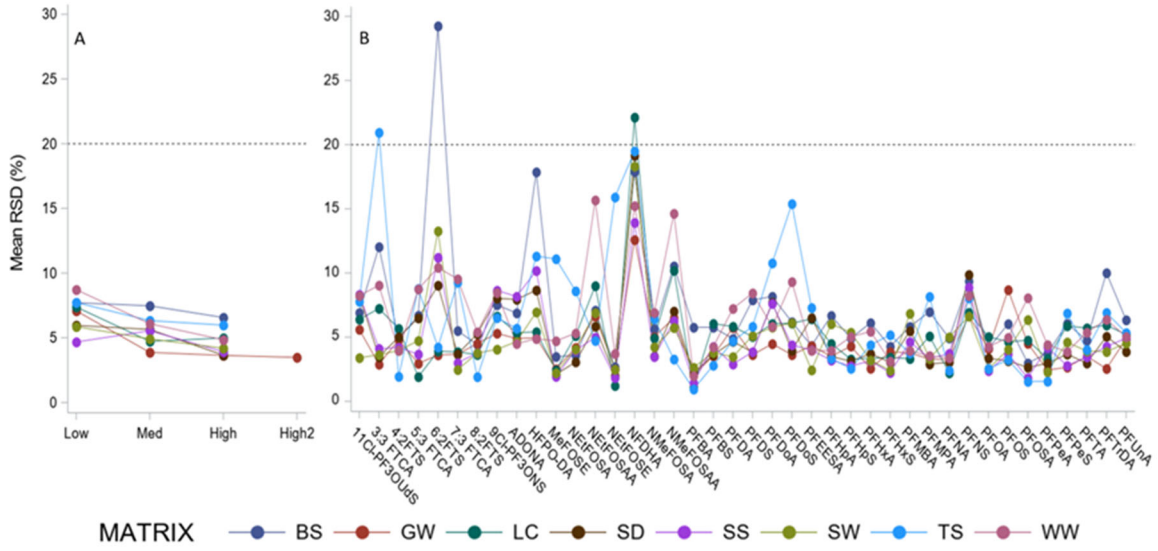


Figure ES-2. Precision Analysis

Significant two-way interactions from ANOVA are presented in the report. Panel A represents mean RSDs for each matrix and spike category combination (the High 2 spike category is specific to the groundwater matrix) and means over all PFAS target analytes. Similarly, Panel B represents each PFAS method analyte and matrix combination and means over the spike categories. The horizontal reference line represents a 20% control limit.

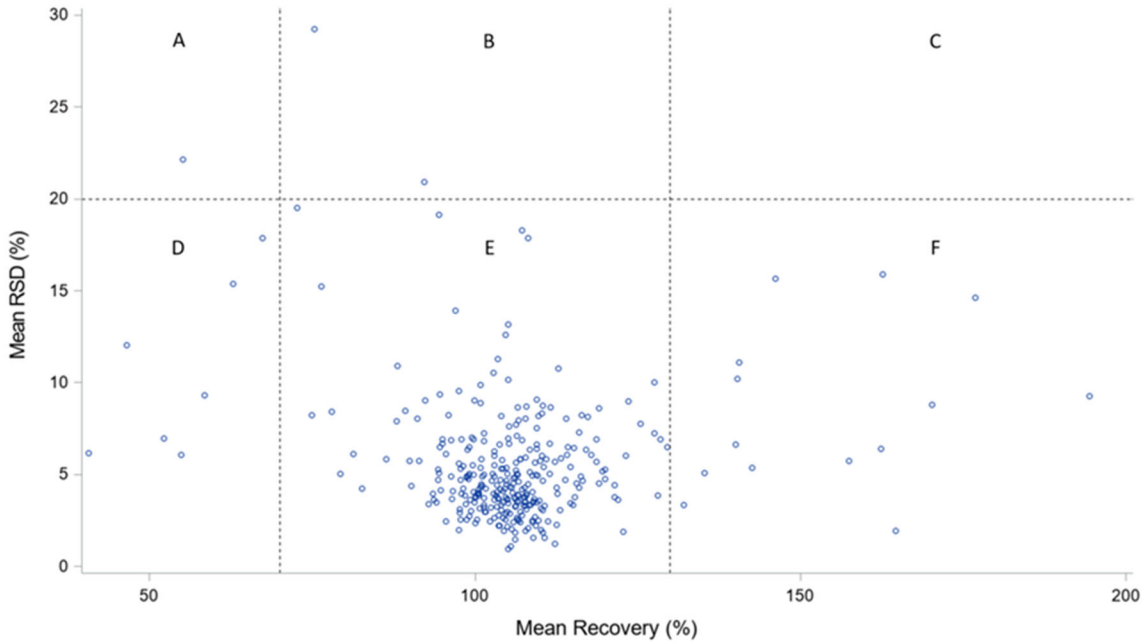


Figure ES-3. Relationship Between Accuracy and Precision

Relationship between accuracy and precision as paired by the two-way interaction results from Figures 1B and 3B above that represent each PFAS method analyte and matrix combination and means over spike category. Dashed reference lines reflect control limits. Panels A, B, C, D, E and F represent permutations with low bias and imprecision, accuracy and imprecision, high bias and imprecision, low bias and precision, accuracy and precision, and high bias and precision, respectively. The vast majority of PFAS method analyte and matrix combinations (>93%) were within accuracy and precision control limits.

SGS AXYS had previously conducted a separate holding time in 2018, which included reagent water, one river surface water and two wastewater effluents. That study was designed to cover different sample containers, three storage temperatures, a holding time of up to 180 days, and tested 29 of the 40 PFAS tested by the current study. The DoD with the EPA decided to incorporate the 2018 SGS AXYS study data with the data acquired in this current study.

EPA conducted the holding time analyses, and a full report on the methods and findings is incorporated as Appendix I. The EPA's analysis of the data determined that aqueous samples may be held for up to 28 days when stored at 4ºC, with the caveat that the study data showed signs of transformation of some precursors to other PFAS when the sample was stored beyond 7 days. This is likely to increase the observed concentrations of some PFAS if precursors are present in the sample. For greater stability of PFAS, the aqueous samples can be stored at -20ºC for up to 90 days.

For biosolids, all PFAS were stable for up to 90 days when stored at either 4ºC or -20ºC. Because microbiological activity in biosolids samples at 0–6ºC may lead to production of gases that may cause the sample to be expelled from the container when it is opened, as well as producing noxious odors, the EPA recommended that samples be frozen if they need to be stored for more than a few days before extraction.

For solids samples, EPA found that samples could be held for 90 days when stored at either 4ºC or -20ºC, except when analyzing for NFDHA, which was not stable at either of the temperatures tested and must be analyzed as soon as possible after collection if it is an analyte of concern.

E.S.6 Implementation and Next Steps

Given the success of the method in this SLVS, the EPA's Office of Water published the draft *Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS*. The importance of the publication of the method (and by extension this Study) is reflected in the DoD's December 7, 2021, *Memorandum for the Update for Establishing a Constituent Methodology for the Analysis of Per- and Polyfluoroalkyl Substances in Media Other than Drinking Water.* The DoD memorandum requires that all new contracts and task orders after December 31, 2021, use draft EPA Method 1633 for the analysis for PFAS in matrices other than drinking water using a laboratory accredited to the method/matrix/analyte by the DoD Environmental Laboratory Accreditation program (ELAP).

The MLVS is already underway. Ten laboratories, seven commercial analytical laboratories, and two state laboratories are initiating the Initial Demonstration of Capabilities for calibration and establishing MDLs. Beginning in early 2022, PFAS-spiked samples of groundwater (3), surface water (3), wastewater (7), soil (3), sediment (3), fish tissue (3), landfill leachate (3), and biosolids (3) will be sent as blind samples to the participating laboratories following the same design that was used in the SLVS. The results from the MLVS will be used to validate the method and refine/set the quality assurance and quality control performance criterion to support promulgation of EPA Method 1633.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

LIST OF ACRONYMS AND ABBREVIATIONS

AFCEC	Air Force Civil Engineer Center
AFFF	aqueous film-forming foam
ANOVA	analysis of variance
CAS	Chemical Abstract Service
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CV	calibration verification
DoD	U.S. Department of Defense
EDD	electronic data deliverable
EIS	extracted internal standard
ELAP	Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
ESTCP	Environmental Security Technology Certification Program
ICAL	initial calibration
IDA	Institute for Defense Analysis
IDC	initial demonstration of capability
IIS	injection internal standard
IPR	initial precision and recovery
ISC	instrument sensitivity check
LCMRL	lowest concentration minimum reporting level
LC–MS/MS	liquid chromatography–tandem mass spectrometry
LHA	Lifetime Health Advisory
LOD	limit of detection
LOQ	limit of quantitation
LLOQ	lower limit of quantitation
LOQVER	lower limit of quantitation verification
MDL	method detection limit

MDL_b	MDL based on method blanks
MDL_s	MDL based on spiked samples
mg/L	milligram per liter
MLV	multi-laboratory validation
MRM	multiple reaction monitoring
NAVSEA	Naval Sea Systems Command
NIS	non-extracted internal standard
OLEM	Office of Land and Emergency Management
OPR	ongoing precision and recovery
ORD	Office of Research and Development
PFAS	per- and polyfluoroalkyl substances
PFAS acronyms	**See** Table 1-1
QA	quality assurance
QC	quality control
QSM	Quality Systems Manual
RF	response factor
RR	response ratio
RSD	relative standard deviation
SERDP	Strategic Environmental Research and Development Program
SLVS	single-laboratory validation study
SOP	standard operating procedure
SOW	statement of work
SPE	solid phase extraction
SW	solid waste
TDS	total dissolved solids
TSS	total suspended solids
USACE	U.S. Army Corps of Engineers

1 INTRODUCTION

This report presents the results of a single-laboratory validation study (SLVS) of a method for the analysis of per- and polyfluoroalkyl substances (PFAS) in environmental matrices other than drinking water. The U.S. Environmental Protection Agency (EPA) previously published a method for select PFAS by solid phase extraction (SPE) and liquid chromatograph-tandem mass spectrometry (LC-MS/MS) in drinking water (Methods 533 and 537.1). While many commercial analytical laboratories adapted the EPA drinking water methods to measure PFAS in other regulated environmental matrices, there remained a critical need to develop and validate methods (e.g., 40 CFR Part 136, SW-846) for complex water matrices, solids, and tissues (EPA, 2020).

The SLVS was undertaken through the U.S. Department of Defense (DoD) Strategic Environmental Research and Development Program (SERDP). Conducted as a joint effort by the DoD and the EPA, the study was designed to provide validating data for the method in critical regulated matrices including aqueous (wastewater, surface waters, groundwaters, landfill leachate), solids (soil, sediment, biosolids), and tissue (fish and clam tissues). The two primary objectives are as follows:

- Complete a SLVS with a PFAS analytical method developed by the EPA and a commercial analytical laboratory (SGS AXYS Analytical Solutions Ltd. Sydney, BC, Canada). The results from this laboratory study will be used to publish a draft method that will be used in a multi-laboratory validation study (MLVS). The details of the method, SLVS design, and results are presented in this report.
- Conduct a MLVS in order to move toward the goal of establishing a standardized analytical method for PFAS. The focus of the MLVS will be to generate the necessary data to document the precision and accuracy of the analytical method for quantitation of PFAS in environmental matrices.

The data from the SLVS were used by the EPA Office of Water in developing the draft *Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS* (EPA Method 1633). EPA Method 1633 will subsequently be used for the MLVS needed to support a final EPA 1600-series method. These studies also are being conducted to include the information that the EPA Office of Land and Emergency Management (OLEM) needs to consider an EPA SW-846 method.

1.1 BACKGROUND

PFAS are a class of chemicals of environmental concern that are increasingly regulated by the EPA and many state agencies due to their widespread occurrence in the environment and demonstrated bioaccumulation in humans and ecological receptors. Evidence that continued exposure to certain PFAS above specific levels may lead to adverse health effects led the EPA (2009) to publish provisional health advisories levels for drinking water for perfluorooctanoic acid (PFOA) and perfluorooctanoic sulfonate (PFOS) in 2009. The EPA subsequently revised their 2009 Lifetime Health Advisories (LHAs) for PFOS and PFOA to a level of 70 parts per thousand (ppt) (individually or combined) in drinking water (EPA, 2016a; 2016b). Since then, several state agencies have published even lower values for drinking water (ITRC, 2020).

Publication of the LHAs led to widespread public concern about PFAS fate and transport, potential deleterious effects on human health and ecological receptors, and how to manage these recalcitrant

compounds. The EPA recognized the need to develop analytical methods for PFAS for other matrices that are regulated under the Clean Water Act, the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), and the Toxic Substance Control Act, as well as other ongoing efforts to demonstrate potential clean-up and PFAS-containing waste disposal (EPA, 2019; 2020).

The DoD has environmental management responsibilities for PFAS released to the environment associated with the use of aqueous film-forming foam (AFFF) (Leeson et al., 2020; Anderson et al., 2020). The use of AFFF has resulted in the widespread occurrence of PFAS in groundwater, drinking water, soils, sediments, receiving waters, and ecological receptors at many current and former military installations as well as more broadly throughout the community. Site characterization and clean-up of these sites is being conducted principally under CERCLA, but these characterizations are hindered by the lack of an EPA analytical method for PFAS in those matrices managed under the Superfund program. As an interim measure, the DoD Environmental Laboratory Accreditation Program (ELAP) provides accreditation to analytical laboratories that demonstrate competency and document conformance to the standards published in the DoD and Department of Energy Consolidated Quality Services Manual (QSM) for Environmental Laboratories, Version 5.3, Table B-15 (QSM 5.3). Commercial environmental laboratories that demonstrate competency and document conformance to QSM 5.3 are accredited for PFAS analysis in environmental matrices other than drinking water.

Recognizing this challenge and opportunity, the EPA and the DoD are collaborating on the development of an isotope dilution method for non-drinking water aqueous matrices (surface water, groundwater, wastewater influent/effluent, landfill leachate), solids (soil, sediment, biosolids), and tissues (fish and clam). The DoD and EPA have worked together to develop the testing method, identify the individual PFAS to be measured, select the performing laboratory, validate the data, and write the present report.

1.2 METHOD SUMMARY

The analytical method validated by the present study included both sample preparation and sample analysis procedures that are applicable to a variety of environmental matrices. The matrices evaluated by this study included wastewater, surface water, groundwater, landfill leachate, soil, sediment, biosolids, and tissue. The aqueous matrices were prepared via SPE and carbon clean-up processes. Soil, sediment, biosolids, and tissue matrices were prepared via solvent extraction and SPE, followed by carbon clean-up processes. The method utilized LC-MS/MS in multiple reaction monitoring (MRM) mode to evaluate quantification and confirmation (where applicable) of ions of each of the 40 target analytes (Table 1-1). Analyte concentrations were determined using either an isotope dilution or extracted internal standard (EIS) quantification scheme; both utilize isotopically labeled compounds (EIS) that were added to the samples prior to extraction. At the time of validation, only 24 isotopically labeled analogs of the 40 target analytes were commercially available, and therefore only 24 target analytes could be quantified using isotope dilution quantitation. All other analytes were quantified using EIS quantitation with these isotopically labeled analogs. Recovery of both quantification schemes corrects the analyte results. Analytes were quantified and reported as their acid form.

Seven injection internal standards (IIS), also known as non-extracted internal standards (NIS), are used to determine isotopically labeled compound (EIS) recoveries and provide a general indicator

of overall analytical quality. A list of the 40 target analytes, 24 EIS compounds, and 7 IIS compounds is provided in Table 1-1.

The method this study validated is based on a SOP that was previously being evaluated by the EPA for development as an isotope dilution method that had previously been in development by the EPA's Office of Land and Emergency Management (OLEM). The SOP was developed by SGS AXYS Analytical Services Ltd (SGS AXYS) in Sydney, British Columbia, Canada. The project team made modifications to the method to ensure the quality control aspects of the method were consistent with those of the DoD and EPA. The complete method used for this study is provided in Appendix A to this report. The list of 40 PFAS that were included in this validation study are listed in Table 1-1.

Table 1-1. Names, Abbreviations, and CAS Registry Numbers for Target PFAS, EIS, and IIS

Analyte Name	Abbreviation	CAS Number
Perfluoroalkyl carboxylic acids		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluoroalkyl sulfonic acids		
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentansulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
Fluorotelomer sulfonic acids		
1*H*,1*H*, 2*H*, 2*H*-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4
1*H*,1*H*, 2*H*, 2*H*-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2

Table 1-1. Names, Abbreviations, and CAS Registry Numbers for Target PFAS, EIS, and IIS

Analyte Name	Abbreviation	CAS Number
1*H*,1*H*, 2*H*, 2*H*-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4
Perfluorooctane sulfonamides		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2
Perfluorooctane sulfonamidoacetic acids		
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
Perfluorooctane sulfonamide ethanols		
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2
Per- and Polyfluoroether carboxylic acids		
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
4,8-Dioxa-3*H*-perfluorononanoic acid	ADONA	919005-14-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Ether sulfonic acids		
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7
Fluorotelomer carboxylic acids		
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5
2*H*,2*H*,3*H*,3*H*-Perfluorooctanoic acid	5:3FTCA	914637-49-3
3-Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4
EIS Compounds		
Perfluoro-n-[$^{13}C_4$]butanoic acid	$^{13}C_4$-PFBA	NA
Perfluoro-n-[$^{13}C_5$]pentanoic acid	$^{13}C_5$-PFPeA	NA
Perfluoro-n-[1,2,3,4,6-$^{13}C_5$]hexanoic acid	$^{13}C_5$-PFHxA	NA
Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid	$^{13}C_4$-PFHpA	NA
Perfluoro-n-[$^{13}C_8$]octanoic acid	$^{13}C_8$-PFOA	NA
Perfluoro-n-[$^{13}C_9$]nonanoic acid	$^{13}C_9$-PFNA	NA
Perfluoro-n-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid	$^{13}C_6$-PFDA	NA

Table 1-1. Names, Abbreviations, and CAS Registry Numbers for Target PFAS, EIS, and IIS

Analyte Name	Abbreviation	CAS Number
Perfluoro-n-[1,2,3,4,5,6,7-$^{13}C_7$]undecanoic acid	$^{13}C_7$-PFUnA	NA
Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid	$^{13}C_2$-PFDoA	NA
Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid	$^{13}C_2$-PFTeDA	NA
Perfluoro-1-[2,3,4-$^{13}C_3$]butanesulfonic acid	$^{13}C_3$-PFBS	NA
Perfluoro-1-[1,2,3-$^{13}C_3$]hexanesulfonic acid	$^{13}C_3$-PFHxS	NA
Perfluoro-1-[$^{13}C_8$]octanesulfonic acid	$^{13}C_8$-PFOS	NA
Perfluoro-1-[$^{13}C_8$]octanesulfonamide	$^{13}C_8$-PFOSA	NA
N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	D_3-NMeFOSAA	NA
N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	D_5-NEtFOSAA	NA
1*H*,1*H*,2*H*,2*H*-Perfluoro-1-[1,2-$^{13}C_2$]hexanesulfonic acid	$^{13}C_2$-4:2FTS	NA
1*H*,1*H*,2*H*,2*H*-Perfluoro-1-[1,2-$^{13}C_2$]octanesulfonic acid	$^{13}C_2$-6:2FTS	NA
1*H*,1*H*,2*H*,2*H*-Perfluoro-1-[1,2-$^{13}C_2$]decanesulfonic acid	$^{13}C_2$-8:2FTS	NA
Tetrafluoro-2-heptafluoropropoxy-$^{13}C_3$-propanoic acid	$^{13}C_3$-HFPO-DA	NA
N-methyl-d_7-perfluorooctanesulfonamidoethanol	D_7-NMeFOSE	NA
N-ethyl-d_9-perfluorooctanesulfonamidoethanol	D_9-NEtFOSE	NA
N-ethyl-d_5-perfluoro-1-octanesulfonamide	D_5-NEtFOSA	NA
N-methyl-d_3-perfluoro-1-octanesulfonamide	D_3-NMeFOSA	NA
IIS Compounds		
Perfluoro-n-[2,3,4-$^{13}C_3$]butanoic acid	$^{13}C_3$-PFBA	NA
Perfluoro-n-[1,2,3,4-$^{13}C_4$]octanoic acid	$^{13}C_4$-PFOA	NA
Perfluoro-n-[1,2-$^{13}C_2$]decanoic acid	$^{13}C_2$-PFDA	NA
Perfluoro-n-[1,2,3,4-$^{13}C_4$]octanesulfonic acid	$^{13}C_4$-PFOS	NA
Perfluoro-n-[1,2,3,4,5-$^{13}C_5$] nonanoic acid	$^{13}C_5$-PFNA	NA
Perfluoro-n-[1,2-$^{13}C_2$]hexanoic acid	$^{13}C_2$-PFHxA	NA
Perfluoro-1-hexane[$^{18}O_2$]sulfonic acid	$^{18}O_2$-PFHxS	NA

2 STUDY MANAGEMENT, OBJECTIVES, DESIGN, AND IMPLEMENTATION

2.1 STUDY MANAGEMENT: PFAS METHOD VALIDATION TEAM

A joint EPA and DoD PFAS Method Validation Team was formed to oversee the PFAS analytical method development and validation. Members on the validation team from the DoD included individuals representing SERDP, the Air Force Civil Engineer Center (AFCEC), the Naval Sea Systems Command, and the Army Corps of Engineers (USACE). For the EPA, this included personnel from the Office of Water, Office of Research and Development, and OLEM. HydroGeoLogic Inc (HGL) was contracted to provide laboratory management. Since the draft method used in this SLV stemmed from an SOP developed by SGS AXYS (Sidney, BC, Canada), they were contracted as the laboratory that conducted the analytical activities for this study. The Institute for Defense Analyses (IDA) conducted the statistical analyses on the resulting data. The funding for the SLVS was provided by SERDP.

2.2 STUDY OBJECTIVE AND DESIGN

The SLVS objectives and design are described in the *Single-Laboratory Validation Study Plan and Analytical Method Standard Operating Procedure*, which is attached to this report as Appendix A. The study was designed to be consistent with the matrix requirements in the *Protocol for Review and Validation of New Methods for Regulated Organic and Inorganic Analytes in Wastewater under EPA's Alternate Test Procedure Program* (EPA Office of Water, 2018). (EPA applies similar matrix testing requirements to new methods as it does for alternate test procedures).

The main objective of this SLVS was to develop a laboratory analysis method that could accomplish the following:

- Identify and quantify PFAS (Table 1-1) in aqueous matrices (groundwater, surface water, landfill leachate, and wastewater), solids (soil, sediment, and biosolids), and tissues using the isotope dilution LC-MS/MS method. Aqueous samples were prepared by SPE, while solids and tissue using solvent extraction techniques.
- Achieve a low ppt level of quantitation (LOQ) in aqueous matrices and parts per billion (ppb) in solid and tissue matrices.
- Capable of being implemented at a typical mid-sized, full-service, environmental laboratory.

The SLVS was conducted in three phases. Specific procedures for these phased studies are given in the SLVS Plan in Appendix A and are briefly summarized below.

- Phase 1: Initial calibration (ICAL), initial demonstration of capability, method detection limit (MDL) study, and limit of detection (LOD) and LOQ verifications.
- Phase 2: Method evaluation in wastewaters, surface water, landfill leachate, and groundwater matrices.
- Phase 3: Method evaluation in biosolids, soil, sediment, and tissue matrices.

In addition, a holding time study was conducted using some of the aqueous and solid matrices indicated for Phases 2 and 3 (see Section 12).

2.3 PFAS PRIORITIZATION

The 40 target PFAS analytes used in this SLVS are listed in Table 1-1. The target analyte list includes those PFAS in EPA Method 537.1 (18 PFAS), Method 533 (25 PFAS), and SW-846 Method 8327 (24 PFAS). After the completion of the Study Plan, but prior to the actual analyses, the MLV Study Team made the decision to expand the target analyte list to the 40 PFAS in Table 1-1.

2.4 MATRICES AND SAMPLE SELECTION

The SLVS was designed to provide a test of the method by analyses of real-world environmental matrices, including wastewaters, groundwater, surface water (fresh and marine), soil/sediment (fresh and marine), fish and clam tissues, landfill leachate, and biosolids. To obtain a wide diversity and sufficient quantity of matrices and samples, SERDP and EPA coordinated with municipal, state, and regional contacts to obtain sufficient volumes/mass used in the study.

The list of samples used in this study is found in the SLVS Plan (Appendix A, Attachment 2). Samples and sources are discussed briefly below.

2.4.1 Aqueous samples

EPA's Office of Water arranged for seven representative wastewaters to be shipped to SGS AXYS for the study. The samples were from a publicly owned treatment facility (influent and effluent), a metal finisher, pulp and paper effluent, and a bus-washing station.

Three groundwater samples were collected and contributed to the study. Two samples were collected by the Colorado School of Mines from locations in the Midwest, and one sample was arranged for collection and shipping by the AFCEC from a site in Oklahoma.

Three surface water samples were collected for this study. The Naval Facilities Engineering Systems Command Atlantic arranged for two freshwater samples to be collected from a site in Pennsylvania and SGS AXYS collected a marine surface water sample from offshore water near Sydney, British Columbia, Canada.

Three landfill leachate samples were analyzed. Two samples were arranged for collection by researchers at the University of Florida. One was a leachate sample collected from a municipal solid waste landfill, and the other was collected by the University of Florida from a construction and debris landfill. The third leachate was provided by the EPA from a municipal solid waste incinerator ash landfill.

2.4.2 Solid samples

Seven soil samples, three sediment samples (two freshwater and one marine), and three biosolid samples were contributed by the EPA (Appendix A, Attachment 2). The soil samples were collected in California, Illinois, Montana, New Mexico (two samples), Tennessee, and Utah.

2.4.3 Tissues

Two fish tissue and one clam tissue samples were provided by the EPA. The fish tissue samples were selected to provide a low lipid (1.2%) and high lipid (4%) sample. They were selected from

a repository of frozen homogenized fish fillet tissues collected from the Great Lakes. The clam samples came from frozen samples collected by EPA's Region 10 Laboratory in Port Orchard, WA.

2.5 PREPARATION OF STUDY SAMPLES

SGS AXYS homogenized the contents of the multiple containers received for each sample and subsampled each into HDPE containers. Twelve new containers were prepared for each sample. A list of the sample containers and the final amount of sample prepared for each matrix type is provided in Table 2-1. The sample volume was reduced for landfill leachates due to the complex nature of this matrix, having an elevated potential for interference and high PFAS concentrations.

Table 2-1. Sample Volumes and Containers

Matrix Type	Sample Size
Wastewater	500 mL
Groundwater	500 mL
Surface Water	500 mL
Landfill Leachate	100 mL
Soil	5 g dry weight
Sediment	5 g dry weight
Biosolid	0.5 g dry weight
Tissue	2 g wet weight

The 12 containers were considered to be the original sample containers with respect to sample preparation procedures. Three of these containers for each sample were processed without the addition of target analytes to determine the background concentrations. These are identified as native samples/native concentrations in this report. SGS AXYS was required to add all 40 target analytes to each of the remaining nine containers for each sample at concentrations relative to the LOQ (Table 2-2).

Table 2-2. Study Sample Spiking Levels

Matrices Type	Native Samples (n = 3)	Low-Level Spiked Samples (n = 3)	Mid-Level Spiked Samples (n = 3)	High-Level Spiked Samples (n = 3)
Aqueous[1]	NA	2.5 × LOQ[3]	5 × LOQ[3]	7.5 × LOQ[3]
Solids[2]	NA	4 × LOQ[4]	6 × LOQ[4]	10 × LOQ[4]

Notes:
1. Aqueous samples included wastewater, groundwater, surface water, and landfill leachate
2. Solids samples included soils, sediments, biosolids, and tissue
3. Aqueous LOQs range from 1.6 ng/L to 40 ng/L
4. Solid LOQs range from 0.32 ng/g to 10.0 ng/g

The mean native concentration of PFOS in all three of the groundwater samples was greater than each of the matrix spike concentrations. The matrix spike evaluation rule used for this study considered samples with mean native concentrations that exceeded the spike concentration unusable, and thus these data were not included in the mean and standard deviation spike recovery calculations. As a result, no usable data points were generated by the spikes listed in Table 2-2 for groundwater matrices. Because of this, SGS AXYS was instructed to dilute the original samples they received by a factor of four with PFAS-free water, after homogenization, to fill four 500-mL containers for each sample. One container was processed without the addition of target analytes, whereas the other three containers were spiked with high concentrations of target analytes (Table 2-3). These matrix spikes are referred to as "High 2" throughout this report. The spiking concentrations were selected to ensure that the concentration of the spike was greater than the concentration of PFOS in the native sample. The resulting samples were prepared in accordance with the draft method in Appendix A.

Table 2-3. Groundwater High 2 Spiking Levels

Sample ID	Native Samples (*n* = 1)	High 2 Spiked Samples (*n* = 3)
GW1	N/A	31.25 × LOQ
GW2 & GW3	N/A	62.7 × LOQ

N/A = Not applicable

Study samples were prepared in preparation batches consisting of 20 study samples or less. Each preparation batch also included three quality control (QC) samples: a method blank, an ongoing precision and recovery (OPR), and a lower limit of quantitation verification (LOQVER) sample (also known as low-level OPR, or LLOPR). All three of these batch QC samples consisted of a reference matrix that simulated, as closely as possible, the sample matrix of the study samples included in the batch. A 500-mL aliquot of PFAS-free reagent water was used for aqueous samples, a 5.0-g aliquot of Ottawa sand wetted with 2.5 g of PFAS-free reagent water was used for solid samples, and a 2.0-g aliquot of lingcod (*Ophiodon elongatus*) was used for tissue samples. During method development, EIS compound loss was observed when using dry Ottawa sand. As a result, QC samples and excessively dry study samples were adjusted to contain moisture prior to extraction. The method blanks underwent the preparation and analysis procedures as study samples to demonstrate the extent of background contamination in the analytical system. Target analytes were added to the OPR and LOQVER samples prior to undergoing the same preparation and analysis procedures as the study sample (see Sections 2.5.1, 2.5.2, and 2.5.3) to demonstrate that the analytical system was within method controls. After the EIS compounds were added to the sample, a known amount of the 40 target analytes was added to the OPR and LOQVER QC samples. The OPR was spiked at a concentration coinciding with the mid-point of the calibration and LOQVER was spiked at two times the limit of quantitation.

2.5.1 Aqueous samples

The entire sample amount contained in the 500-mL container of groundwater, surface water, and wastewater was processed for extraction. For landfill leachate samples, the entire volume of sample in the 100-mL container was processed for extraction. The exact sample volume prepared

was determined by subtracting the weight of the dry empty sample bottle post extraction from the weight of the sample bottle containing the sample prior to preparation. Samples were homogenized by inversion and spiked with the EIS compounds. The pH was adjusted to 6.5 ± 0.5. Samples were extracted using an Oasis WAX SPE® cartridge. A solution of methanolic ammonium hydroxide solution was used to rinse the original sample container and elute PFAS from the WAX SPE. Loose ENVI-Carb® carbon was used to eliminate matrix interferences as much as possible prior to concentration and the addition of IIS compounds.

2.5.2 Solid samples

Soils, sediments, and biosolids were thoroughly mixed, and the percent solid was determined. From this container, approximately 5.0 g (dry weight) of soils and sediments and approximately 0.5 g (dry weight) of biosolids were transferred to a polypropylene centrifuge tube and spiked with EIS compounds. Samples were vortexed to disperse the EIS standard and allowed to equilibrate for a minimum of 30 minutes. A series of three solvent extractions was performed using varying amounts of 0.3% methanolic ammonium hydroxide, resulting in one tube containing the supernatant from all three extractions. Loose ENVI-Carb® carbon was used to eliminate matrix interferences as much as possible prior to concentration to remove methanol. After the addition of reagent water, the sample extract pH was adjusted to pH 6.5 ± 0.5, if needed. Sample extracts were further cleaned using a WAX SPE cartridge. A solution of methanolic ammonium hydroxide solution was used to rinse the evaporation/concentrator tube and elute PFAS from the WAX SPE. IIS compounds were then added to the sample extract. Samples were filtered prior to analysis.

2.5.3 Tissues

The two fish samples and the clam sample were prepared by homogenizing all tissue received for each sample. Approximately 2.0 g (wet weight) of each sample was transferred to a polypropylene centrifuge tube and spiked with EIS compounds. Samples were vortexed to disperse the EIS standard and allowed to equilibrate for a minimum of 30 minutes. A solution of 0.05M potassium hydroxide in methanol was added to each sample and thoroughly mixed on a shaker table for at least 16 hours. The samples were centrifuged, and the supernatant was collected in another polypropylene centrifuge tube. The samples were extracted twice more for lesser periods of time using acetonitrile and 0.05M potassium hydroxide in methanol solution. All supernatant was collected in a single tube for each sample. Loose ENVI-Carb® carbon was used to eliminate matrix interferences as much as possible prior to concentration to remove some of the methanol. After the addition of reagent water, the pH of the sample extract was then adjusted, if needed, to obtain a pH of 6.5 ± 0.5. Sample extracts were further cleaned using a WAX SPE cartridge. A solution of methanolic ammonium hydroxide solution was used to rinse the evaporation/concentrator tube and elute PFAS from the WAX SPE. IIS compounds were then added to the sample extract. Samples were filtered prior to analysis.

2.6 DEVIATIONS FROM THE METHOD OR STUDY DESIGN

In addition to the matrix spikes required by the Study Plan, additional matrix spikes for groundwater samples were added as discussed in Section 2.5.

The Study Plan identified vegetable oil as a suitable reference material to be used in Phase 1 for tissue matrix. However, the laboratory found that lingcod (*Ophiodon elongatus*) was a clean

reference material more representative of tissue matrices; therefore, lingcod was used as the reference material for tissue in both Phase 1 and Phase 3.

The Study Plan required that Phase 1 include the verification of the LOD and LOQ in each matrices type (aqueous, solid, tissue) in accordance with QSM 5.3 requirements. After the initial analyses were conducted, it was determined that the verification scheme required by QSM 5.3 artificially elevated the LOQ in some instances. As a result, the LOD verification was eliminated from the submittal, and the reporting scheme was adjusted to report detections above the MDL, not the LOD, and to qualify detections between the MDL and LOQ with a "J" qualifier. The LOQ verifications conducted in Phase 1 were intended to be carried forward and applied to the quality assurance/quality verification in Phases 2 and 3. Instead, SGS AXYS conducted LOQ verification samples with every spiked matrix sample batch in Phases 2 and 3. Because these verifications met the QSM 5.3 requirements, they were used in lieu of the Phase 1 results. Evaluation of the LOQ verifications associated with study sample preparation batches provides greater insight into the performance of the method.

3 DATA REVIEW AND VALIDATION

Raw data and reporting forms equivalent to a hardcopy data package were submitted for data reported for all phases of the study, and electronic data deliverables (EDDs) in Excel format were submitted for Phase 2 and 3 data. Multiple data packages and EDDs were submitted for each phase of the study, and all of them were reviewed for completeness and data quality. Data were evaluated based on the preliminary performance criteria described by the method and SLVS Plan (Appendix A).

Table 3-1. Summary of Type and Number of Analyses Reviewed

Sample Type	Matrix	# Analyses[1]	# Target Analyte Results	# EIS Results	# IIS Results	Total # Results Reviewed
MDL Study (7 method blanks [MBs], 7 MDL spiked samples)	Aqueous: Reagent Water	14	560	336	98	994
MDL Study (7 MBs, 7 MDL spiked samples)	Solids: Ottawa Sand	14	560	336	98	994
MDL Study (7 MBs, 7 MDL spiked samples)	Tissue: Lingcod	14	560	336	98	994
Initial Precision and Recovery (IPR) Study (1 MB, 4 IPRs)	Aqueous: Reagent Water	5	200	120	35	355
IPR Study and LOQ Verification (1 MB, 4 IPRs)	Solids: Ottawa Sand	5	20	120	35	355
IPR Study and LOQ Verification (1 MB, 4 IPRs)	Tissue: Lingcod	5	200	120	35	355
Native Samples	Groundwater	12	480	288	84	852
Native Samples	Surface water	9	360	216	63	639
Native Samples	Wastewater	21	840	504	147	1,491
Native Samples	Landfill Leachate	9	360	216	63	639
Native Samples	Soil	21	840	504	147	1,491
Native Samples	Sediment	9	360	216	63	639
Native Samples	Biosolids	9	360	216	63	639
Native Samples	Tissue	9	360	216	63	639
Low-Level Spike	Groundwater	9	360	216	63	639
Low-Level Spike	Surface water	9	360	216	63	639

Table 3-1. Summary of Type and Number of Analyses Reviewed

Sample Type	Matrix	# Analyses[1]	# Target Analyte Results	# EIS Results	# IIS Results	Total # Results Reviewed
Low-Level Spike	Wastewater	21	840	504	147	1,491
Low-Level Spike	Landfill Leachate	9	360	216	63	639
Low-Level Spike	Soil	21	840	504	147	1,491
Low-Level Spike	Sediment	9	360	216	63	639
Low-Level Spike	Biosolids	9	360	216	63	639
Low-Level Spike	Tissue	9	360	216	63	639
Mid-Level Spike	Groundwater	9	360	216	63	639
Mid-Level Spike	Surface water	9	360	216	63	639
Mid-Level Spike	Wastewater	21	840	504	147	1,491
Mid-Level Spike	Landfill Leachate	9	360	216	63	639
Mid-Level Spike	Soil	21	840	504	147	1,491
Mid-Level Spike	Sediment	9	360	216	63	639
Mid-Level Spike	Biosolids	9	360	216	63	639
Mid-Level Spike	Tissue	9	360	216	63	639
High-Level Spike	Groundwater	9	360	216	63	639
High-Level Spike	Surface water	9	360	216	63	639
High-Level Spike	Wastewater	21	840	504	147	1,491
High-Level Spike	Landfill Leachate	9	360	216	63	639
High-Level Spike	Soil	21	840	504	147	1,491
High-Level Spike	Sediment	9	360	216	63	639
High-Level Spike	Biosolids	9	360	216	63	639
High-Level Spike	Tissue	9	360	216	63	639
High 2 Level Spike	Groundwater	9	360	216	63	639
Method Blanks	Reagent Water	22	880	528	154	1,562
Method Blanks	Ottawa Sand	17	680	408	119	1,207
Method Blanks	Lingcod	4	160	96	28	284
OPR	Reagent Water	22	880	528	154	1,562
OPR	Ottawa Sand	17	680	408	119	1,207

Table 3-1. Summary of Type and Number of Analyses Reviewed

Sample Type	Matrix	# Analyses[1]	# Target Analyte Results	# EIS Results	# IIS Results	Total # Results Reviewed
OPR	Lingcod	4	160	96	28	284
LOQVER	Reagent Water	22	880	528	154	1,562
LOQVER	Ottawa Sand	17	680	408	119	1,207
LOQVER	Lingcod	4	160	96	28	284
Initial Calibration	N/A	24	960	576	168	1,704
Calibration Verification (CV)	N/A	163	6,520	912	1,141	11,573
Instrument Sensitivity Check (ISC)	N/A	44	760	1,056	308	3,124
					Total Number of Results Reviewed	57,723

N/A = Not Applicable
[1]Number of analyses not including re-analyses due to QC failures and sample dilutions.

Each data package that was submitted was reviewed for completeness and compliance with the requirements of the Study Method (Appendix A).

4 CALIBRATION AND QUANTIFICATION

Sample extracts were analyzed by LC-MS/MS in MRM mode. The mass spectrometer underwent mass calibration to ensure the accuracy of the m/z values assigned to the instrument per the manufacturer's instructions. After the mass calibration had been verified, a multi-point initial calibration was performed using quantitative standards that included 40 target analytes, 24 EIS, and 7 IIS. Twenty-four target analytes with corresponding stable isotope analogs were quantified using isotope dilution quantitation, and 16 target analytes which did not have stable isotope analogs were quantified using an EIS quantitation approach. The IIS compound responses were used to determine the recovery of the EIS compounds. Target analytes were quantified and reported in their acid form. The calibration standards used for PFOS, PFHxS, NMeFOSAA, and NEtFOSAA included branched and linear isomers. All other analytes were calibrated using standards that only included the linear isomer of the analyte. Qualitative standards of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE were analyzed after the calibration curve to identify the retention time of the branched isomers of these analytes. If a quantitative branched/linear isomeric mixture of an analyte was used for calibration standards or a qualitative branched/linear isomeric mixture of an analyte was analyzed after the calibration curve, when detected in a sample, it was included in the quantitation of that analyte.

4.1 MASS CALIBRATION AND MASS CALIBRATION VERIFICATION

Mass calibration and mass calibration verification occurred on January 13, 2020, in accordance with the manufacturer's instructions. The instrument was calibrated for an ion mass range of 22.9898 – 1971.6149, which encompasses all of the ion masses monitored by the method. The mass calibration and mass calibration verification met the criteria of all ion masses and were within ±0.5 amu of their true value. All requirements for mass calibration and mass calibration verification stated in the SLVS Method (Appendix A) were met.

4.2 MULTI-POINT INITIAL CALIBRATION

The SLVS Method (Appendix A) outlines calibration and quantification of 40 PFAS by one of two approaches:

- True isotope dilution quantification: the method analyte response was compared with the response of its isotopically labeled analog. Twenty-four target analytes were quantified by isotope dilution.
- EIS quantification: the method analyte response was compared with the response of the isotopically labeled analog of another method analyte that was closest in chemical structure and retention time. Sixteen target analytes were quantified by EIS.

The EIS approach was utilized for 16 target analytes due to the lack of commercially available isotopically labeled analogs of those analytes during method development and validation. If isotopically labeled analogs of these analytes become available in the future, then isotope dilution quantification would be recommended as it is more accurate.

The isotopically labeled compounds (the EIS) were added before any sample preparation steps were performed. The method analyte results were corrected for any loss or apparent gains

occurring as a result of the sample preparation procedure and analytical process using the response of its associated isotopically labeled compound.

Table 4-1 provides a list of the 40 target PFAS and their associated quantification and calibration information.

Table 4-1. Quantification Reference and Calibration Approach for the Target Analytes

Target Analyte	Quantification Reference Compound (EIS)	Calibration Approach[1]
PFBA	$^{13}C_4$-PFBA	ID
PFPeA	$^{13}C_5$-PFPeA	ID
PFHxA	$^{13}C_5$-PFHxA	ID
PFHpA	$^{13}C_4$-PFHpA	ID
PFOA	$^{13}C_8$-PFOA	ID
PFNA	$^{13}C_9$-PFNA	ID
PFDA	$^{13}C_6$-PFDA	ID
PFUnA	$^{13}C_7$-PFUnA	ID
PFDoA	$^{13}C_2$-PFDoA	ID
PFTrDA	avg.$^{13}C_2$-PFTeDA and$^{13}C_2$-PFDoA	EIS
PFTeDA	$^{13}C_2$-PFTeDA	ID
PFBS	$^{13}C_3$-PFBS	ID
PFPeS	$^{13}C_3$-PFHxS	EIS
PFHxS	$^{13}C_3$-PFHxS	ID
PFHpS	$^{13}C_8$-PFOS	EIS
PFOS	$^{13}C_8$-PFOS	ID
PFNS	$^{13}C_8$-PFOS	EIS
PFDS	$^{13}C_8$-PFOS	EIS
PFDoS	$^{13}C_8$-PFOS	EIS
4:2FTS	$^{13}C_2$-4:2FTS	ID
6:2FTS	$^{13}C_2$-6:2FTS	ID
8:2FTS	$^{13}C_2$-8:2FTS	ID
PFOSA	$^{13}C_8$-PFOSA	ID
NMeFOSA	D_3-NMeFOSA	ID
NEtFOSA	D_5-NEtFOSA	ID

Table 4-1. Quantification Reference and Calibration Approach for the Target Analytes

Target Analyte	Quantification Reference Compound (EIS)	Calibration Approach[1]
NMeFOSAA	D_3-NMeFOSAA	ID
NEtFOSAA	D_5-N-EtFOSAA	ID
NMeFOSE	D_7-NMeFOSE	ID
NEtFOSE	D_9-NEtFOSE	ID
HFPO-DA	$^{13}C_3$-HFPO-DA	ID
ADONA	$^{13}C_3$-HFPO-DA	EIS
PFMPA	$^{13}C_5$-PFPeA	EIS
PFMBA	$^{13}C_5$-PFPeA	EIS
NFDHA	$^{13}C_5$-PFHxA	EIS
9Cl-PF3ONS	$^{13}C_3$-HFPO-DA	EIS
11Cl-PF3OUdS	$^{13}C_3$-HFPO-DA	EIS
PFEESA	$^{13}C_5$-PFHxA	EIS
3:3FTCA	$^{13}C_5$-PFPeA	EIS
5:3FTCA	$^{13}C_5$-PFHxA	EIS
7:3FTCA	$^{13}C_5$-PFHxA	EIS

[1] Isotope dilution (ID) and extracted internal standard (EIS)

In addition to the EIS compounds added before sample preparation, an additional seven isotopically labeled analogs were added after extraction. These seven IIS compounds (also known as NIS) were used to calculate the recoveries of the 24 EIS compounds.

Table 4-2 provides a list of the 24 EIS and their associated IIS values.

Table 4-2. EIS Compounds and Their Associated IIS Compounds

EIS Compound	Associated IIS Compound
$^{13}C_4$-PFBA	$^{13}C_3$-PFBA
$^{13}C_5$-PFPeA	$^{13}C_2$-PFHxA
$^{13}C_5$-PFHxA	$^{13}C_2$-PFHxA
$^{13}C_4$-PFHpA	$^{13}C_2$-PFHxA
$^{13}C_8$-PFOA	$^{13}C_4$-PFOA
$^{13}C_9$-PFNA	$^{13}C_5$-PFNA
$^{13}C_6$-PFDA	$^{13}C_2$-PFDA
$^{13}C_7$-PFUnA	$^{13}C_2$-PFDA
$^{13}C_2$-PFDoA	$^{13}C_2$-PFDA
$^{13}C_2$-PFTeDA	$^{13}C_2$-PFDA
$^{13}C_3$-PFBS	$^{18}O_2$-PFHxS
$^{13}C_3$-PFHxS	$^{18}O_2$-PFHxS
$^{13}C_8$-PFOS	$^{13}C_4$-PFOS
$^{13}C_2$-4:2FTS	$^{18}O_2$-PFHxS
$^{13}C_2$-6:2FTS	$^{18}O_2$-PFHxS
$^{13}C_2$-8:2FTS	$^{18}O_2$-PFHxS
$^{13}C_8$-PFOSA	$^{13}C_4$-PFOS
D_3-NMeFOSA	$^{13}C_4$-PFOS
D_5-NEtFOSA	$^{13}C_4$-PFOS
D_3-NMeFOSAA	$^{13}C_4$-PFOS
D_5-NEtFOSAA	$^{13}C_4$-PFOS
D_7-NMeFOSE	$^{13}C_4$-PFOS
D_9-NEtFOSE	$^{13}C_4$-PFOS
$^{13}C_3$-HFPO-DA	$^{13}C_2$-PFHxA

A total of three calibrations were submitted by SGS AXYS to demonstrate the applicable range of the procedure per the SLVS Plan (Appendix A). All requirements for initial calibration stated in the Study Plan were met, with one exception. The lowest concentration calibration standard included in the calibration curve analyzed on March 12, 2020, failed to meet the 70–130% recovery acceptance criteria for NMeFOSAA, yielding 65.0% of its true concentration. This calibration curve was utilized during Phase 1 data collection only. Given that this standard was significantly

lower than the LOQ, this failure had no significant impact on the data, and therefore no data were rejected as a result of this failure.

Following verification of the LOQ, the calibration range was adjusted, eliminating this lowest concentration calibration standard. The concentrations of the initial calibration standards included in the initial calibration used to calibrate the LC-MS/MS for Phases 2 and 3 are provided in Table 4-3. CAL 8 was not used for PFUnA, 4:2FTS, 6:2FTS, and 8:2FTS due to carryover of these analytes into the instrument blank analyzed immediately following this standard. This level also was eliminated for the EIS compounds associated with these analytes as well.

Table 4-3. Initial Calibration Standard Concentrations (ng/mL)

	Calibration Standard							
	1	**2**	**3**	**4 (CAL-VER)**	**5**	**6**	**7**	**8**
Perfluoroalkyl carboxylates								
PFBA	0.4	0.8	2	5.0	10	20	50	250
PFPeA	0.2	0.4	1	2.5	5	10	25	125
PFHxA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFHpA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFOA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFNA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFDA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFUnA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	NA[2]
PFDoA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFTrDA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFTA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Perfluoroalkyl sulfonates								
PFBS	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFPeS	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFHxS[1]	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFHpS	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFOS[1]	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFNS	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFDS	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFDoS	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Fluorotelomer sulfonates								
4:2FTS	0.4	0.8	2	5	10	20	50	NA[2]

Table 4-3. Initial Calibration Standard Concentrations (ng/mL)

	Calibration Standard							
	1	**2**	**3**	**4 (CAL-VER)**	**5**	**6**	**7**	**8**
6:2FTS	0.36	0.72	1.8	4.51	9.01	18.03	45.07	NA[2]
8:2FTS	0.4	0.8	2	5	10	20	50	NA[2]
Perfluorooctane sulfonamides								
PFOSA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
NMeFOSA	0.115	0.23	0.575	1.4375	2.875	5.75	12.5	62.5
NEtFOSA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Perfluorooctane sulfonamidoacetic acids								
NMeFOSAA[1]	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
NEtFOSAA [1]	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Perfluorooctane sulfonamide ethanols								
NMeFOSE	1.0	2.0	5	12.5	25	50	125	625
NEtFOSE	0.75	1.5	3.75	9.37	18.74	37.49	93.75	468.75
Per- and polyfluoroether carboxylates								
HFPO-DA	0.38	0.76	1.9	4.75	9.5	19	47.5	237.6
ADONA	0.4	0.8	2	5.0	10	20	50	250
PFMPA	0.2	0.4	1	2.5	5	10	25	215
PFMBA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
NFDHA	0.2	0.4	1	2.5	5	10	25	125
Ether sulfonates								
9Cl-PF3ONS	0.4	0.8	2	5.0	10	20	50	250
11-Cl-PF3OUdS	0.4	0.8	2	5.0	10	20	50	250
PFEESA	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Fluorotelomer carboxylates								
3:3 FTCA	0.4	0.8	2	5	10	20	50	250
5:3 FTCA	2.5	5	12.5	31.3	62.5	125	315	1560
7:3 FTCA	2.5	5	12.5	31.3	62.5	125	315	1560
Extracted Internal Standards								
$^{13}C_4$-PFBA	10	10	10	10	10	10	10	10
$^{13}C_5$-PFPeA	5	5	5	5	5	5	5	5
$^{13}C_5$-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5

Table 4-3. Initial Calibration Standard Concentrations (ng/mL)

	Calibration Standard							
	1	**2**	**3**	**4 (CAL-VER)**	**5**	**6**	**7**	**8**
$^{13}C_4$-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_8$-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_9$-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_6$-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_7$-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	NA[2]
$^{13}C_2$-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_2$-PFTA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_3$-PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_3$-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_8$-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_2$-4:2FTS	5	5	5	5	5	5	5	NA[2]
$^{13}C_2$-6:2FTS	5	5	5	5	5	5	5	NA[2]
$^{13}C_2$-8:2FTS	5	5	5	5	5	5	5	NA[2]
$^{13}C_8$-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_3-NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_3-NMeFOSAA	5	5	5	5	5	5	5	5
D_5-NEtFOSAA	5	5	5	5	5	5	5	5
D_7-NMeFOSE	25	25	25	25	25	25	25	25
D_9-NEtFOSE	25	25	25	25	25	25	25	25
$^{13}C_3$-HFPO-DA	10	10	10	10	10	10	10	10
Injection Internal Standards								
$^{13}C_3$-PFBA	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
$^{13}C_2$-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_4$-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_5$-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_2$-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{18}O_2$-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_4$-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5

[1]Calibration standard included branched and linear isomers.
[2]For an explanation of NA (not analyzed), see the preceding paragraph.

4.3 RESPONSE RATIOS AND RESPONSE FACTORS

The response ratio (RR) for each method analyte calibrated by isotope dilution was calculated according to the equation below, separately for each of the calibration standards, using the areas of the quantification ion masses shown in Table 4-4. RR was used for the 24 target analytes quantified by isotope dilution.

$$RR = \frac{Area_n\, M_l}{Area_l\, M_n}$$

where

$Area_n$ = The measured area of the quantification ion mass for the target analyte
$Area_l$ = The measured area at the quantification ion mass for the corresponding EIS added to the sample before extraction
M_l = The mass of the EIS in the calibration standard
M_n = The mass of the target analyte in the calibration standard

The response factor (RF) for each method analyte calibrated by EIS was calculated according to the equation below. RF was used for the 16 target analytes quantified by EIS.

$$RF = \frac{Area_s\, M_{EIS}}{Area_{EIS}\, M_s}$$

where

$Area_s$ = The measured area of the quantification ion mass for the target analyte
$Area_{EIS}$ = The measured area at the quantification ion mass for the EIS
M_{EIS} = The mass of the EIS in the calibration standard
M_s = The mass of the target analyte in the calibration standard

The response factor (RF_s) of each EIS was calculated for each calibration standard using the equation below. RF_s was used for the 24 EIS quantified by IIS.

$$RF_s = \frac{Area_l\, M_{NIS}}{Area_{NIS}\, M_l}$$

where

$Area_l$ = The measured area of the quantification ion mass for the EIS
$Area_{IIS}$ = The measured area of the quantification ion mass for the IIS
M_{IIS} = The mass of the IIS in the calibration standard
M_l = The mass of the EIS

4.4 ION MASS MONITORING

The LC-MS/MS device was operated under the mass spectrometer negative electrospray ionization conditions stated in the SLVS Plan (Appendix A). MRM mode was utilized to monitor the ion masses provided in Table 4-4. Where possible, two ion masses were monitored for target analytes.

The area of the more intense of the two ion masses (the quantification ion mass) was the basis for the RR and RF equations provided above. Although the confirmation ion masses were not used to calculate RRs and RFs, they were used in a qualitative manner. The confirmation ion mass must be present in a sample in order for that method analyte to be considered present. The ratio of the quantitative ion mass to the confirmation ion mass, or ion ratio, is used as an indicator of possible bias. If the ion ratio falls outside acceptance criteria based on the ion ratios observed in standards, this indicates that a higher degree of uncertainty is associated with the analyte, and therefore its quantitated value should be considered as an estimated value. Typical ion ratios observed are provided in Table 4-4.

Table 4-4. Target Analyte Ions Monitored, Extracted Internal Standards, and Non-extracted Internal Standards Used for Quantification

Abbreviation	Example Retention Time	Parent Ion Mass	Quantitative Ion Mass	Confirmation Ion Mass	Typical Ion Ratio	Quantification Reference Compound
Target Analytes						
PFBA	1.96	212.8	168.9	NA	NA	$^{13}C_4$-PFBA
PFPeA	4.18	263.0	219.0	68.9	NA	$^{13}C_5$-PFPeA
PFHxA	4.81	313.0	269.0	118.9	13	$^{13}C_5$-PFHxA
PFHpA	5.32	363.1	319.0	169.0	3.5	$^{13}C_4$-PFHpA
PFOA	6.16	413.0	369.0	169.0	3.0	$^{13}C_8$-PFOA
PFNA	6.99	463.0	419.0	219.0	4.9	$^{13}C_9$-PFNA
PFDA	7.47	512.9	469.0	219.0	5.5	$^{13}C_6$-PFDA
PFUnA	7.81	563.1	519.0	269.1	6.9	$^{13}C_7$-PFUnA
PFDoA	8.13	613.1	569.0	319.0	10	$^{13}C_2$-PFDoA
PFTrDA	8.53	663.0	619.0	168.9	6.7	avg.$^{13}C_2$-PFTeDA and$^{13}C_2$-PFDoA
PFTeDA	8.96	713.1	669.0	168.9	6.0	$^{13}C_2$-PFTeDA
PFBS	4.79	298.7	79.9	98.8	2.1	$^{13}C_3$-PFBS
PFPeS	5.38	349.1	79.9	98.9	1.8	$^{13}C_3$-PFHxS
PFHxS	6.31	398.7	79.9	98.9	1.9	$^{13}C_3$-PFHxS
PFHpS	7.11	449.0	79.9	98.8	1.7	$^{13}C_8$-PFOS
PFOS	7.59	498.9	79.9	98.8	2.3	$^{13}C_8$-PFOS
PFNS	7.92	548.8	79.9	98.8	1.9	$^{13}C_8$-PFOS
PFDS	8.28	599.0	79.9	98.8	1.9	$^{13}C_8$-PFOS
PFDoS	9.14	699.1	79.9	98.8	1.9	$^{13}C_8$-PFOS
4:2FTS	4.67	327.1	307.0	80.9	1.7	$^{13}C_2$-4:2FTS
6:2FTS	5.81	427.1	407.0	80.9	1.9	$^{13}C_2$-6:2FTS

Table 4-4. Target Analyte Ions Monitored, Extracted Internal Standards, and Non-extracted Internal Standards Used for Quantification

Abbreviation	Example Retention Time	Parent Ion Mass	Quantitative Ion Mass	Confirmation Ion Mass	Typical Ion Ratio	Quantification Reference Compound
8:2FTS	7.28	527.1	507.0	80.8	3.0	$^{13}C_2$-8:2FTS
PFOSA	8.41	498.1	77.9	478.0	47	$^{13}C_8$-PFOSA
NMeFOSA	9.70	511.9	219.0	169.0	0.66	D_3-NMeFOSA
NEtFOSA	9.94	526.0	219.0	169.0	0.63	D_5-NEtFOSA
NMeFOSAA	7.51	570.1	419.0	483.0	2.0	D_3-NMeFOSAA
NEtFOSAA	7.65	584.2	419.1	526.0	1.2	D_5-NEtFOSAA
NMeFOSE	9.57	616.1	58.9	NA	NA	D_7-NMeFOSE
NEtFOSE	9.85	630.0	58.9	NA	NA	D_9-NEtFOSE
HFPO-DA	4.97	284.9	168.9	184.9	1.95	$^{13}C_3$-HFPO-DA
ADONA	5.79	376.9	250.9	84.8	2.8	$^{13}C_3$-HFPO-DA
9Cl-PF3ONS	7.82	530.8	351.0	532.8→ 353.0	3.2	$^{13}C_3$-HFPO-DA
11Cl-PF3OUdS	8.62	630.9	450.9	632.9→ 452.9	3.0	$^{13}C_3$-HFPO-DA
PFEESA	5.08	314.8	134.9	82.9	9.22	$^{13}C_5$-PFHxA
PFMPA	3.21	229.0	84.9	NA	NA	$^{13}C_5$-PFPeA
PFMBA	4.53	279.0	85.1	NA	NA	$^{13}C_5$-PFPeA
NFDHA	4.84	295.0	201.0	84.9	1.46	$^{13}C_5$-PFHxA
3:3FTCA	3.89	241.0	177.0	117.0	1.70	$^{13}C_5$-PFPeA
5:3FTCA	5.14	341.0	237.1	217.0	1.16	$^{13}C_5$-PFHxA
7:3FTCA	6.76	441.0	316.9	336.9	0.69	$^{13}C_5$-PFHxA
Extracted Internal Standards						
$^{13}C_4$-PFBA	1.95	216.8	171.9	NA		$^{13}C_3$-PFBA
$^{13}C_5$-PFPeA	4.18	268.3	223.0	NA		$^{13}C_2$-PFHxA
$^{13}C_5$-PFHxA	4.80	318.0	273.0	120.3		$^{13}C_2$-PFHxA
$^{13}C_4$-PFHpA	5.32	367.1	322.0	NA		$^{13}C_2$-PFHxA
$^{13}C_8$-PFOA	6.16	421.1	376.0	NA		$^{13}C_4$-PFOA
$^{13}C_9$-PFNA	6.99	472.1	427.0	NA		$^{13}C_5$-PFNA
$^{13}C_6$-PFDA	7.47	519.1	474.1	NA		$^{13}C_2$-PFDA
$^{13}C_7$-PFUnA	7.81	570.0	525.1	NA		$^{13}C_2$-PFDA
$^{13}C_2$-PFDoA	8.13	615.1	570.0	NA		$^{13}C_2$-PFDA

Table 4-4. Target Analyte Ions Monitored, Extracted Internal Standards, and Non-extracted Internal Standards Used for Quantification

Abbreviation	Example Retention Time	Parent Ion Mass	Quantitative Ion Mass	Confirmation Ion Mass	Typical Ion Ratio	Quantification Reference Compound
$^{13}C_2$-PFTeDA	8.96	715.2	670.0	NA		$^{13}C_2$-PFDA
$^{13}C_3$-PFBS	4.78	302.1	79.9	98.9		$^{18}O_2$-PFHxS
$^{13}C_3$-PFHxS	6.30	402.1	79.9	98.8		$^{18}O_2$-PFHxS
$^{13}C_8$-PFOS	7.59	507.1	79.9	98.9		$^{13}C_4$-PFOS
$^{13}C_2$-4:2FTS	4.67	329.1	80.9	309.0		$^{18}O_2$-PFHxS
$^{13}C_2$-6:2FTS	5.82	429.1	80.9	409.0		$^{18}O_2$-PFHxS
$^{13}C_2$-8:2FTS	7.28	529.1	80.9	509.0		$^{18}O_2$-PFHxS
$^{13}C_8$-PFOSA	8.41	506.1	77.8	NA		$^{13}C_4$-PFOS
D_3-NMeFOSA	9.70	515.0	219.0	NA		$^{13}C_4$-PFOS
D_5-NEtFOSA	9.94	531.1	219.0	NA		$^{13}C_4$-PFOS
D_3-NMeFOSAA	7.51	573.2	419.0	NA		$^{13}C_4$-PFOS
D_5-NEtFOSAA	7.65	589.2	419.0	NA		$^{13}C_4$-PFOS
D_7-NMeFOSE	9.56	623.2	58.9	NA		$^{13}C_4$-PFOS
D_9-NEtFOSE	9.83	639.2	58.9	NA		$^{13}C_4$-PFOS
$^{13}C_3$-HFPO-DA	4.97	286.9	168.9	184.9		$^{13}C_2$-PFHxA
Injection Internal Standards						
$^{13}C_3$-PFBA	1.95	216.0	172.0	NA		
$^{13}C_2$-PFHxA	4.80	315.1	270.0	119.4		
$^{13}C_4$-PFOA	6.16	417.1	172.0	NA		
$^{13}C_5$-PFNA	6.99	468.0	423.0	NA		
$^{13}C_2$-PFDA	7.47	515.1	470.1	NA		
$^{18}O_2$-PFHxS	6.30	403.0	83.9	NA		
$^{13}C_4$-PFOS	7.59	502.8	79.9	98.9		

NA = Not analyzed

4.5 CALIBRATION LINEARITY AND STABILITY

A summary of the RRs and RFs from the three calibrations that were provided by SGS AXYS is provided in Table 4-5, including the relative standard deviation (RSD).

Table 4-5. Summary of RRs and RFs from the Three Calibrations

Abbreviation	Mean RR or RF		Mean RSD	
	Low	High	Low	High
Target Analytes				
PFBA	0.93	0.97	2.17	5.34
PFPeA	1.08	1.18	4.69	9.77
PFHxA	0.95	1.04	4.18	8.77
PFHpA	1.01	1.08	6.17	13.3
PFOA	1.37	1.50	7.4	13.0
PFNA	0.94	1.05	3.22	6.9
PFDA	0.78	0.83	2.8	10.8
PFUnA	0.72	0.8	3.57	9.65
PFDoA	0.99	1.08	5.22	11.2
PFTrDA	0.78	0.85	7.28	12.0
PFTeDA	0.69	0.75	7.19	12.2
PFBS	1.04	1.11	2.73	5.64
PFPeS	1.02	1.07	2.47	8.3
PFHxS	1.05	1.15	4.11	8.09
PFHpS	1.01	1.09	3.15	4.42
PFOS	1.09	1.12	3.26	7.02
PFNS	1.05	1.11	2.34	12.9
PFDS	1.00	1.04	3.47	6.48
PFDoS	0.87	0.89	4.87	5.78
4:2FTS	0.44	0.49	3.35	10.0
6:2FTS	0.47	0.47	3.68	14.6
8:2FTS	0.25	0.29	5.46	10.4
PFOSA	0.91	0.94	3.04	6.40
NMeFOSA	1.10	1.18	4.01	12.2
NEtFOSA	1.09	1.13	2.76	5.62
NMeFOSAA	0.91	0.93	7.51	18.4

Table 4-5. Summary of RRs and RFs from the Three Calibrations

Abbreviation	Mean RR or RF		Mean RSD	
	Low	High	Low	High
NEtFOSAA	0.67	0.79	8.36	13.1
NMeFOSE	1.03	1.06	2.44	4.72
NEtFOSE	1.11	1.16	2.37	5.82
HFPO-DA	1.05	1.09	5.55	12.3
ADONA	7.81	8.61	5.5	9.53
9Cl-PF3ONS	2.03	2.33	3.87	13.9
11Cl-PF3OUdS	1.02	1.24	5.75	11.4
PFEESA	2.87	3.12	2.84	5.19
PFMPA	1.63	2.20	5.83	11.3
PFMBA	1.63	2.18	7.78	9.61
NFDHA	0.03	0.04	10.8	19.0
3:3FTCA	0.08	0.09	10.5	13.7
5:3FTCA	0.17	0.18	2.73	4.53
7:3FTCA	0.09	0.1	3.21	6.56
EIS Compounds				
$^{13}C_4$-PFBA	1.09	1.14	0.95	2.86
$^{13}C_5$-PFPeA	0.81	0.85	4.8	6.4
$^{13}C_5$-PFHxA	0.71	0.72	1.6	4.07
$^{13}C_4$-PFHpA	3.11	3.36	4.31	5.74
$^{13}C_8$-PFOA	3.48	3.56	1.34	4.07
$^{13}C_9$-PFNA	1.08	1.13	1.55	4.26
$^{13}C_6$-PFDA	1	1.02	4.33	7.72
$^{13}C_7$-PFUnA	1.03	1.1	9.21	12.1
$^{13}C_2$-PFDoA	0.87	0.94	4.12	11.2
$^{13}C_2$-PFTeDA	0.76	0.82	3.09	3.83
$^{13}C_3$-PFBS	1.28	1.31	10.1	12.6
$^{13}C_3$-PFHxS	1.11	1.12	1.37	4.49

Table 4-5. Summary of RRs and RFs from the Three Calibrations

Abbreviation	Mean RR or RF		Mean RSD	
	Low	High	Low	High
$^{13}C_8$-PFOS	0.96	0.96	2.94	6.1
$^{13}C_2$-4:2FTS	0.4	0.47	9.59	15.4
$^{13}C_2$-6:2FTS	0.37	0.44	9.41	12.9
$^{13}C_2$-8:2FTS	0.47	0.54	10.8	13.1
$^{13}C_8$-PFOSA	1.91	2.05	5.03	9.03
D_3-NMeFOSA	0.26	0.29	6.01	10.3
D_5-NEtFOSA	0.24	0.28	2.17	7.92
D_3-NMeFOSAA	0.24	0.28	3.99	8.59
D_5-NEtFOSAA	0.22	0.26	3.11	5.68
D_7-NMeFOSE	1.8	2.11	2.33	8.44
D_9-NEtFOSE	2.19	2.71	2.4	9.8
$^{13}C_3$-HFPO-DA	0.26	0.29	10.2	10.9

4.6 QUALITATIVE STANDARDS

For this study, qualitative standards that included the branched and linear isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NEtFOSE, and NMeFOSE were required to be analyzed with every batch, prior to sample analyses, to determine the retention time of the branched isomers or isomeric groups of these analytes. The aim was to identify branched isomers in study samples through comparison of retention times with these standards; the peak response of the branched isomers was included in the quantitation of the analyte. When the branched isomers were determined to be present in study samples, the peak response in the study sample was included with the peak response of the linear isomer for the quantitation of the method analyte. Only in these instances and when branched isomers were determined to be present through comparison with quantitative standards used for calibration that contained branched isomers (PFOS, PFHxS, NMeFOSAA, and NEtFOSAA) were branched isomers included in the quantitation of target analytes. At the time of the study, qualitative standards were commercially available for only the seven target analytes listed above. Phase 2 and 3 data included analysis of the required qualitative standards at the frequency required and were used to confirm the identity of branched isomers in samples.

4.7 CALIBRATION VERIFICATION

Calibration verification (CV) standards were required to be analyzed daily, prior to analysis of samples, after every 10 study samples or less, and at the end of the analytical sequence. Target analytes and EIS compounds were required to recover within ±30% of their true value. For Phases

2 and 3, the concentration of the CV standard was that of the CAL 4 standard listed in Table 4-3. During this study, 163 CVs were analyzed. Of the 163 CVs, 17 failed to meet recovery criteria for one or more target analytes, and 14 failed to meet recovery criteria for one or more EIS compounds. Of the 6,520 method analyte results associated with CVs, 24 failed to meet recovery criteria, resulting in a failure rate of 0.4%. Of the 3,912 EIS compound results associated with CVs, 22 failed to meet recovery criteria, resulting in a failure rate of 0.6%. The method analyte with the highest failure rate was NFDHA, which failed to meet criteria seven times. Other target analytes that failed to meet recovery criteria were 6:2FTS, 11Cl-PF3OUdS, ADONA, PFDoS, 3:3FTCA, and PFMPA. In these instances, the same target analytes not detected were not reported in associated samples. When method analyte results were reported in samples, these results were included in the statistical evaluation of the sample results per the Study Plan. The EIS compounds that failed to meet recovery criteria included D_3-NMeFOSAA, D_5-NEtFOSAA, $^{13}C_8$-PFOSA, $^{13}C_2$-4:2FTS, $^{13}C_2$-6:2FTS, $^{13}C_2$-8:2FTS, D_7-NMeFOSE, and D_5-NEtFOSE. All EIS recoveries that failed exceeded the upper recovery criteria limit, except for those associated with D_7-NMeFOSE, and D_5-NEtFOSE, whose recoveries fell below the lower recovery criteria limit. Since their associated target analytes met their recovery criteria, sample data associated with these failures were not qualified. No sample results were eliminated from the study due to CV failures.

4.8 INSTRUMENT SENSITIVITY CHECK

For this study, an instrument sensitivity check (ISC) standard containing the target analytes at their LOQ concentrations was used to verify the sensitivity of the instrument. This ISC standard was required to be analyzed prior to analysis and every 12 hours thereafter during sample analysis. Target analytes and EIS compounds were required to recover within ±30% of their true value. All ISC standard criteria were met in Phase 2 and 3 with a few exceptions. In one instance, data were reported for study samples after the 12-hour window was exceeded. The retention times of the branched and linear isomers reported in the study samples analyzed outside of the 12-hour window were consistent with those reported in samples within the 12-hour window, and ion ratios were within the study acceptance criteria. Data were not qualified for this failure given that there was no apparent adverse effect on the data. Six of the 44 ISC standards analyzed failed to meet the study criteria for all method analyte recoveries, and 1 of the 44 failed to meet the study criteria for all EIS compound recoveries. The method analyte that failed most frequently was NFDHA, which failed to meet criteria four times. Other target analytes that failed to meet criteria were PFHpA, 6:2FTS, PFDoS, 11Cl-PF3OUdS, 3:3FTCA, PFEESA, PFMBA, and PFMPA. Of the 1,760 method analyte results reported for ISC standards, 14 failed to meet recovery criteria, resulting in a failure rate of 0.8%. Since data were not reported for the failed target analytes in these instances, no data were qualified as a result of these failures. Only one ISC standard failed to meet the EIS recovery criteria. In this instance, $^{13}C_2$-4:2FTS, $^{13}C_2$-6:2FTS, and $^{13}C_2$-8:2FTS exceeded the ±30% recovery criteria. Since their associated target analytes met their recovery criteria, sample data associated with these failures was not qualified. No sample results were eliminated from the study due to ISC failures.

5 PRECISION AND RECOVERY

5.1 AQUEOUS IPR RESULTS

IPR studies were performed in aqueous matrices. Four aliquots of 0.5 L of PFAS-free reagent water were spiked with all 40 target analytes and 24 EIS and 7 IIS compounds and prepared and analyzed in accordance with the requirements of the SLVS Plan (Appendix A). The spiking level is presented in Table 5-1 along with the mean percent recovery, standard deviation (SD), and RSD. Spike concentrations are provided as the concentration in the final sample extract.

Table 5-1. Aqueous IPR Results

Analyte	Spike Concentration in Final Extract (ng/L)	Mean % Recovery	SD	RSD
Target Analyte				
PFBA	16.0	97.9	4.7	4.8
PFPeA	8.0	95.4	5.3	5.5
PFHxA	4.0	92.0	8.4	9.1
PFHpA	4.0	94.3	3.9	4.1
PFOA	4.0	92.8	2.6	2.8
PFNA	4.0	95.8	4.0	4.1
PFDA	4.0	99.0	8.3	8.3
PFUnA	4.0	90.8	3.8	4.2
PFDoA	4.0	84.5	13.3	15.7
PFTrDA	4.0	95.9	7.8	8.1
PFTeDA	4.0	95.4	3.9	4.1
PFBS	4.0	98.2	6.5	6.6
PFPeS	4.0	101.4	7.0	6.9
PFHxS	4.0	98.6	4.4	4.4
PFHpS	4.0	105.0	10.7	10.2
PFOS	4.0	107.7	7.2	6.7
PFNS	4.0	102.3	19.2	18.8
PFDS	4.0	98.0	11.5	11.7
PFDoS	4.0	94.2	10.0	10.6
4:2FTS	16.0	99.3	11.9	12.0

Table 5-1. Aqueous IPR Results

Analyte	Spike Concentration in Final Extract (ng/L)	Mean % Recovery	SD	RSD
6:2FTS	14.4	109.9	19.3	17.5
8:2FTS	16.0	97.3	5.9	6.1
PFOSA	4.0	98.5	4.4	4.4
NMeFOSA	4.6	84.0	3.0	3.6
NEtFOSA	10.0	88.0	4.4	5.0
NMeFOSAA	4.0	98.5	8.1	8.2
NEtFOSAA	4.0	99.6	10.2	10.3
NMeFOSE	40.0	94.6	3.7	3.9
NEtFOSE	30.0	95.4	4.5	4.7
HFPO-DA	15.2	101.2	6.6	6.5
ADONA	16.0	91.4	7.2	7.9
PFEESA	4.0	92.2	6.1	6.6
PFMPA	8.0	96.0	5.0	5.2
PFMBA	4.0	92.3	15.0	16.3
NFDHA	8.0	96.5	26.4	27.4
9Cl-PF3ONS	16.0	92.7	4.2	4.5
11Cl-PF3OUdS	16.0	87.4	3.8	4.4
3:3FTCA	16.0	93.8	4.7	5.0
5:3FTCA	100.0	92.7	4.3	4.6
7:3FTCA	100.0	90.2	6.3	7.0
Extracted Internal Standard (EIS) Compounds				
$^{13}C_4$-PFBA	80.0	87.8	1.4	1.6
$^{13}C_5$-PFPEA	40.0	91.0	2.2	2.4
$^{13}C_5$-PFHXA	20.0	88.6	1.7	1.9
$^{13}C_4$-PFHPA	20.0	88.9	5.5	6.2
$^{13}C_8$-PFOA	20.0	87.2	5.2	6.0
$^{13}C_9$-PFNA	10.00	88.8	3.4	3.8
$^{13}C_6$-PFDA	10.00	89.3	4.2	4.7

Table 5-1. Aqueous IPR Results

Analyte	Spike Concentration in Final Extract (ng/L)	Mean % Recovery	SD	RSD
$^{13}C_7$-PFUNA	10.00	91.9	4.1	4.4
$^{13}C_2$-PFDOA	10.00	82.0	10.6	12.9
$^{13}C_2$-PFTEDA	10.00	80.6	4.3	5.4
$^{13}C_3$-PFBS	20.0	90.4	1.8	2.0
$^{13}C_3$-PFHXS	20.0	86.1	1.6	1.9
$^{13}C_8$-PFOS	20.0	84.9	3.3	3.9
^{13}C 4:2 FTS	40.0	85.0	10.3	12.1
^{13}C 6:2 FTS	40.0	97.5	2.2	2.2
^{13}C 8:2 FTS	40.0	103.8	2.6	2.5
$^{13}C_8$-PFOSA	20.0	83.2	11.9	14.2
D_3-NMEFOSA	20.0	69.8	7.6	10.8
D_5-NETFOSA	20.0	72.7	9.3	12.9
D_3-MEFOSAA	40.0	90.4	13.5	14.9
D_5-ETFOSAA	40.0	90.2	12.3	13.7
D_7-NMeFOSE	200	83.6	11.3	13.6
D_9-NEtFOSE	200	85.3	11.3	13.2
$^{13}C_3$-HFPO-DA	80.0	97.3	4.4	4.5

All target analyte mean percent recoveries fell between the study interim acceptance criteria of 70–130%, falling between 84.0 and 109.9%, and the RSDs were between 2.8 and 27.4%. All EIS compound mean recoveries fell between 69.8 and 103.8%, and the RSDs were between 1.6 and 14.9%.

5.2 SOLID IPR RESULTS

IPR studies in solid matrices were performed using four aliquots of 2.5 g of Ottawa sand which were spiked with all 40 target analytes and 24 EIS and 7 IIS compounds and prepared and analyzed in accordance with the requirements of the study method and Study Plan. The spiking level is presented in Table 5-2 along with the mean percent recovery, SD, and RSD. Spike concentrations are provided as the concentration in the final sample extract.

Table 5-2. Solids IPR Results

Analyte	Spike Concentration in final extract (ng/g)	Mean % Recovery	SD	RSD
Target Analytes				
PFBA	4.00	97.3	1.0	1.0
PFPeA	2.00	98.3	3.3	3.4
PFHxA	1.00	97.0	2.2	2.2
PFHpA	1.00	98.0	2.2	2.2
PFOA	1.00	95.8	1.9	2.0
PFNA	1.00	96.8	2.6	2.7
PFDA	1.00	100.3	1.5	1.5
PFUnA	1.00	99.3	3.9	4.0
PFDoA	1.00	96.8	11.7	12.1
PFTrDA	1.00	101.3	5.3	5.2
PFTeDA	1.00	98.8	2.5	2.5
PFBS	1.00	96.8	3.1	3.2
PFPeS	1.00	94.8	4.0	4.3
PFHxS	1.00	102.3	2.1	2.0
PFHpS	1.00	95.8	4.2	4.4
PFOS	1.00	101.5	3.4	3.4
PFNS	1.00	104.5	3.1	3.0
PFDS	1.00	92.5	4.8	5.2
PFDoS	1.00	87.3	5.7	6.5
4:2FTS	4.00	99.3	0.5	0.5
6:2FTS	3.61	108.5	7.0	6.5
8:2FTS	4.00	118.5	4.5	3.8
PFOSA	1.00	99.0	3.4	3.4
NMeFOSA	1.15	95.8	4.2	4.4
NEtFOSA	2.50	100.3	1.0	1.0
NMeFOSAA	1.00	98.8	3.9	4.0
NEtFOSAA	1.00	105.0	1.6	1.6

Table 5-2. Solids IPR Results

Analyte	Spike Concentration in final extract (ng/g)	Mean % Recovery	SD	RSD
NMeFOSE	10.00	100.5	1.3	1.3
NEtFOSE	7.50	100.5	1.9	1.9
HFPO-DA	3.80	93.8	5.6	5.9
ADONA	4.00	90.5	2.9	3.2
PFEESA	1.0	98.3	4.0	4.1
PFMPA	2.0	94.5	1.7	1.8
PFMBA	1.0	92.5	2.4	2.6
NFDHA	2.0	77.8	12.6	16.2
9Cl-PF3ONS	4.0	91.8	4.0	4.4
11Cl-PF3OUdS	4.0	90.0	2.9	3.3
3:3FTCA	4.0	86.3	3.3	3.8
5:3FTCA	25.0	91.5	3.0	3.3
7:3FTCA	25.0	88.8	2.8	3.1
Extracted Internal Standard (EIS) Compounds				
$^{13}C_4$-PFBA	8.0	95.4	1.5	1.6
$^{13}C_5$-PFPEA	4.0	95.8	5.1	5.3
$^{13}C_5$-PFHXA	2.0	91.8	4.4	4.8
$^{13}C_4$-PFHPA	2.0	94.7	3.9	4.1
$^{13}C_8$-PFOA	2.0	94.9	3.0	3.2
$^{13}C_9$-PFNA	1.0	93.3	3.8	4.1
$^{13}C_6$-PFDA	1.0	90.2	5.4	6.0
$^{13}C_7$-PFUNA	1.0	93.8	5.0	5.4
$^{13}C_2$-PFDOA	1.0	81.6	5.8	7.1
$^{13}C_2$-PFTEDA	1.0	85.3	1.3	1.5
$^{13}C_3$-PFBS	2.0	100.8	1.9	1.8
$^{13}C_3$-PFHXS	2.0	94.4	1.3	1.4
$^{13}C_8$-PFOS	2.0	97.0	4.8	4.9
^{13}C 4:2 FTS	4.0	133.6	0.8	0.6

Table 5-2. Solids IPR Results

Analyte	Spike Concentration in final extract (ng/g)	Mean % Recovery	SD	RSD
^{13}C 6:2 FTS	4.0	123.5	2.8	2.3
^{13}C 8:2 FTS	4.0	109.2	6.6	6.1
$^{13}C_8$-PFOSA	2.0	77.8	4.2	5.4
D_3-NMeFOSA	2.0	52.9	2.9	5.4
D_5-NEtFOSA	2.0	47.0	2.1	4.5
D_3-MeFOSAA	4.0	102.7	2.1	2.1
D_5-ETFOSAA	4.0	101.0	1.3	1.3
D_7-NMeFOSE	20.0	55.4	2.9	5.1
D_9-NEtFOSE	20.0	51.2	2.8	5.5
$^{13}C_3$-HFPO-DA	8.0	103.2	2.4	2.4

All target analyte mean percent recoveries fell within the study interim acceptance criteria of 70–130% (actual recoveries between 77.8 and 118.5%), and the RSDs were between 0.5 and 16.2%. All EIS compound mean recoveries fell between the study interim acceptance criteria of 50–200% with the exception of D_5-NEtFOSA, which had a recovery of 47.0%. This low recovery was consistent with the recovery of this isotopically labeled analog in the MDL study samples (see Section 6). The RSDs for D_5-NEtFOSA ranged from 0.6 to 7.1%.

5.3 TISSUE IPR RESULTS

IPR studies in tissue matrices were performed using four aliquots of 2.0 g of lingcod which were spiked with all 40 target analytes and 24 EIS and 7 IIS compounds and prepared and analyzed in accordance with the requirements of the Study Plan. The spiking levels are presented in Table 5-3 along with the mean percent recovery, SD, and RSD. Spike concentrations are provided as the concentration in the final sample extract.

All target analyte mean percent recoveries fell between the study interim acceptance criteria of 70–130% with the exception of NMeFOSE (181.7%) and NFDHA (67.5%). The RSDs ranged from 2.4 to 30.3%. All EIS compound mean recoveries fell within the study interim acceptance criteria of 50–200% with the exception of ^{13}C 8:2FTS (239.2%), D_3-NMeFOSA (38.7%), D_5-NEtFOSA (42.8%), D_7-NMeFOSE (4.4%), and D_9-NEtFOSE (20.9%). These recoveries were consistent with the recovery of this isotopically labeled analog in the MDL study samples (see Section 6).

Table 5-3. Tissue IPR Results

Analyte	Spike Concentration in final extract (ng/g)	Mean % Recovery	SD	RSD
Target Analyte				
PFBA	4.00	96.1	3.8	3.9
PFPeA	2.00	89.4	4.5	5.0
PFHxA	1.00	91.1	9.3	10.2
PFHpA	1.00	94.2	3.8	4.0
PFOA	1.00	81.5	2.0	2.4
PFNA	1.00	97.4	6.1	6.3
PFDA	1.00	95.9	9.8	10.2
PFUnA	1.00	92.8	4.7	5.1
PFDoA	1.00	94.0	5.3	5.7
PFTrDA	1.00	102.9	5.5	5.3
PFTeDA	1.00	89.8	6.7	7.4
PFBS	1.00	87.1	9.0	10.3
PFPeS	1.00	86.3	4.7	5.4
PFHxS	1.00	91.2	4.8	5.3
PFHpS	1.00	92.7	7.8	8.4
PFOS	1.00	105.0	3.4	3.2
PFNS	1.00	76.2	5.7	7.5
PFDS	1.00	88.0	3.1	3.6
PFDoS	1.00	84.4	5.9	6.9
4:2FTS	4.00	95.6	15.0	15.6
6:2FTS	3.61	91.1	7.1	7.8
8:2FTS	4.00	107.1	20.7	19.3
PFOSA	1.00	104.1	5.9	5.7
NMeFOSA	1.15	90.5	4.9	5.5
NEtFOSA	2.50	93.8	10.0	10.7
NMeFOSAA	1.00	112.9	11.8	10.4
NEtFOSAA	1.00	84.2	15.4	18.3
NMeFOSE	10.00	181.7	55.1	30.3

Table 5-3. Tissue IPR Results

Analyte	Spike Concentration in final extract (ng/g)	Mean % Recovery	SD	RSD
NEtFOSE	7.50	115.1	9.2	8.0
HFPO-DA	3.80	86.9	6.8	7.8
ADONA	4.00	88.8	3.3	3.8
PFEESA	1.0	83.9	7.8	9.3
PFMPA	2.0	85.5	3.6	4.2
PFMBA	1.0	89.0	7.5	8.4
NFDHA	2.0	67.5	9.3	13.8
9Cl-PF3ONS	4.0	83.5	7.2	8.7
11Cl-PF3OUdS	4.0	92.5	4.0	4.3
3:3FTCA	4,0	80.0	7.2	9.0
5:3FTCA	25.0	113.3	9.0	7.9
7:3FTCA	25.0	97.4	6.6	6.7
Extracted Internal Standard (EIS) Compounds				
$^{13}C_4$-PFBA	20.0	95.2	0.9	1.0
$^{13}C_5$-PFPEA	10.0	96.7	5.8	6.0
$^{13}C_5$-PFHXA	5.0	95.2	8.1	8.5
$^{13}C_4$-PFHPA	5.0	90.3	1.2	1.3
$^{13}C_8$-PFOA	5.0	94.5	1.6	1.7
$^{13}C_9$-PFNA	1.0	97.1	3.2	3.3
$^{13}C_6$-PFDA	1.0	96.4	3.9	4.0
$^{13}C_7$-PFUNA	1.0	101.1	8.5	8.4
$^{13}C_2$-PFDOA	1.0	110.2	7.5	6.8
$^{13}C_2$-PFTEDA	1.0	97.6	8.3	8.5
$^{13}C_3$-PFBS	5.0	100.4	6.6	6.5
$^{13}C_3$-PFHXS	5.0	94.9	1.3	1.4
$^{13}C_8$-PFOS	5.0	89.7	1.4	1.6
^{13}C 4:2 FTS	10.0	163.8	28.8	17.6
^{13}C 6:2 FTS	10.0	110.8	12.0	10.8
^{13}C 8:2 FTS	10.0	239.2	29.9	12.5

Table 5-3. Tissue IPR Results

Analyte	Spike Concentration in final extract (ng/g)	Mean % Recovery	SD	RSD
$^{13}C_8$-PFOSA	5.0	128.6	12.1	9.4
D_3-NMeFOSA	5.0	38.7	9.5	24.5
D_5-NEtFOSA	5.0	42.8	6.5	15.2
D_3-MeFOSAA	10.0	144.5	21.3	14.7
D_5-ETFOSAA	10.0	196.6	9.5	4.9
D_7-NMeFOSE	50.0	4.4	0.5	11.6
D_9-NEtFOSE	50.0	20.9	6.3	30.0
$^{13}C_3$-HFPO-DA	20.0	96.4	4.7	4.9

6 METHOD DETECTION LIMITS

As part of Phase 1, the laboratory determined the MDLs for all 40 target analytes in aqueous, solids, and tissue matrices. The MDLs were determined following the 2017 revision of the MDL procedure codified at 40 CFR 136 (Appendix B).

6.1 AQUEOUS MDL DETERMINATIONS

For the determination of aqueous MDLs, PFAS-free reagent water was used to prepare seven method blank replicates spiked with the 24 EIS and 7 IIS compounds and seven samples s spiked with the 40 analytes, EIS, and IIS compounds. All blanks and samples were prepared per the draft study method, in at least three batches on three separate calendar dates and analyzed on three separate calendar dates. The EIS and IIS compounds were spiked at the same concentrations as the ICAL standards. MDL based on method blanks (MDL_b) and spiked samples (MDL_S) were calculated by the laboratory following data review, and an initial MDL was determined as the higher of these two values (Table 6-1).

The preliminary acceptance criterion for EIS recovery stated in the SLVS Plan was 50–200% recovery. All EIS compounds met this criterion for all analyses.

Table 6-1. Aqueous Method Detection Limit Study Results

Target Analyte	MDL_S (ng/L)	MDL_b (ng/L)	Initial MDL (ng/L)
PFBA	0.330	0.249	0.330
PFPeA	0.184	0.196	0.196
PFHxA	0.318	0.118	0.318
PFHpA	0.221	0.146	0.221
PFOA	0.211	0.302	0.302
PFNA	0.221	0.082	0.221
PFDA	0.333	0.084	0.333
PFUnA	0.264	0.104	0.264
PFDoA	0.379	0.073	0.379
PFTrDA	0.238	0.137	0.238
PFTeDA	0.264	0.228	0.264
PFBS	0.245	0.048	0.245
PFPeS	0.204	0.011	0.204
PFHxS	0.217	0.118	0.217
PFHpS	0.137	0.008	0.137
PFOS	0.327	0.118	0.327
PFNS	0.303	0.025	0.303

Table 6-1. Aqueous Method Detection Limit Study Results

Target Analyte	MDL_S (ng/L)	MDL_b (ng/L)	Initial MDL (ng/L)
PFDS	0.334	0.039	0.334
PFDoS	0.179	0.052	0.179
4:2FTS	2.281	0.056	2.281
6:2FTS	3.973	0.848	3.973
8:2FTS	1.566	0.036	1.566
PFOSA	0.227	0.200	0.227
NMeFOSA	0.196	0.045	0.196
NEtFOSA	0.585	0.102	0.585
NMeFOSAA	0.586	0.029	0.586
NEtFOSAA	0.324	0.000	0.328
NMeFOSE	1.191	1.072	1.191
NEtFOSE	0.914	1.022	0.914
HFPO-DA	0.406	0.000	0.406
ADONA	0.779	0.045	0.779
PFEESA	0.137	0.000	0.137
PFMPA	0.177	0.028	0.177
PFMBA	0.117	0.026	0.117
NFDHA	1.384	0.000	1.384
9Cl-PF3ONS	0.871	0.037	0.871
11Cl-PF3OUdS	0.819	0.086	0.819
3:3FTCA	0.721	0.000	0.721
5:3FTCA	5.066	2.913	5.066
7:3FTCA	5.942	1.221	5.942

6.2 SOIL/SEDIMENT AND BIOSOLIDS MDL DETERMINATIONS

For the determination of solid MDLs, wetted Ottawa sand was used to prepare seven method blank replicates spiked with the EIS and IIS compounds and seven replicates spiked with the 40 target analytes, EIS and IIS compounds. Replicates were prepared per the SLVS Method, in at least three batches on three separate calendar dates and analyzed on three separate calendar dates. The EIS and IIS compounds were spiked at the same concentrations as the ICAL standards. MDL based on method blanks (MDL_b) and spiked samples (MDL_S) were calculated by the laboratory following data review, and an initial MDL was determined to be the higher of these two values (Table 6-2).

Table 6-2. Solids Method Detection Limit Study Results

Target Analyte	MDL_S (ng/g)	MDL_b (ng/g)	Initial MDL (ng/g)
PFBA	0.307	0.401	0.401
PFPeA	0.015	0.021	0.021
PFHxA	0.020	0.012	0.020
PFHpA	0.029	0.016	0.029
PFOA	0.029	0.037	0.037
PFNA	0.031	0.086	0.086
PFDA	0.031	0.000	0.031
PFUnA	0.032	0.033	0.033
PFDoA	0.059	0.010	0.059
PFTrDA	0.038	0.013	0.038
PFTeDA	0.032	0.021	0.032
PFBS	0.014	0.003	0.014
PFPeS	0.015	0.000	0.015
PFHxS	0.018	0.013	0.018
PFHpS	0.057	0.000	0.057
PFOS	0.067	0.024	0.067
PFNS	0.046	0.000	0.046
PFDS	0.040	0.000	0.040
PFDoS	0.038	0.001	0.038
4:2FTS	0.282	0.001	0.282
6:2FTS	0.116	0.035	0.116
8:2FTS	0.225	0.017	0.225
PFOSA	0.025	0.068	0.068
NMeFOSA	0.049	0.016	0.049
NEtFOSA	0.038	0.032	0.038
NMeFOSAA	0.030	0.007	0.030
NEtFOSAA	0.044	0.015	0.044
NMeFOSE	0.108	0.203	0.203
NEtFOSE	0.058	0.247	0.247

Table 6-2. Solids Method Detection Limit Study Results

Target Analyte	MDL_S (ng/g)	MDL_b (ng/g)	Initial MDL (ng/g)
HFPO-DA	0.136	0.003	0.136
ADONA	0.057	0.001	0.057
PFEESA	0.018	0.000	0.018
PFMPA	0.033	0.002	0.033
PFMBA	0.029	0.002	0.029
NFDHA	0.084	0.000	0.084
9Cl-PF3ONS	0.038	0.000	0.038
11Cl-PF3OUdS	0.071	0.000	0.071
3:3FTCA	0.060	0.000	0.060
5:3FTCA	0.348	0.363	0.363
7:3FTCA	0.308	0.082	0.308

The preliminary acceptance criterion for EIS recovery stated in the Study Plan was 50–200% recovery. All EIS compounds met this criterion for all analyses with the exception of the EIS compounds listed in Table 6-3.

Table 6-3. EIS Preliminary Acceptance Criteria Exceedances for Solids Method Detection Limit Study Results

EIS Compounds	Lowest % Recovery	Highest % Recovery
D_3-NMeFOSA	35	55
D_5-NEtFOSA	31	52
D_7-NMeFOSE	46	76
D_9-NEtFOSE	47	86

6.3 TISSUE MDL DETERMINATIONS

For the determination of tissue MDLs, PFAS-free fish tissue (lingcod, *Ophiodon elongatus*) was used to prepare seven method blank replicates spiked with the EIS and IIS compounds and seven replicates spiked with the 40 target analytes, EIS and IIS compounds. The replicates were prepared per the draft study method in at least three batches on three separate calendar dates and analyzed on three separate calendar dates. The EIS and IIS compounds were spiked at the same concentration as the ICAL standards. MDL based on method blanks (MDL_b) and spiked samples (MDL_S) were calculated by the laboratory following data review, and an initial MDL was determined as the higher of these two values (Table 6-4).

Table 6-4. Tissue Method Detection Limit Study Results

Target Analyte	MDL_S (ng/g)	MDL_b (ng/g)	Final MDL (ng/g)
PFBA	0.456	0.593	0.593
PFPeA	0.083	0.068	0.068
PFHxA	0.066	0.096	0.096
PFHpA	0.088	0.036	0.088
PFOA	0.056	0.086	0.086
PFNA	0.097	0.160	0.097
PFDA	0.124	0.035	0.124
PFUnA	0.152	0.137	0.152
PFDoA	0.130	0.039	0.130
PFTrDA	0.086	0.079	0.086
PFTeDA	0.185	0.034	0.185
PFBS	0.070	0.003	0.070
PFPeS	0.032	0.000	0.032
PFHxS	0.083	0.032	0.083
PFHpS	0.043	0.000	0.043
PFOS	0.294	0.158	0.294
PFNS	0.114	0.000	0.114
PFDS	0.101	0.010	0.101
PFDoS	0.177	0.001	0.177
4:2FTS	0.740	0.000	0.740
6:2FTS	0.718	1.149	0.718
8:2FTS	0.373	0.022	0.373
PFOSA	0.070	0.094	0.070
NMeFOSA	0.161	0.037	0.161
NEtFOSA	0.169	0.028	0.169
NMeFOSAA	0.093	0.014	0.093
NEtFOSAA	0.124	0.138	0.138
NMeFOSE	9.977	1.300	9.977
NEtFOSE	1.500	0.484	1.500

Table 6-4. Tissue Method Detection Limit Study Results

Target Analyte	MDL_S (ng/g)	MDL_b (ng/g)	Final MDL (ng/g)
HFPO-DA	0.161	0.006	0.161
ADONA	0.081	0.082	0.082
PFEESA	0.045	0.000	0.045
PFMPA	0.070	0.010	0.070
PFMBA	0.069	0.016	0.069
NFDHA	0.294	0.000	0.294
9Cl-PF3ONS	0.152	0.000	0.152
11Cl-PF3OUdS	0.312	0.000	0.312
3:3FTCA	0.247	0.000	0.247
5:3FTCA	1.537	0.946	1.537
7:3FTCA	0.845	0.284	0.845

The preliminary acceptance criterion for EIS recovery stated in the Study Plan was 50–200% recovery. All EIS compounds met this criterion for all analyses with the exception of the EIS compounds listed in Table 6-5.

Table 6-5. EIS Preliminary Acceptance Criteria Exceedances in Soil, Sediment, and Biosolids Method Detection Limit Study Results

EIS Compounds	Lowest % Recovery	Highest % Recovery
^{13}C-8:2FTS	121	267
D_3-NMeFOSA	19	69
D_5-NEtFOSA	18	69
D_3-NMeFOSAA	107	213
D_5-NEtFOSAA	146	256
D_7-NMeFOSE	1	27
D_9-NEtFOSE	9	71

7 METHOD BLANK ANALYSES

Method blanks are included in the procedure to evaluate the potential for background contamination introduced during sample preparation in the laboratory. Reagent water, Ottawa sand, and lingcod were used to prepare method blanks for aqueous, solid, and tissue samples, respectively. A method blank was included in every preparatory batch in this study. The target analytes 6:2FTS, PFHxA, and PFPeA were those most frequently detected above the MDL in the method blanks (Table 7-1). If the concentration of the method blank was >MDL but <LOQ, the analyte was qualified in the method blank with a "J" data qualifier.

If the concentration of the analyte in the method blank was >½ the LOQ and <10 times the concentration of the analyte in associated samples, a "B" data qualifier was appended to the result for that analyte in the sample. In only five instances was the concentration of a method analyte in the method blank >½ the LOQ and <10 times the concentration of the analyte in associated samples; four were associated with aqueous samples and one with solid samples. In all instances, the method analyte that exceeded these limits was 6:2FTS. The concentration detected in these method blanks exceeded the LOQ in three of these instances. In two of the three instances, the method blank was associated with the preparation of aqueous samples that contained very high concentrations of 6:2FTS that resulted in the sample extract requiring dilution in order to bring the concentration into the quantification range. These high concentration samples are most likely the cause of the 6:2FTS contamination found in these method blanks. Method blank contamination resulted in the "B" qualification of 23 aqueous study sample results and 12 soil study sample results. Thus, these measured concentrations were only sufficient to warrant "B" flags for what ultimately represented <0.3% of the final data set. The method blanks demonstrate that any bias associated with background contamination introduced during sample preparation was negligible.

Table 7-1. Blank Detection Frequencies for the Various Matrices Evaluated

Target Analyte	Blank Detection Frequency (%)		
	Regent Water (*n* = 22)	Ottawa Sand (*n* = 17)	Lingcod (*n* = 4)
11Cl-PF3OUdS	5	0	0
6:2FTS	18	47	0
MeFOSE	5	0	0
NEtFOSE	5	0	0
PFBA	50	0	0
PFDoS	5	0	0
PFHxA	0	18	25
PFOS	5	0	0
PFOSA	27	0	50
PFPeA	64	47	0
PFPeS	0	0	50
PFTrDA	0	0	50

8 LIMIT OF QUANTITATION VERIFICATION ANALYSES

Limit of quantitation verification (LOQVER) samples were prepared with each preparation batch along with a method blank and OPR samples. The Study Plan specified that the LOQVER should consist of a reference matrix spiked with all the target analytes at a concentration of one to two times their LOQs, plus the EIS and IIS compounds, and that was carried through the entire analytical process. The LOQVER samples prepared in this study were spiked at a concentration two times the LOQ. The reference matrices used for the LOQVER were reagent water, wetted Ottawa sand, and lingcod, for the aqueous, solid, and tissue samples, respectively.

LOQVER spike recovery tables were generated for aqueous media (groundwater, surface water, wastewater, and landfill leachate), solids (sediment, soil, and biosolids), and tissues (fish and clam. Compilation and analysis of the data were done by the IDA; the methods are detailed in Appendix B.

Appendix I presents the results. Each table includes the calculated mean of the LOQVER subsample percent recoveries and % RSD, SD, 2× SD, and 3× SD for the target analytes and EIS compounds from the 32 samples for aqueous and solid matrices (including biosolids, sediment, and soil, but excluding tissue). A separate table is included with the calculated mean of the LOQVER subsample percent recoveries, % RSD, SD, 2× SD, and 3× SD for target analytes and EIS compounds from the tissue matrix. All LOQVER percent recovery values in the EDD were used except for those values qualified with a "B" data qualifier, indicating that the method blank associated with the LOQVER reported a value >½ LOQ for that analyte.

Table 8-1 presents a summary of the findings reported in Appendix I. The percent recoveries were evaluated relative to the interim recovery limits of 70–130% as specified in the Study Plan. In addition, the recoveries also were evaluated relative to the EPA Safe Drinking Water Act lowest concentration minimum reporting level (LCMRL) verification criterion of 50–150% recovery. The interim acceptance criterion for EIS recoveries is 50–200% recovery as specified in the Study Plan.

8.1 AQUEOUS LOQVER RESULTS

The aqueous study samples were prepared and analyzed in a total of 22 preparatory batches. One LOQVER sample was included in each of these batches, as required. For the aqueous target analytes, all LOQVER mean percent recovery were within the interim recovery limits of 70–130%, as well as the 50 – 150% LCMRL verification criteria. For all EIS compounds, the mean percent recovery was between 50 – 200% recovery as specified in the Study Plan.

8.2 SOLID LOQVER RESULTS

The solid study samples were prepared and analyzed in a total of 17 preparatory batches. One LOQVER sample was included in each of these batches, as required. For the target analytes, all LOQVER mean percent recovery were within the interim recovery limits of 70–130%, as well as the 50 – 150% LCMRL verification criteria.

Two EIS mean percent recoveries failed to meet the interim acceptance criteria for EIS recoveries of 50–200%. These failures were associated with D_3-NMeFOSAA and D_5-NEtFOSAA. It is

worth noting that D_7-NMeFOSE and D_9-NEtFOSE were only slightly above the lower 50% acceptance criteria.

8.3 TISSUE LOQVER RESULTS

The tissue study samples were prepared and analyzed in a total of four preparatory batches. One LOQVER sample was included in each of these batches, as required. All target analytes were within the 70 – 130% interim recovery limits, with two exceptions. PFDoS at 61.35% mean recovery was below the interim recovery limits but was within the LCMRL verification criteria limits. MeFOSE at 237.5% mean recovery exceeded the Study Plan upper limit of 130%, as well as the upper LCMRL limit.

Seven EIS mean percent recoveries exceeded the interim acceptance criteria. Mean percent recoveries of $^{13}C_2$-PFTeDA, D_3-NMeFOSAA, D_5-NEtFOSAA, D_7-NMeFOSE, and D_9-NEtFOSE were below the lower limit. The upper limit was exceeded for $^{13}C_2$-4:2FTS and D_5-NEtFOSAA which exceeded the upper limit.

Table 8-1. Matrices LOQVER Spike Recoveries

Compound	Acronym	Mean % Recovery		
		Aqueous Samples	Solid Samples	Tissue Samples
PFAS Target Analytes				
Perfluorobutanoic acid	PFBA	105.08	100.96	104.45
Perfluoropentanoic acid	PFPeA	109.19	100.44	108.62
Perfluorohexanoic acid	PFHxA	104.86	97.24	104.90
Perfluoroheptanoic acid	PFHpA	103.90	97.36	102.15
Perfluorooctanoic acid	PFOA	106.84	98.34	103.05
Perfluorononanoic acid	PFNA	104.36	98.82	106.25
Perfluorodecanoic acid	PFDA	105.48	100.06	104.12
Perfluoroundecanoic acid	PFUnA	103.11	97.56	118.25
Perfluorododecanoic acid	PFDoA	101.98	98.55	101.95
Perfluorotridecanoic acid	PFTrDA	107.77	102.25	126.00
Perfluorotetradecanoic acid	PFTA	103.91	100.28	105.67
Perfluorobutanesulfonic acid	PFBS	104.04	100.66	103.25
Perfluoropentanesulfonic acid	PFPeS	102.25	97.71	101.38
Perfluorohexanesulfonic acid	PFHxS	112.50	103.94	111.00
Perfluoroheptanesulfonic acid	PFHpS	99.92	93.24	98.47
Perfluorooctanesulfonic acid	PFOS	108.39	100.27	126.75
Perfluorononanesulfonic acid	PFNS	102.84	88.45	102.28
Perfluorodecanesulfonic acid	PFDS	96.65	91.68	96.55

Table 8-1. Matrices LOQVER Spike Recoveries

Compound	Acronym	Mean % Recovery		
		Aqueous Samples	Solid Samples	Tissue Samples
Perfluorododecanesulfonic acid	PFDoS	88.30	85.00	61.35
4:2 fluorotelomersulfonic acid	4:2FTS	106.14	96.69	99.38
6:2 fluorotelomersulfonic acid	6:2FTS	118.93	128.41	116.75
8:2 fluorotelomersulfonic acid	8:2FTS	115.92	113.05	128.00
Perfluorooctanesulfonamide	PFOSA	107.30	104.66	116.75
N-methyl perfluorooctanesulfonamide	NMeFOSA	78.96	95.91	99.23
N-ethyl perfluorooctanesulfonamide	NEtFOSA	79.02	99.75	109.83
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	101.66	92.45	87.55
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	103.10	99.38	107.35
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	99.43	97.56	237.25
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	99.22	100.80	125.50
Hexafluoropropylene oxide dimer acid	HFPO-DA	102.38	95.55	95.00
4,8-dioxa-3H-perfluorononanoic acid	ADONA	98.20	92.54	109.42
Perfluoro-3-methoxypropanoic acid	PFMPA	103.02	96.12	94.62
Perfluoro-4-methoxybutanoic acid	PFMBA	102.37	95.32	102.05
Perfluoro-3,6-dioxaheptanoic acid	NFDHA	100.44	90.95	92.80
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	101.63	97.06	113.50
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	95.93	94.19	117.00
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	104.70	93.18	96.72
2H, 2H, 3H, 3H-perfluorohexanoic acid	3:3FTCA	94.36	88.01	81.53
2H, 2H, 3H, 3H-perfluorooctanoic acid	5:3FTCA	99.81	86.91	136.07

Table 8-1. Matrices LOQVER Spike Recoveries

Compound	Acronym	Mean % Recovery		
		Aqueous Samples	Solid Samples	Tissue Samples
2H, 2H, 3H, 3H-perfluorodecanoic acid	7:3FTCA	101.45	86.06	117.00
EIS Compounds				
Perfluoro-n-[$^{13}C_4$]butanoic acid	$^{13}C_4$ -PFBA	97.32	102.26	98.23
Perfluoro-n-[$^{13}C_5$]pentanoic acid	$^{13}C_5$ -PFPeA	96.17	95.68	100.85
Perfluoro-n-[1,2,3,4,6-$^{13}C_5$]hexanoic acid	$^{13}C_5$ -PFHxA	93.57	99.08	96.47
Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid	$^{13}C_4$ -PFHpA	92.56	95.31	90.62
Perfluoro-n-[$^{13}C_8$]octanoic acid	$^{13}C_8$ -PFOA	95.42	98.42	97.05
Perfluoro-n-[$^{13}C_9$]nonanoic acid	$^{13}C_9$ -PFNA	94.45	98.99	97.08
Perfluoro-n-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid	$^{13}C_6$ -PFDA	94.56	97.68	99.62
Perfluoro-n-[1,2,3,4,5,6,7-$^{13}C_7$]undecanoic acid	$^{13}C_7$ -PFUnA	96.73	101.82	99.92
Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid	$^{13}C_2$ -PFDoA	87.03	91.36	95.35
Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid	$^{13}C_2$ -PFTeDA	85.18	88.42	47.90
Perfluoro-1-[2,3,4-$^{13}C_3$]butanesulfonic acid	$^{13}C_3$ -PFBS	98.04	103.06	96.10
Perfluoro-1-[1,2,3-$^{13}C_3$]hexanesulfonic acid	$^{13}C_3$ -PFHxS	94.28	98.15	96.20
Perfluoro-1-[$^{13}C_8$]octanesulfonic acid	$^{13}C_8$ -PFOS	97.43	101.51	99.70
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]hexanesulfonic acid	$^{13}C_2$ -4:2FTS	115.73	143.29	221.50
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]octanesulfonic acid	$^{13}C_2$ -6:2FTS	110.10	122.47	127.00
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]decanesulfonic acid	$^{13}C_2$ -8:2FTS	105.67	109.51	180.75
Perfluoro-1-[$^{13}C_8$]octanesulfonamide	$^{13}C_8$-PFOSA	81.18	84.56	103.85
N-methyl-d_3-perfluoro-1-octanesulfonamide	D_3-NMeFOSA	57.27	42.41	18.27

Table 8-1. Matrices LOQVER Spike Recoveries

Compound	Acronym	Mean % Recovery		
		Aqueous Samples	Solid Samples	Tissue Samples
N-ethyl-d_5-perfluoro-1-octanesulfonamide	D_5-NEtFOSA	58.33	35.94	19.65
N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	D_3-NMeFOSAA	91.09	98.52	171.25
N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	D_5-NEtFOSAA	89.27	97.76	203.50
N-methyl-d_7-perfluorooctanesulfonamidoethanol	D_7-NMeFOSE	67.21	54.89	1.52
N-ethyl-d_9-perfluorooctane sulfonamidoethanol	D_9-NEtFOSE	63.92	51.39	10.04
Tetrafluoro-2-heptafluoropropoxy-$^{13}C_3$-propanoic acid	$^{13}C_3$-HFPO-DA	104.52	106.15	96.00

Recovery within interim study acceptance limit of 70–130% (target analytes) or 50–200% (EIS compounds)
Recovery of EIS compound exceeds interim study limits of 50–200%
Recovery of method analyte exceeds interim study limits of 70–130%
Recovery target analytes exceed interim study limits and EPA LCMRL verification acceptance limit.

9 ONGOING PRECISION AND RECOVERY ANALYSES

Ongoing precision and recovery (OPR) samples, known in some methods as laboratory control samples, were prepared with each preparation batch along with the method blank and LOQVERs. Like the LOQVER, the OPR indicates the performance of the sample preparation. The Study Plan specified that the OPR sample consist of a reference matrix spiked with target analytes at a concentration greater than or equal to the LOQ and less than or equal to the midpoint of the calibration, plus EIS and IIS compounds, which was carried through the entire analytical process. The OPR samples prepared in this study were spiked at the concentration of the CAL 4 standard (Table 4-3). The reference matrices used for the OPR samples were reagent water, wetted Ottawa Sand, and lingcod, for aqueous, solid, and tissue samples, respectively.

OPR sample spike recovery tables were generated for the batched aqueous, solids, and tissue samples. The analyses included the 40 targeted PFAS analytes, and the 24 EIS and IIS compounds. Compilation and analysis of the data were done by IDA; the methods are detailed in Appendix B.

The full results are presented in Appendix H. Each appendix table includes the calculated mean of the OPR subsample percent recoveries and percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes, from the 32 samples, for aqueous and solid matrices (including biosolid, sediment, and soil, but excluding tissue). A separate table is included with the calculated mean of the OPR subsample percent recoveries, percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes from the tissue matrix. All sample percent recoveries values in the EDD were used except that for those data that had B flagged values (where the results were associated with a contaminated blank).

9.1 OPR RESULTS

Table 9-1 presents a summary of the findings reported in Appendix H. The mean percent recoveries were evaluated relative to the interim recovery limits of 70–130% for target analyte recoveries and 50–200% for EIS compounds.

9.2 AQUEOUS OPR RESULTS

For the aqueous target analytes, all OPR mean percent recovery were within the interim recovery limits of 70–130%, as well as the 50 – 150% LCMRL verification criteria. For all EIS compounds, the mean percent recovery was between 50 – 200% recovery as specified in the Study Plan.

9.3 SOLIDS OPR RESULTS

The solid study samples were prepared and analyzed in a total of 17 preparatory batches. One OPR sample was included in each of these batches, as required. For the target analytes, all OPR mean percent recovery were within the interim recovery limits of 70–130%, as well as the 50 – 150% LCMRL verification criteria.

Two OPR mean percent recoveries failed to meet the interim acceptance criteria for EIS recoveries of 50–200%. These failures were associated with D_3-NMeFOSAA and D_5-

NEtFOSAA. It is worth noting that D_7-NMeFOSE and D_9-NEtFOSE were only slightly above the lower 50% acceptance criteria.

9.4 TISSUE OPR RESULTS

The tissue study samples were prepared and analyzed in a total of four preparatory batches. One OPR was included in each of these batches, as required. For the target analytes, all OPR mean percent recoveries were within the interim recovery limits and the LCMRL verification criteria. The one exception was MeFOSE with a mean percent recovery of 231.25%

Average OPR mean percent recoveries were outside the interim recovery limits. OPR mean percent recoveries for D_3-NMeFOSAA, D_5-NEtFOSA, D_7-NMeFOSE, and D_9-NEtFOSE fell below the lower limit in all four OPR samples, ranging from 1.15 to 46.5%. The mean percent recovery for $^{13}C_2$-4:2FTS exceeded the 200% criteria. It is worth noting that $^{13}C_2$-8:2FTS and D_5-NEtFOSA were close to exceeding the upper limit.

Table 9-1. Matrices OPR Spike Recoveries

Compound	Acronym	Mean Percent Recovery		
		Aqueous Samples	Solid Samples	Tissue Samples
PFAS Target Analytes				
Perfluorobutanoic acid	PFBA	100.91	100.27	99.88
Perfluoropentanoic acid	PFPeA	105.41	104.64	104.78
Perfluorohexanoic acid	PFHxA	99.78	97.67	100.62
Perfluoroheptanoic acid	PFHpA	99.91	97.86	102.53
Perfluorooctanoic acid	PFOA	99.48	97.91	97.70
Perfluorononanoic acid	PFNA	100.45	99.71	102.70
Perfluorodecanoic acid	PFDA	103.51	103.44	98.35
Perfluoroundecanoic acid	PFUnA	100.61	101.25	103.53
Perfluorododecanoic acid	PFDoA	103.19	103.40	108.97
Perfluorotridecanoic acid	PFTrDA	105.53	106.82	119.75
Perfluorotetradecanoic acid	PFTA	102.62	100.78	100.90
Perfluorobutanesulfonic acid	PFBS	101.55	101.02	102.88
Perfluoropentanesulfonic acid	PFPeS	100.65	100.37	100.80
Perfluorohexanesulfonic acid	PFHxS	108.40	104.84	106.92
Perfluoroheptanesulfonic acid	PFHpS	100.01	96.13	97.05
Perfluorooctanesulfonic acid	PFOS	105.46	104.32	110.75
Perfluorononanesulfonic acid	PFNS	104.36	96.74	99.50
Perfluorodecanesulfonic acid	PFDS	95.50	95.25	93.90
Perfluorododecanesulfonic acid	PFDoS	90.15	88.19	68.43
4:2 fluorotelomersulfonic acid	4:2FTS	105.24	99.98	96.40
6:2 fluorotelomersulfonic acid	6:2FTS	104.99	113.13	105.35
8:2 fluorotelomersulfonic acid	8:2FTS	111.41	115.65	118.75
Perfluorooctanesulfonamide	PFOSA	106.50	103.85	108.95
N-methyl perfluorooctanesulfonamide	NMeFOSA	97.66	103.89	101.55

Table 9-1. Matrices OPR Spike Recoveries

Compound	Acronym	Mean Percent Recovery		
		Aqueous Samples	Solid Samples	Tissue Samples
N-ethyl perfluorooctanesulfonamide	NEtFOSA	95.91	105.61	108.75
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	100.52	101.46	104.72
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	104.31	102.06	103.58
N-methyl perfluorooctanesulfonamidoethanol	MeFOSE	103.30	103.06	231.25
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	104.79	105.59	109.90
Hexafluoropropylene oxide dimer acid	HFPO-DA	100.84	99.88	100.03
4,8-dioxa-3H-perfluorononanoic acid	ADONA	97.06	99.79	109.03
Perfluoro-3-methoxypropanoic acid	PFMPA	101.28	100.76	97.60
Perfluoro-4-methoxybutanoic acid	PFMBA	97.80	102.83	100.45
Perfluoro-3,6-dioxaheptanoic acid	NFDHA	97.35	97.38	85.70
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	100.09	105.15	110.25
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	95.95	101.78	116.25
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	100.20	98.60	97.38
2H, 2H, 3H, 3H-perfluorohexanoic acid	3:3FTCA	96.71	96.13	83.28
2H, 2H, 3H, 3H-perfluorooctanoic acid	5:3FTCA	98.18	90.43	138.53
2H, 2H, 3H, 3H-perfluorodecanoic acid	7:3FTCA	98.76	89.52	119.00
EIS Compounds				
Perfluoro-n-[$^{13}C_4$]butanoic acid	$^{13}C_4$-PFBA	97.90	102.22	100.00
Perfluoro-n-[$^{13}C_5$]pentanoic acid	$^{13}C_5$-PFPeA	97.70	95.24	95.97
Perfluoro-n-[1,2,3,4,6-$^{13}C_5$]hexanoic acid	$^{13}C_5$-PFHxA	95.54	98.76	93.12
Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid	$^{13}C_4$-PFHpA	94.45	94.92	90.95
Perfluoro-n-[$^{13}C_8$]octanoic acid	$^{13}C_8$-PFOA	95.67	98.12	93.78
Perfluoro-n-[$^{13}C_9$]nonanoic acid	$^{13}C_9$-PFNA	95.51	98.28	95.22
Perfluoro-n-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid	$^{13}C_6$-PFDA	94.90	97.58	96.88
Perfluoro-n-[1,2,3,4,5,6,7-$^{13}C_7$]undecanoic acid	$^{13}C_7$-PFUnA	96.57	103.36	98.75

Table 9-1. Matrices OPR Spike Recoveries

Compound	Acronym	Mean Percent Recovery		
		Aqueous Samples	Solid Samples	Tissue Samples
Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid	$^{13}C_2$-PFDoA	86.85	89.60	88.95
Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid	$^{13}C_2$-PFTeDA	85.06	90.61	59.85
Perfluoro-1-[2,3,4-$^{13}C_3$]butanesulfonic acid	13C3 -PFBS	99.07	102.68	100.15
Perfluoro-1-[1,2,3-$^{13}C_3$]hexanesulfonic acid	$^{13}C_3$-PFHxS	94.09	99.18	96.70
Perfluoro-1-[13C_8]octanesulfonic acid	$^{13}C_8$-PFOS	97.90	101.68	98.95
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]hexanesulfonic acid	$^{13}C_2$-4:2FTS	111.97	134.29	223.25
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]octanesulfonic acid	$^{13}C_2$-6:2FTS	107.71	121.00	132.75
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]decanesulfonic acid	$^{13}C_2$-8:2FTS	103.74	108.38	191.75
Perfluoro-1-[$^{13}C_8$]octanesulfonamide	$^{13}C_8$-PFOSA	82.62	82.65	103.80
N-methyl-d_3-perfluoro-1-octanesulfonamide	D_3-NMeFOSA	61.62	44.26	18.32
N-ethyl-d_5-perfluoro-1-octanesulfonamide	D_5-NEtFOSA	63.29	38.19	26.40
N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	D_3-NMeFOSAA	91.56	97.82	169.75
N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	D_5-NEtFOSAA	89.20	96.82	199.00
N-methyl-d_7-perfluorooctanesulfonamidoethanol	D_7-NMeFOSE	70.44	55.52	2.56
N-ethyl-d_9-perfluorooctane sulfonamidoethanol	D_9-NEtFOSE	66.97	51.94	14.07
Tetrafluoro-2-heptafluoropropoxy-$^{13}C_3$-propanoic acid	$^{13}C_3$-HFPO-DA	104.45	103.81	93.22

Recovery within interim study acceptance limit of 70–130% (target analytes) or 50–200% (EIS compounds)
Recovery of EIS compound exceeds interim study limits of 50–200%
Recovery of method analyte exceeds interim study limits of 70–130%

10 MATRIX SPIKE ANALYSES

Validation of the draft study method used matrices that met the specifications listed in EPA's *Protocol for Review and Validation of New Methods for Regulated Organic and Inorganic Analytes in Wastewater Under EPA's Alternate Test Procedure Program* (EPA, 2018b). Although this is not an alternate test procedure, EPA applies similar matrix testing requirements to methods for new analytes. The environmental matrices discussed previously in Section 2.6 were first measured in triplicate for the native (or background) concentrations of the 40 PFAS target analytes. Each individual environmental sample was spiked in triplicate with all 40 PFAS analytes at the three levels identified in Section 2.5 (low, medium, and high). As noted in Section 2.5, for groundwater, the native concentrations of PFOS in all samples exceeded the "high" spike levels. An additional "High 2" spike levels were added that while nominally targeted to PFOS, included all 40 PFAS analytes (see Table 2-3. Groundwater High 2 Spiking Levels). See Table 3-1 of this report for a breakdown of the number of samples prepared and analyzed for each matrix type.

Samples were prepared by SGS AXYS as stated in Section 2.5 of this report. All samples were prepared, analyzed, and reported as stated in the Study Plan. Once validated, sample results were transmitted to IDA in the form of EDDs and spreadsheets. IDA developed scripts for extracting the relevant data from the EDDs and submitted spreadsheets to conduct the statistical analyses. The methods used by IDA for compiling and statistically analyzing the data are detailed in Appendix B. Results of those analyses are summarized in the following sections.

10.1 NATIVE PFAS CONCENTRATIONS IN MATRICES

The summarized results of the background or "native" concentrations for each of the environmental matrices are presented in Appendix C. Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021). A result was reported for every method analyte, EIS compound, and IIS compound for every sample. The number of detections reported and used for subsequent analyses were dependent upon the associated qualifier. Results with a "U" or "B "flag were not used in the calculation of the mean sample concentration, or calculation of the relative standard deviation. Equations and data rules applied by IDA are in Appendix B, Section 2.

There are 35 tables in Appendix C reporting the native PFAS concentrations in each matrix. Target analytes were detected in all aqueous matrices samples. In groundwater and wastewater samples, PFBA, PFPeA PFHxA, PFOA, PFNA, PFDA, PFBS, PFPeS, PFHxS, PFHpS, and PFOS were measured in all samples (Tables C1–C10). The fluorotelomer sulfates (FTSs) and PFOSA were often detected, but at a lower frequency in the groundwater and wastewater samples. The two freshwater surface waters had similar composition of analytes to the groundwater and wastewater samples (Tables C-11 and C-12), while the marine surface water had fewer detections with all the values being "J" flagged. Of the landfill leachate samples, two were compositionally similar to the groundwater and wastewater samples (see Tables C-14 and C-16), while one of the leachate samples (Table C-15) had 34 of the 40 target analytes reported at relatively elevated levels.

Native soil concentrations in the three samples varied; one sample (Table C-17) reported 20 PFAS including PFOA and PFOS, while the other two soil samples had 4–6 detected target analytes, including PFOA and PFOS. The marine sediment sample had one detection (in the triplicate analyses) of PFPeA, PFHxS, and 6:2 FTS (Table 2-24). One of the freshwater sediment samples

had "J" qualified data for PFPeA and PFHxA (Table C-26), while the other sample had low levels of PFAS ("J"-qualified) including PFOA and PFOS. Each biosolid sample had between 13 and 27 detected target analytes (Tables C-28 through C-29), all including PFOA and PFOS.

In the two fish tissue samples, 8–10 target analytes were reported, although compositionally, the detected compounds are different (Tables C-30 and C-31). Both PFOA and PFOS were detected in one sample (Table C-30), while PFOS, but not PFOA, was detected in the second sample. In the clam tissue, the only analyte detected was 6:2 FTS, but it was detected in only one of the triplicate measures.

Of all the target analytes measured, the following analytes were not detected in any groundwater, wastewater, surface water, soil, sediment, or tissue samples: HFPO-DA, ADONA, PFMBA, PFMPA, NFDHA, 9Cl-PF3ONS, 11Cl-PF3OUdS, PFEESA, or 3:3 FTCA. HFPO-DA, PFMBA, PFEESA, and 3:3 FTCA were reported in one of the three landfill leachate samples (Table C-15), but not in the other two. The only matrices reporting FTCA were the landfill leachates and the biosolids samples.

10.2 AQUEOUS MATRIX SPIKE RESULTS

Aqueous matrices reported included groundwater, wastewater, surface water, and landfill leachate. The methods used by IDA to calculate the percent recoveries, relative standard deviation, and standard deviations are in Appendix B, Section 2. Briefly, the process included determining the mean spike concentration and mean percent recovery for the target analytes, from three subsamples, for each environmental sample. Spike concentration indicates the known amount of analyte added to the native sample, while percent recovery indicates the percent of the analyte detected compared with the known amount. The native concentration was subtracted from the spike concentration before calculating the percent recovery for each subsample. Only positive percent recovery values are reported. Spike concentrations that were flagged by the lab as either U (where it was not detected and less than the limit of detection) or B (where the results were associated with a contaminated blank) were not used to calculate percent recoveries. The results of the determination of the matrix spike recoveries for the individual samples are presented in Appendix D.

The results of the individual sample measurements were then summarized by matrix (e.g., all groundwater, all wastewater, etc.). The results presented in Appendix D for each of the eight environmental matrices are combined and presented in Appendix E. Matrix spike sample recoveries for the four aqueous matrices (groundwater, wastewater, surface water, landfill leachate), the three solid matrices (soil, sediment, biosolids), and tissues were reported. The first eight tables (E1 – E8) report the mean of the sample mean percent recovery, RSD, and SD for the target analytes for the individual matrices from the tables in Appendix D. Two additional summary tables are presented for the combined aqueous matrices, and for the combined solid matrices (Tables E-9, E-10).

The summary statistics by matrix are presented in Table 10-1. The percent mean recovery is the mean for all samples, spike levels, and triplicate analysis for each of the target analytes. For example, the groundwater mean percent recovery is for the three individual groundwater samples, the four (low, medium, high and "high 2") spike levels, conducted in triplicate for the target analytes. It must be noted that non-detected (U-flagged) and blank-contaminated (B-flagged) data are not included in these calculations. Table 10-1 also includes the lowest and highest reported

percent recovery for the individual samples, and the RSD and SD for all matrix samples. The table provides a comparison of the mean, lowest, and highest recoveries for each matrix for each analyte to the Study Plan interim acceptance criteria used to evaluate the QC samples (OPR and LOQVER, or LLOPR), 70–130%.

For groundwater, the mean matrix spike recoveries for all target analytes were between 93.7–118%; within the interim acceptance range of 70 – 130%

The mean matrix spike recoveries for all analytes in the wastewater samples was within a range of 58.7–176.8% for the individual analyte recoveries. Analytes for which the mean was outside the 70–130% range are highlighted in red in Table 10-1, and include PFDoS (58.7%), NMeFOSAA (177.9%), and NEtFOSAA (146.1%).

For surface water, the range mean percent recovery across all samples was 81.3–157.4%. The mean for each analyte was between 70–130% with the exception of NMeFOSAA, with a mean recovery of 157.4%.

Landfill leachate across all 40 analytes was within a range of 54.9 to 164.7%. Nine analytes had means outside of 70–130%: two below 70% (PFDoS and NFDHA), and seven above 130% (NMeFOSAA, ADONA, PFMPA, PFMBA, 9Cl-PF3ONS, PFEESA, 5:3FTCA).

Overall, most of the data fell within the 70–130% goal in the study plan, and almost all of the data were between 50% to 200%.

Table 10-1. Aqueous Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
Groundwater – All Spike Recoveries					
PFBA	105.17	99.21	116.73	5.82	6.12
PFPeA	111.77	104.01	118.19	5.29	5.92
PFHxA	98.68	97.80	99.90	NA	NA
PFHpA	104.84	97.66	115.22	4.83	5.06
PFOA	96.56	96.05	97.05	NA	NA
PFNA	103.34	90.56	112.89	5.44	5.62
PFDA	109.97	96.23	124.71	6.41	7.05
PFUnA	106.10	91.09	119.07	6.78	7.19
PFDoA	104.71	82.26	120.89	8.29	8.68
PFTrDA	106.45	89.23	126.37	10.69	11.38
PFTeDA	106.62	91.86	121.26	8.25	8.79
PFBS	107.60	101.13	113.28	5.53	5.95
PFPeS	105.45	91.43	121.36	13.38	14.11
PFHxS	106.72	104.81	108.21	NA	NA
PFHpS	104.97	89.60	131.50	14.15	14.85
PFOS	109.76	108.59	110.92	NA	NA
PFNS	111.42	83.08	130.47	8.43	9.39
PFDS	99.08	78.04	115.08	6.63	6.56
PFDoS	93.66	74.59	105.99	7.25	6.79
4:2 FTS	105.71	87.74	119.66	7.63	8.07
6:2 FTS	112.55	97.04	130.47	9.89	11.13
8:2 FTS	117.58	104.19	134.00	8.51	10.00
PFOSA	117.88	101.57	147.49	12.58	14.83
NMeFOSA	101.34	73.25	115.11	10.28	10.42
NEtFOSA	100.47	73.76	113.73	9.72	9.77
NMeFOSAA	114.13	90.25	151.29	17.43	19.89
NEtFOSAA	109.44	87.55	137.41	14.25	15.59
NMeFOSE	106.68	85.57	117.66	7.18	7.66
NEtFOSE	109.72	87.64	122.13	8.23	9.03
HFPO-DA	108.79	92.23	125.39	8.28	9.00

Table 10-1. Aqueous Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
ADONA	101.07	83.75	136.20	14.42	14.58
PFMPA	106.29	95.42	120.45	6.96	7.40
PFMBA	104.63	84.43	116.45	5.87	6.15
NFDHA	104.49	82.88	138.81	4.02	4.20
9Cl-PF3ONS	104.21	82.45	142.53	12.86	13.40
11Cl-PF3OUdS	97.43	72.26	125.93	10.02	9.76
PFEESA	105.38	91.34	118.04	6.58	6.93
3:3 FTCA	104.67	91.75	119.89	8.73	9.14
5:3 FTCA	101.61	85.42	118.28	10.17	10.33
7:3 FTCA	104.82	83.50	122.44	7.28	7.64
Wastewater – All Spike Recoveries					
PFBA	97.55	86.44	118.89	1.81	1.77
PFPeA	103.25	89.30	125.80	1.30	1.34
PFHxA	98.02	83.88	114.33	3.16	3.09
PFHpA	97.89	89.40	115.32	0.66	0.65
PFOA	98.08	83.56	119.23	0.83	0.81
PFNA	98.65	79.75	120.61	2.01	1.98
PFDA	101.34	78.41	122.52	3.22	3.26
PFUnA	99.11	86.75	118.55	2.28	2.26
PFDoA	101.04	87.95	117.68	1.79	1.81
PFTrDA	108.46	91.69	136.24	1.80	1.95
PFTeDA	100.79	88.67	122.38	1.81	1.83
PFBS	104.32	93.97	120.06	1.83	1.91
PFPeS	98.41	88.86	118.01	2.71	2.67
PFHxS	107.89	95.26	125.40	1.00	1.08
PFHpS	104.94	84.43	132.05	0.77	0.81
PFOS	99.42	91.35	107.45	4.60	4.58
PFNS	95.97	76.66	107.60	4.45	4.27
PFDS	78.03	57.66	96.04	2.71	2.11
PFDoS	58.69	24.85	82.95	7.39	4.34
4:2 FTS	100.64	87.32	117.95	2.39	2.41

Table 10-1. Aqueous Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
6:2 FTS	115.15	88.81	149.19	6.04	6.96
8:2 FTS	117.24	105.02	139.39	1.98	2.32
PFOSA	107.74	89.35	134.69	2.38	2.56
NMeFOSA	97.92	79.36	132.85	7.13	6.98
NEtFOSA	97.85	80.51	135.08	6.45	6.31
NMeFOSAA	176.88	85.62	411.90	9.62	17.02
NEtFOSAA	146.11	54.83	260.82	8.91	13.02
NMeFOSE	94.29	76.32	123.75	2.10	1.98
NEtFOSE	101.41	85.23	135.86	2.30	2.33
HFPO-DA	98.68	84.47	116.41	1.01	1.00
ADONA	100.94	82.04	120.46	2.06	2.08
PFMPA	108.90	86.64	135.68	1.91	2.08
PFMBA	100.32	85.18	134.95	2.71	2.72
NFDHA	76.44	42.25	118.99	5.92	4.52
9Cl-PF3ONS	89.22	49.80	128.10	3.30	2.95
11Cl-PF3OUdS	74.96	30.92	116.46	1.03	0.78
PFEESA	102.81	85.34	116.25	1.77	1.82
3:3 FTCA	92.38	23.76	135.53	6.45	5.96
5:3 FTCA	110.37	68.65	163.17	2.67	2.95
7:3 FTCA	97.57	60.18	120.60	3.69	3.60
Surface Water – All Spike Recoveries					
PFBA	104.29	91.46	118.49	7.47	7.79
PFPeA	112.25	97.58	130.04	6.09	6.83
PFHxA	100.53	93.37	114.37	6.22	6.25
PFHpA	101.39	81.06	125.56	12.70	12.88
PFOA	97.81	88.00	102.87	8.69	8.50
PFNA	106.62	90.69	123.85	6.72	7.16
PFDA	107.57	91.30	121.81	9.14	9.83
PFUnA	105.94	90.41	119.96	7.87	8.34
PFDoA	104.20	86.28	123.36	8.33	8.68
PFTrDA	107.95	87.19	124.54	8.40	9.07

Table 10-1. Aqueous Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFTeDA	106.50	87.20	122.51	8.24	8.78
PFBS	104.58	94.37	123.82	8.46	8.84
PFPeS	104.11	95.13	125.46	7.59	7.90
PFHxS	106.96	97.34	111.89	7.79	8.33
PFHpS	104.81	84.70	123.33	7.62	7.98
PFOS	108.81	99.16	115.94	7.97	8.67
PFNS	103.70	76.88	123.16	7.03	7.29
PFDS	94.50	74.12	106.91	8.56	8.09
PFDoS	81.30	59.08	101.71	8.61	7.00
4:2 FTS	107.85	96.77	122.34	7.64	8.24
6:2 FTS	115.09	65.44	141.26	12.76	14.68
8:2 FTS	115.30	100.45	127.91	9.40	10.83
PFOSA	117.07	88.51	141.32	8.22	9.62
NMeFOSA	101.69	79.01	118.48	12.72	12.94
NEtFOSA	99.81	80.03	113.43	13.45	13.43
NMeFOSAA	157.41	105.71	204.78	10.41	16.39
NEtFOSAA	128.45	100.81	159.22	12.93	16.60
NMeFOSE	103.60	86.52	119.46	9.88	10.24
NEtFOSE	108.79	90.90	124.21	10.89	11.84
HFPO-DA	105.10	83.30	120.55	8.81	9.26
ADONA	102.52	86.93	117.64	8.99	9.21
PFMPA	104.05	92.90	124.26	7.58	7.89
PFMBA	101.41	85.76	119.53	8.48	8.60
NFDHA	107.20	79.02	139.28	6.76	7.24
9Cl-PF3ONS	103.31	77.46	123.13	9.90	10.22
11Cl-PF3OUdS	92.84	63.79	109.19	11.21	10.41
PFEESA	102.37	93.21	116.74	5.28	5.41
3:3 FTCA	102.97	87.55	120.65	6.16	6.34
5:3 FTCA	97.77	80.99	115.19	6.51	6.37
7:3 FTCA	95.52	74.95	114.27	8.95	8.55
Landfill Leachate – All Spike Recoveries					

Table 10-1. Aqueous Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFBA	104.99	100.64	109.03	2.20	2.31
PFPeA	115.04	112.68	117.40	2.90	3.34
PFHxA	NA	NA	NA	NA	NA
PFHpA	108.22	105.38	110.12	2.32	2.51
PFOA	106.43	104.30	107.86	1.77	1.88
PFNA	103.74	98.10	110.47	5.14	5.34
PFDA	110.96	107.86	113.26	1.92	2.13
PFUnA	106.32	94.76	115.21	6.52	6.93
PFDoA	109.65	97.83	129.86	8.10	8.88
PFTrDA	107.73	99.27	114.38	2.75	2.97
PFTeDA	112.25	104.22	120.07	1.79	2.00
PFBS	117.91	117.91	117.91	NA	NA
PFPeS	112.10	100.70	124.31	6.91	7.75
PFHxS	114.74	103.56	118.54	2.80	3.22
PFHpS	110.62	101.07	119.05	5.44	6.01
PFOS	114.97	107.10	123.36	6.19	7.11
PFNS	99.76	87.55	109.08	2.59	2.59
PFDS	79.36	55.96	94.89	1.05	0.83
PFDoS	54.89	22.91	77.60	6.30	3.46
4:2 FTS	109.19	107.75	112.09	0.89	0.97
6:2 FTS	107.50	101.85	112.18	1.32	1.42
8:2 FTS	121.93	114.29	132.49	3.79	4.62
PFOSA	121.87	102.22	178.31	13.08	15.94
NMeFOSA	109.63	94.28	119.40	4.27	4.68
NEtFOSA	108.43	91.92	116.52	5.13	5.56
NMeFOSAA	140.33	87.39	220.51	8.58	12.05
NEtFOSAA	123.56	87.72	155.67	1.78	2.20
NMeFOSE	108.77	101.75	112.83	3.35	3.64
NEtFOSE	112.31	105.37	116.38	3.95	4.43
HFPO-DA	110.48	106.90	118.41	1.62	1.79
ADONA	142.56	103.60	196.71	1.16	1.66

Table 10-1. Aqueous Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFMPA	135.33	64.16	197.46	14.34	19.41
PFMBA	132.06	109.93	166.11	2.58	3.41
NFDHA	55.08	17.30	102.96	5.54	3.05
9Cl-PF3ONS	140.04	92.47	200.22	1.82	2.55
11Cl-PF3OUdS	109.47	82.93	141.74	1.38	1.51
PFEESA	162.38	105.31	274.32	10.29	16.71
3:3 FTCA	127.51	29.12	210.24	14.87	18.96
5:3 FTCA	164.68	109.67	219.25	3.18	5.23
7:3 FTCA	128.06	85.99	167.59	12.52	16.04

Mean matrix spike recoveries between 70–130%. Some individual samples outside that range
Mean and individual matrix spike recoveries outside of 70–130%
NA = Not applicable. SD could not be calculated

10.3 Soil/Sediment and Biosolids Matrix Spike Results

Results for soils, sediments, and biosolids are summarized in Table 10-2. A complete compilation of all individual sample results and statistical calculations are found in Appendices D and E.

The mean percent recovery for the soils was within a range of 90.2–121.5%. All mean percent recoveries for all target analytes were between 70–130%, but with individual measures above or below the interim criteria. Of note is that while for 6:2FTS the mean percent recovery was 95.7%, one individual soil measure was at 11.2% recovery, and a separate measure at 147.5%. Only one additional individual measure exceeded 130%: 8:2FTS at 131.2%.

For the three sediment samples, the average recovery for the target analytes was 104.3%, with a range of 98.6–120%. The sole individual measure below 70% recovery was for NFDHA (68.2%). The sole individual measure exceeding 130% was a marginal exceedance (130.8%) for 11Cl-PF3OUdS.

For biosolids, the average recovery across all analytes was 98.4%, with a range of 40.6–127.6%. There are four analytes for which the mean percent recovery was less than 70%: PFDoS (40.6), PFMPA (52.2), NFDHA (67.3), and 3:3FTCA (46.5). There were no analytes for which the mean percent recovery exceeded 130%. Nine individual measures were below 70%, the lowest being 3:3FTCA at 10.1%. Additional low individual recoveries worth noting included PFBA (40%), PFDoS (29.11), PFMPA (16.7), and NFDHA (29%). Eleven individual measures exceeded 130%; these were at or below 178.7%. One individual measure of PFOS in biosolids was reported at 178.7% recovery. Of additional note is that for PFDoS, the mean recovery across all measures was 40.6%, with a low of 29.1% and the highest measure at 59.3% recovery.

Table 10-2. Solid Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
Soil – All Spike Recoveries					
PFBA	106.16	101.56	108.65	0.46	0.49
PFPeA	107.25	97.74	115.34	2.22	2.38
PFHxA	100.75	95.71	106.25	0.79	0.80
PFHpA	101.42	96.88	107.24	0.75	0.76
PFOA	100.00	94.20	106.42	0.97	0.97
PFNA	103.50	98.27	110.54	0.52	0.54
PFDA	105.35	99.30	112.17	1.80	1.90
PFUnA	102.95	91.73	108.82	1.69	1.74
PFDoA	105.13	90.57	116.27	1.61	1.69
PFTrDA	112.59	105.58	119.32	1.67	1.88
PFTeDA	105.12	93.43	112.88	1.25	1.31
PFBS	105.67	99.21	109.65	1.22	1.29
PFPeS	103.70	98.11	110.76	0.35	0.37
PFHxS	109.32	104.73	114.88	1.61	1.76
PFHpS	99.09	94.00	103.28	1.21	1.20
PFOS	109.15	102.49	118.60	1.51	1.65
PFNS	100.81	88.02	111.46	0.87	0.87
PFDS	100.60	91.87	106.42	1.64	1.65
PFDoS	90.24	76.70	100.10	1.47	1.33
4:2 FTS	103.91	96.06	110.38	2.71	2.82
6:2 FTS	95.72	11.24	147.45	15.32	14.66
8:2 FTS	121.49	113.31	131.95	0.94	1.14
PFOSA	106.16	102.02	110.54	1.57	1.66
NMeFOSA	108.89	102.26	113.86	0.47	0.51
NEtFOSA	112.68	108.13	117.03	1.27	1.43
NMeFOSAA	103.02	97.15	110.15	2.15	2.21
NEtFOSAA	109.41	101.21	114.53	1.18	1.29
NMeFOSE	107.76	104.07	112.51	1.94	2.09
NEtFOSE	110.39	105.49	114.25	1.64	1.81
HFPO-DA	105.05	87.79	119.70	0.72	0.76

Table 10-2. Solid Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
ADONA	104.05	91.91	123.47	3.95	4.11
PFMPA	108.53	98.34	116.95	5.72	6.21
PFMBA	110.44	90.68	122.27	7.78	8.59
NFDHA	96.98	70.63	125.78	6.73	6.52
9Cl-PF3ONS	111.64	96.26	130.43	5.29	5.91
11Cl-PF3OUdS	110.29	93.93	127.26	3.02	3.34
PFEESA	100.59	94.93	106.75	2.41	2.42
3:3 FTCA	104.30	92.31	112.86	5.47	5.70
5:3 FTCA	96.42	86.49	108.30	3.27	3.16
7:3 FTCA	99.32	90.65	108.69	1.36	1.35
Sediment – All Spike Recoveries					
PFBA	104.95	97.66	108.49	2.20	2.31
PFPeA	106.99	99.77	111.87	4.02	4.31
PFHxA	100.04	95.70	103.54	2.72	2.73
PFHpA	99.87	94.59	103.65	3.87	3.86
PFOA	101.37	95.06	106.25	2.30	2.34
PFNA	103.98	96.20	109.79	3.54	3.68
PFDA	104.79	99.54	108.54	2.13	2.23
PFUnA	103.17	97.27	108.96	4.31	4.44
PFDoA	106.23	97.71	112.58	2.32	2.47
PFTrDA	111.43	103.81	114.54	0.40	0.45
PFTeDA	104.94	97.55	108.32	3.17	3.33
PFBS	104.95	98.18	109.49	3.05	3.20
PFPeS	103.58	99.07	106.86	2.98	3.09
PFHxS	113.08	107.99	120.66	2.75	3.11
PFHpS	97.60	91.69	103.33	2.61	2.55
PFOS	109.60	105.24	115.21	3.46	3.79
PFNS	100.83	87.99	112.69	9.56	9.64
PFDS	100.01	94.39	110.62	4.49	4.49
PFDoS	93.55	90.12	100.42	1.94	1.81
4:2 FTS	102.77	96.74	106.56	2.48	2.54

Table 10-2. Solid Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
6:2 FTS	99.92	76.33	126.38	8.18	8.18
8:2 FTS	120.04	110.16	128.13	4.29	5.15
PFOSA	106.36	98.23	110.21	3.26	3.46
NMeFOSA	107.77	104.33	111.23	2.38	2.56
NEtFOSA	110.39	102.71	116.03	2.90	3.21
NMeFOSAA	99.47	90.89	107.50	5.88	5.85
NEtFOSAA	106.99	99.90	114.96	0.44	0.47
NMeFOSE	108.20	96.00	117.68	4.24	4.59
NEtFOSE	110.14	101.32	114.17	3.63	4.00
HFPO-DA	106.61	90.26	127.30	8.82	9.40
ADONA	106.68	93.00	119.42	5.41	5.77
PFMPA	97.62	94.01	100.40	1.85	1.81
PFMBA	99.66	93.43	103.75	3.39	3.38
NFDHA	94.45	68.24	115.78	11.94	11.28
9Cl-PF3ONS	113.98	101.31	127.54	4.62	5.26
11Cl-PF3OUdS	116.46	102.35	130.84	4.81	5.60
PFEESA	99.06	91.46	103.84	4.11	4.07
3:3 FTCA	94.01	88.45	100.16	0.79	0.74
5:3 FTCA	94.57	78.79	106.04	1.69	1.60
7:3 FTCA	106.18	94.50	113.62	2.69	2.85
Biosolids - All Spike Recoveries					
PFBA	90.01	36.96	109.20	21.42	19.28
PFPeA	105.99	89.97	120.88	11.08	11.75
PFHxA	95.48	81.01	105.37	10.75	10.27
PFHpA	95.06	83.64	103.44	7.07	6.73
PFOA	94.74	81.40	105.60	10.58	10.02
PFNA	98.94	86.54	107.09	8.94	8.84
PFDA	96.29	70.74	109.69	12.70	12.23
PFUnA	98.90	87.30	110.11	8.69	8.59
PFDoA	109.80	78.21	156.18	15.98	17.54
PFTrDA	127.57	88.91	174.79	16.08	20.51

Table 10-2. Solid Samples Summary Matrix Recovery Table

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFTeDA	98.93	81.34	107.42	11.58	11.45
PFBS	91.38	55.94	117.74	7.05	6.44
PFPeS	114.16	89.24	143.01	4.24	4.84
PFHxS	116.01	98.61	134.03	7.44	8.63
PFHpS	116.25	81.92	178.70	8.84	10.28
PFOS	116.34	96.23	178.73	20.92	24.34
PFNS	94.53	77.83	105.59	11.07	10.46
PFDS	87.98	56.78	115.12	9.48	8.34
PFDoS	40.62	29.11	59.34	8.65	3.51
4:2 FTS	98.68	84.77	109.06	8.86	8.74
6:2 FTS	83.73	52.20	111.45	9.96	8.34
8:2 FTS	121.42	102.08	134.24	9.83	11.93
PFOSA	101.48	86.03	112.23	9.30	9.43
NMeFOSA	106.25	92.03	117.92	7.99	8.49
NEtFOSA	109.88	94.72	121.50	8.70	9.56
NMeFOSAA	101.82	86.58	117.02	11.73	11.94
NEtFOSAA	105.05	88.45	115.26	9.57	10.06
NMeFOSE	109.42	93.73	128.23	3.55	3.88
NEtFOSE	109.21	79.56	137.44	11.67	12.74
HFPO-DA	108.09	81.76	141.72	16.97	18.34
ADONA	107.21	87.69	126.07	12.08	12.95
PFMPA	52.19	16.69	88.82	54.34	28.36
PFMBA	106.90	86.22	146.51	15.50	16.57
NFDHA	67.30	28.99	110.24	12.41	8.35
9Cl-PF3ONS	109.51	86.25	128.01	13.12	14.37
11Cl-PF3OUdS	94.96	61.55	125.54	19.21	18.25
PFEESA	100.83	89.03	117.98	7.76	7.82
3:3 FTCA	46.45	10.06	89.65	63.46	29.48
5:3 FTCA	105.26	82.44	129.98	10.39	10.94
7:3 FTCA	102.95	70.17	135.71	17.60	18.12

Mean matrix spike recoveries between 70–130%. Some individual samples outside that range
Mean and individual matrix spike recoveries outside of 70–130%

10.4 TISSUE MATRIX SPIKE RESULTS

Tissue matrix spike recoveries for the two fish and one clam tissue samples are summarized in Table 10-3. For these tissue samples, the range of the mean percent recovery for the target analytes was 62.8–194.4%. The analyte for which the average recovery was below 70% is PFDoS (63%). The PFAS for which the average matrix spike recovery exceeded 130% include NMeFOSE (140.6%), NEtFOSE (162.7%), 5:3FTCA (170.2%) and 7:3FTCA (194.4%). There were four individual analyte measurements that were less than 70% recovery. Of particular note are PFDoS (33.4%) and 6:2FTS (17.6%); thirteen individual measurements exceeded 130% recovery.

Table 10-3. Tissue Samples Summary Matrix Recovery (Fish Tissue – All Spike Recoveries)

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFBA	105.07	99.59	109.29	3.84	4.03
PFPeA	110.70	104.80	117.77	3.49	3.87
PFHxA	101.14	92.12	110.37	5.05	5.10
PFHpA	108.24	97.85	116.37	3.69	4.00
PFOA	104.63	97.94	111.61	4.34	4.54
PFNA	105.89	98.76	113.28	4.34	4.60
PFDA	103.90	96.51	108.90	3.76	3.91
PFUnA	104.08	95.59	107.92	4.50	4.68
PFDoA	112.84	94.53	139.53	9.61	10.84
PFTrDA	118.60	99.83	146.96	9.52	11.29
PFTeDA	105.71	99.92	112.02	4.57	4.83
PFBS	107.25	102.06	112.44	3.84	4.12
PFPeS	96.35	87.06	106.02	3.17	3.05
PFHxS	119.63	100.43	150.76	5.91	7.07
PFHpS	100.32	94.23	106.84	4.05	4.06
PFOS	106.10	99.33	112.96	5.21	5.53
PFNS	91.16	74.07	116.79	5.26	4.80
PFDS	86.29	72.44	97.81	4.77	4.12
PFDoS	62.79	33.41	94.40	16.83	10.56
4:2 FTS	104.44	97.10	110.96	4.92	5.14
6:2 FTS	84.96	17.62	118.02	17.15	14.57
8:2 FTS	122.80	114.11	130.88	4.10	5.04
PFOSA	108.96	103.39	113.28	4.04	4.40
NMeFOSA	115.19	98.43	130.07	4.13	4.76
NEtFOSA	119.02	95.03	148.61	7.80	9.29

Table 10-3. Tissue Samples Summary Matrix Recovery (Fish Tissue – All Spike Recoveries)

Analyte	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
NMeFOSAA	107.77	99.25	116.60	4.76	5.13
NEtFOSAA	113.85	104.05	125.92	4.01	4.56
NMeFOSE	140.57	104.63	186.40	12.48	17.55
NEtFOSE	162.70	89.20	342.83	28.75	46.78
HFPO-DA	103.51	89.50	118.48	6.37	6.59
ADONA	118.62	102.47	151.24	12.40	14.71
PFMPA	117.29	97.97	143.51	3.58	4.20
PFMBA	107.68	99.78	116.56	3.59	3.86
NFDHA	72.67	50.31	111.28	16.89	12.27
9Cl-PF3ONS	129.58	101.85	160.19	9.55	12.38
11Cl-PF3OUdS	125.48	83.01	158.53	8.75	10.99
PFEESA	116.03	94.47	136.93	6.50	7.55
3:3 FTCA	92.16	68.70	118.06	1.43	1.32
5:3 FTCA	170.20	129.70	233.54	10.02	17.06
7:3 FTCA	194.43	114.19	276.83	7.56	14.70

Mean matrix spike recoveries between 70–130%. Some individual samples outside that range
Mean and individual matrix spike recoveries outside of 70–130%

10.5 Summary by Matrix of Matrix Spike Results

Table 10-4 summarizes by matrices the findings discussed in Sections 10.1 through 10.4. When evaluated against the 70–130% interim OPR/LOQVER acceptance criterion, generally, the matrix spike recoveries with the analytical procedure were good across all aqueous matrices. This is especially true for groundwater and surface water, where all recoveries but for one target analyte were within 70–130%. The one exception was NMeFOSAA in surface water. For wastewater, the analytical method appears to be able to routinely meet this interim criterion, with the exceptions of PFDoS, NMeFOSAA, and NEtFOSAA. For landfill leachate, eight analytes did not meet the interim criterion: PFDoS, NMeFOSAA, ADONA, PFMPA, NFDHA, 9Cl-PF3ONS, PFEESA, and 5:3FTCA.

For soils and sediment, the extraction and analyses were robust for all target analytes; the averages for all 40 fell within the 70–130% recovery criterion. For biosolids, there are four analytes that exceeded the interim study criterion: PFDoS, PFMPA, NFDHA, and 3:3FTCA. For tissues, matrix spike recoveries for PFDOS, NMeFOSE, NEtFOSE, HFPO-DA, 5:3FTCA, and 7:3FTCA were outside the interim criterion.

Table 10-4. Summary by Matrix of Matrix Spike Results

Analyte	Aqueous Samples				Solid Samples			Tissue Samples
	Groundwater	Surface Water	Wastewater	Landfill Leachate	Soil	Sediment	Biosolid	Fish Tissue
PFBA	+	+	+	+	+	+	+	+
PFPeA	+	+	+	+	+	+	+	+
PFHxA	+	+	+	+	+	+	+	+
PFHpA	+	+	+	+	+	+	+	+
PFOA	+	+	+	+	+	+	+	+
PFNA	+	+	+	+	+	+	+	+
PFDA	+	+	+	+	+	+	+	+
PFUnA	+	+	+	+	+	+	+	+
PFDoA	+	+	+	+	+	+	+	+
PFTrDA	+	+	+	+	+	+	+	+
PFTeDA	+	+	+	+	+	+	+	+
PFBS	+	+	+	+	+	+	+	+
PFPeS	+	+	+	+	+	+	+	+
PFHxS	+	+	+	+	+	+	+	+
PFHpS	+	+	+	+	+	+	+	+
PFOS	+	+	+	+	+	+	+	+
PFNS	+	+	+	+	+	+	+	+
PFDS	+	+	+	+	+	+	+	+
PFDoS	+	+	-	-	+	+	-	-
4:2 FTS	+	+	+	+	+	+	+	+
6:2 FTS	+	+	+	+	+	+	+	+

Table 10-4. Summary by Matrix of Matrix Spike Results

Analyte	Aqueous Samples				Solid Samples			Tissue Samples
	Groundwater	Surface Water	Wastewater	Landfill Leachate	Soil	Sediment	Biosolid	Fish Tissue
8:2 FTS	+	+	+	+	+	+	+	+
PFOSA	+	+	+	+	+	+	+	+
NMeFOSA	+	+	+	+	+	+	+	+
NEtFOSA	+	+	+	+	+	+	+	+
NMeFOSAA	+	-	-	-	+	+	+	+
NEtFOSAA	+	+	-	+	+	+	+	+
NMeFOSE	+	+	+	+	+	+	+	-
NEtFOSE	+	+	+	+	+	+	+	-
HFPO-DA	+	+	+	+	+	+	+	-
ADONA	+	+	+	-	+	+	+	+
PFMPA	+	+	+	+	+	+	-	+
PFMBA	+	+	+	-	+	+	+	+
NFDHA	+	+	+	-	+	+	-	+
9Cl-PF3ONS	+	+	+	-	+	+	+	+
11Cl-PF3OUdS	+	+	+	+	+	+	+	+
PFEESA	+	+	+	-	+	+	+	+
3:3 FTCA	+	+	+	+	+	+	-	+
5:3 FTCA	+	+	+	-	+	+	+	-
7:3 FTCA	+	+	+	+	+	+	+	-

+ = Average spike recoveries between 70–130%; - = Average matrix spike recoveries outside of 70–130%

10.6 SUMMARY OF MATRIX SPIKE RESULTS

Percent recoveries of all matrix spikes were statistically evaluated as the metric of method accuracy. All "B" flagged data were eliminated from further analysis. Prior to analysis, the mean native concentration in unspiked sample aliquots for each target analyte (reported in Table 10-5) was subtracted from the measured concentration in each spiked aliquot before calculating percent recoveries (no "background" correction was made for samples in which the unspiked aliquots exclusively contained "U" flagged results). All data associated with samples where the mean native concentration exceeded the spiked concentration for a given method analyte also were eliminated from further analysis. Calculated mean recoveries among laboratory subsamples were subsequently used as the dependent variable in a multiple factor ANOVA where categorical spike concentration (SPIKE; low, medium, high), matrix (MATRIX; biosolids; BS, groundwater; GW, landfill leachate; LC, sediment; SD, surface soil; SS, surface water; SW, tissue; TS, and wastewater; WW), and PFAS (PFAS; the 40 target analytes) were main effects with evaluation of all interaction effects therein. The RSD among laboratory subsamples were similarly evaluated as the metric of method precision.

ANOVA results for the accuracy analysis are presented in Table 10-5. The two-way interactions MATRIX*SPIKE and MATRIX*PFAS were significant and are presented in Figure 1. No trend was apparent with increasing spike concentration, but mean percent recoveries were well within control limits (Figure 1A). However, mean percent recoveries for several PFAS*MATRIX combinations were outside of the lower and upper interim control limits established at 70% and 130% percent recovery, respectively (Figure 1B). These same data (presented in Figure 1B) were dichotomized in terms of compliance with the interim control limits and are presented in Figure 2. The cell for the PFHxA-LC combination is blank because all samples contained native concentrations in excess of the highest spike concentration and, thus, all of those data were deemed unusable.

Table 10-5. Accuracy Analysis: ANOVA Results for Calculated Matrix Spike Recoveries

Effect[1,2]	F Value	P Value[3]
A*B	**10.80**	**<0.0001**
A*C	**6.32**	**<0.0001**
B*C	0.44	1.0000
A*B*C	0.43	1.0000

[1]A = Matrix; [1]B = Spike Category; [1]C = PFAS Target analytes
[2]Because significant interactions included all variables evaluated, main effect results are not shown.
[3]Bolded entries are significant at $\alpha = 0.05$.

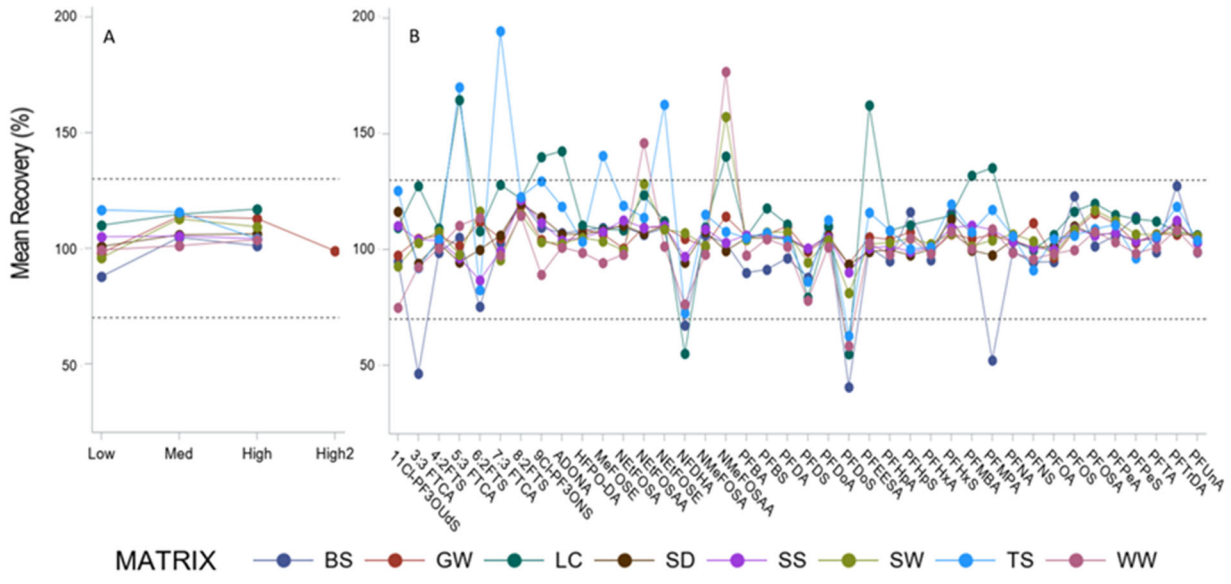


Figure 1. Accuracy Analysis

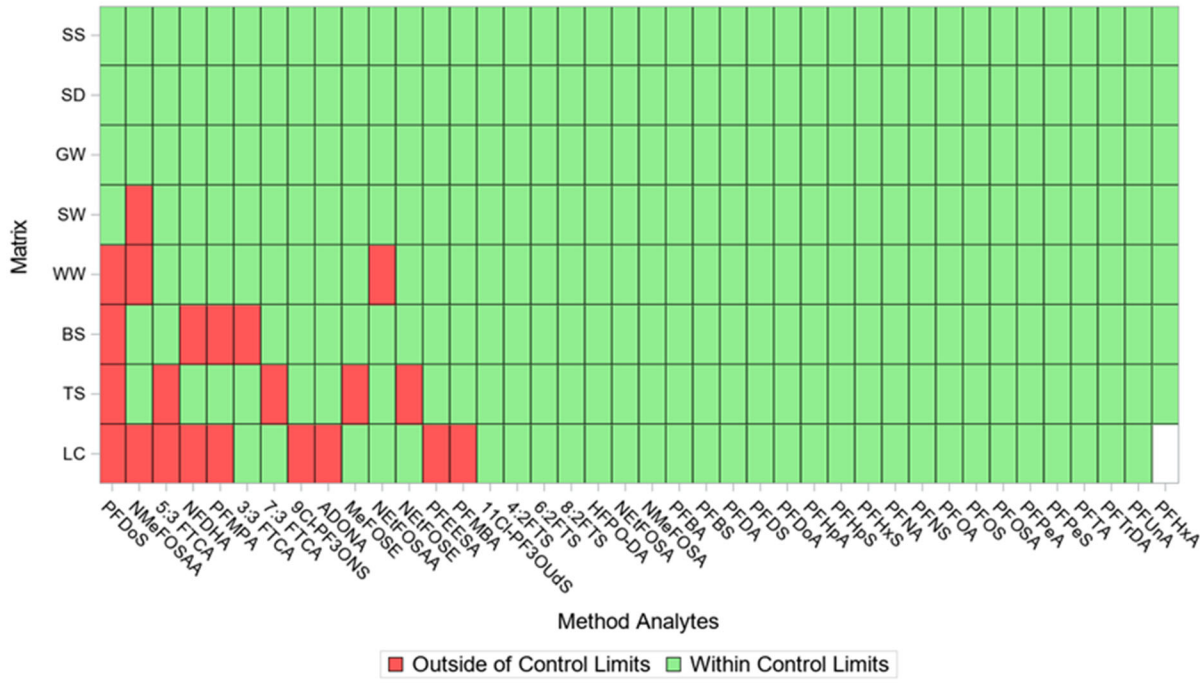


Figure 2. Accuracy Analysis

Significant two-way interactions from ANOVA are presented in Table 10-5. Panel A (Figure 1A) represents mean spike recoveries for each matrix and spike category combination (the High 2 spike category is specific to the groundwater matrix) and averages over all PFAS target analytes. Similarly, Panel B (Figure 1B) represents each PFAS method analyte and matrix combination and averages over spike category. Horizontal reference lines represent 70% and 130% interim control limits.

Data presented in Figure 2 above are dichotomized in terms of compliance with interim control limits. The PFHxA-LC cell is blank because the mean native concentration in all samples was higher than the spiked concentrations and, thus, all those data were deemed unusable.

Similarly, ANOVA results for the precision analysis are presented in Table 10-6. The two-way interactions MATRIX*SPIKE and MATRIX*PFAS were again significant and are presented in Figure 3. A decreasing trend was apparent with increasing spike concentration, indicating greater method precision at higher concentrations (Figure 3A). As with the accuracy analysis, however, mean RSDs for several PFAS*MATRIX combinations were outside of the interim control limit established at 20% (Figure 3B). These same data (presented in Figure 3B) were dichotomized in terms of compliance with the control limit and are presented in Figure 4. As with Figure 2, the cell for the PFHxA-LC combination is blank because all samples contained native concentrations in excess of the highest spike concentration and, thus, all those data were deemed unusable. All data used in the evaluation of method accuracy and precision were combined and evaluated collectively in terms of compliance with the control limits and are presented in Figure 5. The vast majority of PFAS method analyte and matrix combinations (>93%) were within accuracy and precision interim control limits. Those PFAS method analyte and matrix combinations with mean recoveries and/or RSDs outside of the interim control limits can be determined from Figures 1-4.

Table 10-6. Precision Analysis: ANOVA Results for Calculated RSDs among Matrix Spike Recoveries

Effect[1,2]	F Value	P Value[3]
A*B	**2.57**	**0.0011**
A*C	**1.82**	**<0.0001**
B*C	0.44	0.9981
A*B*C	0.43	0.9899

[1]A = Matrix; [1]B = Spike Category; [1]C = PFAS Target analytes
[2]Because significant interactions included all variables evaluated, main effect results are not shown.
[3]Bolded entries are significant at α = 0.05.

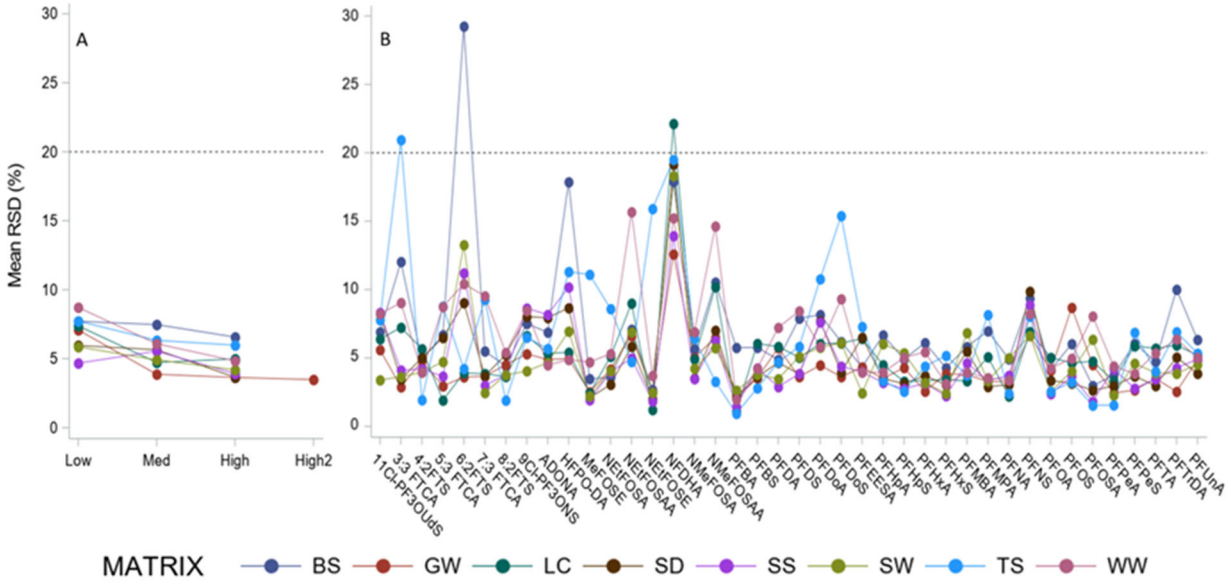


Figure 3. Precision Analysis

Significant two-way interactions from ANOVA are presented in Table 10-6. Panel A (Figure 3A) represents mean RSDs for each matrix and spike category combination (the High 2 spike category is specific to the groundwater matrix) and averages over all PFAS target analytes. Similarly, Panel B (Figure 3B) represents each PFAS method analyte and matrix combination and averages over the spike categories. The horizontal reference line represents an interim 20% control limit.

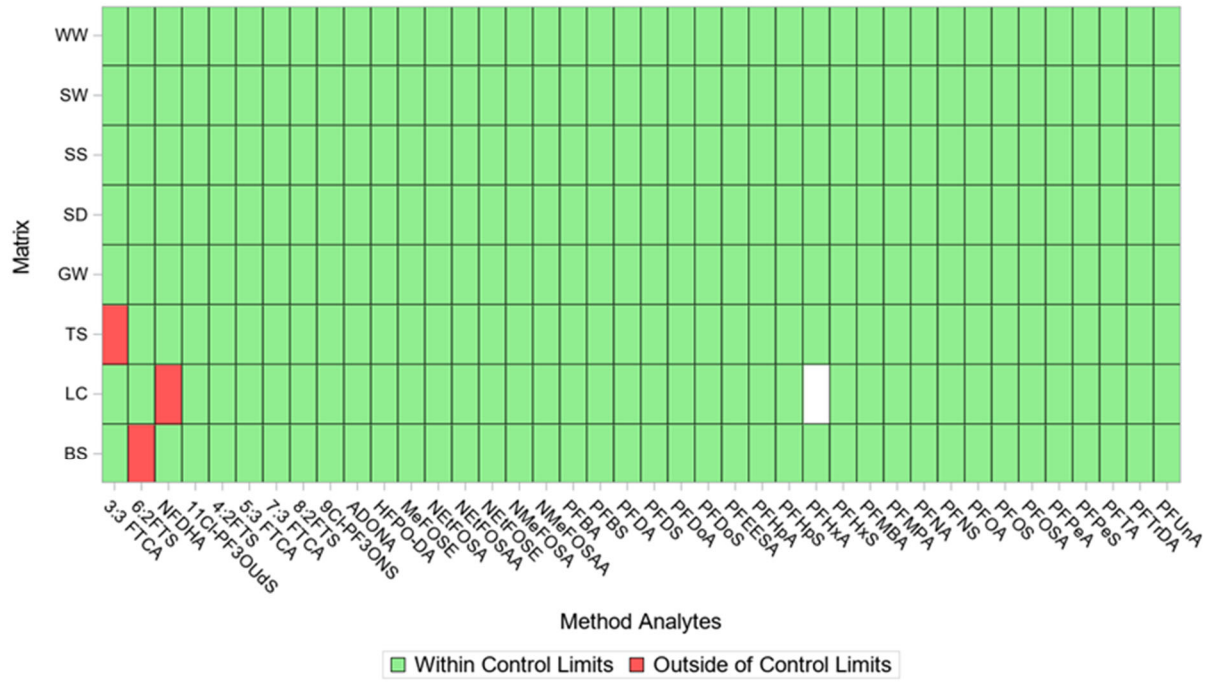


Figure 4. Precision Analysis

Data presented in Figure 4 dichotomized in terms of compliance with the interim control limit. The PFHxA-LC cell is blank because the mean native concentration in all samples was higher than the spiked concentrations and, thus, all those data were deemed unusable.

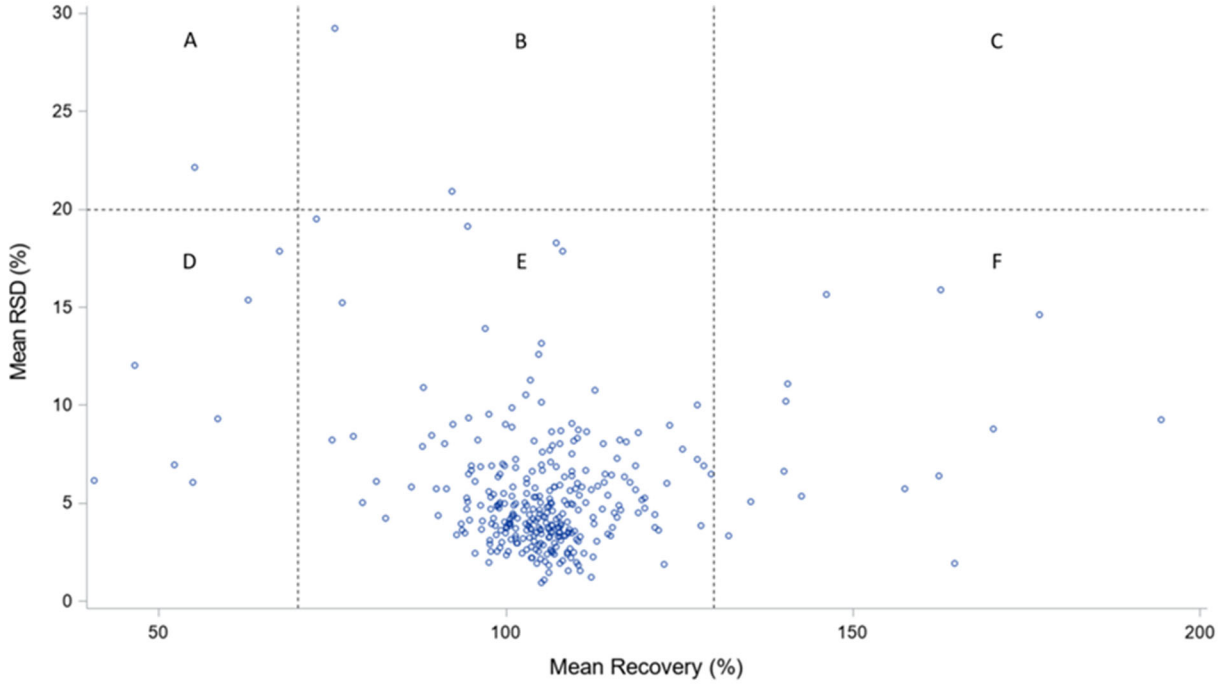


Figure 5. Relationship Between Accuracy and Precision

The relationship between accuracy and precision as paired by the two-way interaction results from Figures 1B and 3B above that represent each PFAS method analyte and matrix combination and averages over spike category. Dashed reference lines reflect interim control limits. Panels A, B, C, D, E and F represent permutations with low bias and imprecision, accuracy and imprecision, high bias and imprecision, low bias and precision, accuracy and precision, and high bias and precision, respectively. The vast majority of PFAS method analyte and matrix combinations (>93%) were within accuracy and precision interim control limits.

The method has been tested in a wide variety of environmental matrices. Overall, the method performance is better than most non-drinking water EPA methods for organic analytes. This justifies proceeding to the multi-laboratory validation study where method performance at a typical full service environmental laboratory will be determined. The 70–130% criteria was a goal, not a requirement, and was mostly achieved. EPA will develop method performance criteria from the results of the MLVS.

11 EXTRACTED INTERNAL STANDARD AND NON-EXTRACTED INTERNAL STANDARD RECOVERY ANALYSES

EIS and IIS compounds were run as quality control with each sample and sample batch. ^{13}C- and deuterium-labeled EIS standards were specified in the SLVS Method (Appendix A) and are listed in each of Tables 11-1 through 11-4.

For this report, the mean and range percent recoveries are discussed. As specified in the SLVS Method (Appendix A), an interim recovery criterion of 50–200% for EIS compounds was applied to these results. For IIS compounds, the same criteria of 50–200 % was also applied for discussion purposes only. Actual target recoveries will be set after completion of the MLVS.

11.1 EIS RESULTS

Appendix F presents 11 summary tables of EIS spike recoveries for each of the eight environmental matrices: groundwater, wastewater, surface water, landfill leachate, soils, sediment, biosolids and tissue. Summary data in those tables for each individual sample include the mean spike concentration, the number of spikes, the mean percent recovery, and the percent RSD for 24 isotopically labeled analog PFAS analytes. For each matrix (e.g., groundwater, biosolids) a set of summary statistics is presented with mean spike concentration, number of EIS spikes (i.e., the average of the number of samples by matrices), the mean percent recovery, and the RSD.

Summaries by aqueous, solid, and tissue matrix types derived from the Appendix F tables are shown in Tables 11-1 through 11-3. The matrix summary tables include the mean of the matrix mean concentrations; mean of the matrix mean percent recoveries; and percent RSD, SD, and 2×SD of the matrix mean percent recoveries for the 24 EIS compounds.

Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021).

11.1.1 Aqueous EIS Results

Table 11-1 shows EIS recovery results averaged across groundwater, surface water, wastewater, and landfill leachate samples. The mean percent recovery for EIS analytes across all aqueous matrices is within interim recovery limits. EIS compound recoveries are within the interim limits for all individual groundwater and surface water measurements (Appendix F, Tables F1-F2).

For the seven wastewater samples (Appendix F, Table F-3), EIS compounds where the mean percent recovery is reported below the target criteria included D_3-NMeFOSAA, D_5-NEtFOSAA, D_7-NMeFOSE, and D_9-NEtFOSE. In addition, individual samples where for which EIS recoveries were below 50% included $^{13}C_4$-PFBA, $^{13}C_5$-PFPeA, $^{13}C_8$-PFOSA, $^{13}C_2$-PFDoA, $^{13}C_2$-PFTeDA, D_3-NMeFOSA, D_5-NEtFOSA.

For the three individual landfill leachate samples (Appendix F, Table F-4), the mean percent recovery of the three samples was within the target criteria, with the exception of $^{13}C_2$-PFTeDA. For one of the three leachate samples, several EIS compounds were below the criteria including $^{13}C_4$-PFBA, $^{13}C_5$-PFPeA, $^{13}C_2$-PFDoA, $^{13}C_2$-PFTeDA, D_3-NMeFOSAA, D_5-NEtFOSAA, D_7-NMeFOSE, and D_9-NEtFOSE.

The high and low EIS recoveries were only observed with the wastewater and landfill leachate samples (Table 11-1).

Table 11-1. All Aqueous Sample EIS Spike Recoveries (groundwater, surface water, wastewater, leachate)

Compound	Acronym	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	Highest % Recovery	Lowest % Recovery	RSD	SD	2x SD
Perfluoro-n-[$^{13}C_4$]butanoic acid	$^{13}C_4$ -PFBA	142	4	81.9	97.21	8.99	15.9	13.02	26.05
Perfluoro-n-[$^{13}C_5$]pentanoic acid	$^{13}C_5$ -PFPeA	70.8	4	87.89	103.4	38.88	13.34	11.73	23.45
Perfluoro-n-[1,2,3,4,6-$^{13}C_5$]hexanoic acid	$^{13}C_5$ -PFHxA	35.4	4	91.19	96.9	72.87	2.7	2.46	4.92
Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid	$^{13}C_4$ -PFHpA	35.4	4	89.89	94.73	76.61	2.39	2.15	4.3
Perfluoro-n-[$^{13}C_8$]octanoic acid	$^{13}C_8$ -PFOA	35.8	4	92.35	95.05	87.27	0.81	0.75	1.49
Perfluoro-n-[$^{13}C_9$]nonanoic acid	$^{13}C_9$ -PFNA	17.7	4	91.22	95.19	82.37	1.59	1.45	2.89
Perfluoro-n-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid	$^{13}C_6$ -PFDA	17.7	4	87.48	92.71	70.56	3.31	2.9	5.79
Perfluoro-n-[1,2,3,4,5,6,7-$^{13}C_7$]undecanoic acid	$^{13}C_7$ -PFUnA	17.7	4	85.66	94.49	56.25	6.46	5.53	11.06
Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid	$^{13}C_2$ -PFDoA	17.7	4	72.27	86.74	33.68	13.68	9.88	19.77
Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid	$^{13}C_2$ -PFTeDA	17.7	4	64.04	153.44	16.96	26.17	16.76	33.51
Perfluoro-1-[2,3,4-$^{13}C_3$]butanesulfonic acid	$^{13}C_3$ -PFBS	35.4	4	93.55	99.66	72.28	4.7	4.39	8.79
Perfluoro-1-[1,2,3-$^{13}C_3$]hexanesulfonic acid	$^{13}C_3$ -PFHxS	35.8	4	92.05	94.78	79.44	1.57	1.45	2.89
Perfluoro-1-[$^{13}C_8$]octanesulfonic acid	$^{13}C_8$ -PFOS	35.8	4	91.33	96.04	66.75	3.58	3.27	6.55
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]hexanesulfonic acid	$^{13}C_2$ -4:2FTS	71.2	4	124.2	199	80.57	14.82	18.41	36.82

Table 11-1. All Aqueous Sample EIS Spike Recoveries (groundwater, surface water, wastewater, leachate)

Compound	Acronym	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	Highest % Recovery	Lowest % Recovery	RSD	SD	2x SD
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]octanesulfonic acid	$^{13}C_2$ -6:2FTS	70.8	4	105.29	182.5	64.02	16.36	17.22	34.45
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]decanesulfonic acid	$^{13}C_2$ -8:2FTS	70.8	4	93.4	139.33	65.08	8.38	7.83	15.66
Perfluoro-1-[$^{13}C_8$]octanesulfonamide	$^{13}C_8$ -PFOSA	35.4	4	77.6	92.62	26.68	15.42	11.97	23.94
N-methyl-d_3-perfluoro-1-octanesulfonamide	D_3-NMeFOSAA	35.4	4	59.82	74.05	13.51	16.42	9.82	19.64
N-ethyl-d_5-perfluoro-1-octanesulfonamide	D_5-NEtFOSAA	35.4	4	58.44	70.18	11.93	16.53	9.66	19.31
N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	D_3-NMeFOSA	70.8	4	87.42	113.46	21.46	7.32	6.4	12.8
N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	D_5-NEtFOSA	70.8	4	85.78	105.67	11.68	8.21	7.04	14.09
N-methyl-d_7-perfluorooctanesulfonamidoethanol	D_7-NMeFOSE	354	4	61.92	77.01	10.99	18.55	11.48	22.97
N-ethyl-d_9-perfluorooctane sulfonamidoethanol	D_9-NEtFOSE	354	4	57.63	72.61	7.99	19.62	11.31	22.61
Tetrafluoro-2-heptafluoropropoxy-$^{13}C_3$-propanoic acid	$^{13}C_3$ -HFPO-DA	142	4	101.07	112.5	91.83	2.02	2.04	4.08

Percent Recovery < 50%; Percent Recovery > 200%; RSD = Relative Standard Deviation; SD = Standard Deviation; 2x SD = two times Standard Deviation

11.1.2 Solid EIS Results

Table 11-2 provides the summary statistics for soil, sediment, and biosolids. For the combined data for all three matrices, the mean percent recoveries are within the target range, with the single exception of D_5-NEtFOSAA.

For each individual soil samples (Appendix F, Table F-5), the EIS recoveries were generally acceptable. The two exceptions were D_3-NMeFOSAA and D_5-NEtFOSAA where the mean recoveries were between 37–49%. Recoveries in the EIS-spiked individual sediment samples were within the interim criteria range for all EIS compounds except for D_5-NEtFOSAA (Appendix F, Table F-6). However, that exception occurred in only one of the three tested sediments.

For the biosolid samples (Appendix F, Table F-7), eight EIS compounds showed recoveries outside the interim criteria where $^{13}C_4$-PFBA, $^{13}C_2$-PFDoA, $^{13}C_2$-PFTeDA, D_5-NEtFOSAA, D_7-NMeFOSE, and D_9-NEtFOSE had recoveries below 50% recovery; no compound exceeded the upper criteria limit.

11.1.3 Tissue EIS Results

Tissue samples followed a similar pattern as the biosolids (Table 11-3). For the combined fish and clam tissue samples, the mean percent recoveries for four EIS compounds were outside the interim criteria. Of those, D_3-NMeFOSAA, D_5-NEtFOSAA, D_7-NMeFOSE, and D_9-NEtFOSE were below 50% recovery, and $^{13}C_2$-4:2FTS was above 200% recovery. Additional EIS compounds for which individual sample results were outside the criteria (Appendix F, Table F-8) included $^{13}C_2$-PFTeDA, $^{13}C_2$-6:2FTS, and $^{13}C_2$-8:2FTS.

11.2 IIS Results

Appendix G includes 11 summary tables per matrix of the IIS spike recoveries. IIS spike recoveries are an indicator of instrument performance. IIS summaries were generated for each of the eight environmental matrices: groundwater, wastewater, surface water, landfill leachate, soils, sediment, biosolids and tissue. Each table includes the mean of the matrix mean percent recoveries, RSD, SD, 2× SD, and 3× SD of the matrix mean percent recoveries for the seven IIS compounds for aqueous, solid, and tissue samples.

Summaries by aqueous, solid, and tissue matrices derived from the Appendix G tables are shown in Tables 11-4 through 11-6. The Study Plan did not identify acceptance criteria for the IIS results; those will be established during the MLVS. For discussion purposes IIS below 50% or greater than 200% will be included here.

The summary tables include the mean of the matrix mean concentrations; mean of the matrix mean percent recoveries; lowest and highest percent recoveries, percent RSD, SD, and 2×SD of the matrix mean percent recoveries for the seven IIS compounds. A difference in the report tables, relative to Appendix G, is that the appendix summary for all solids included soils, sediment, biosolids, and tissues. For this report, the "solids" table was recalculated and the soils/sediment/biosolids (Table 11-5) is presented separately from the tissue samples (Table 11-6). In addition, the tables in Appendix G do not contain the minimum and maximum IIS recoveries observed; those are included here.

Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021).

11.2.1 Aqueous IIS Results

Table 11-4 shows the IIS recovery results averaged across the groundwater, surface water, wastewater, and landfill leachate samples. IIS mean percent recovery for all compounds was between 68 – 76%. For the individual matrices, IIS recoveries for groundwater and surface water were all between 50–150%. For wastewater samples the average percent recoveries ranged from 65–82%, but for one or more of the individual samples the IIS recoveries were below 50% for ^{13}C3-PFHxA, $^{13}C_2$-PFDA, and $^{13}C_4$-PFOS. In the landfill leachate individual samples low recoveries were reported for 5 of the 7 IIS compounds: $^{13}C_3$-PFBA, $^{13}C_2$-PFHxA, $^{13}C_4$-PFOA, $^{18}O_2$-PFHxS, and $^{13}C_4$-PFOS.

11.2.2 Solid IIS Results

Table 11-5 includes the summary statistics for soil, sediment, and biosolids. For the combined data for all three matrices, the mean percent, lowest, and highest recoveries are all within 50–150%.

11.2.3 Tissue IIS Results

Tissue sample IIS results are presented in Table 11-6. The mean and highest percent recoveries were all within the target recovery limits. The lowest percent recoveries all were within the range with two IIS compounds below 50%; $^{13}C_2$-PFHxA and $^{13}C_2$-PFDA.

.

Table 11-2. All Solid Sample EIS Spike Recoveries (soils, sediment, biosolids)

Compound	Acronym	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	Highest % Recovery	Lowest % Recovery	RSD[1]	SD[2]	2x SD[3]
Perfluoro-n-[$^{13}C_4$]butanoic acid	$^{13}C_4$ -PFBA	32.61	3	78.98	101.9	27.03	45.5	35.90	71.81
Perfluoro-n-[$^{13}C_5$]pentanoic acid	$^{13}C_5$ - PFPeA	29.88	3	88.42	97.87	52.2	9.43	8.34	16.67
Perfluoro-n-[1,2,3,4,6-$^{13}C_5$]hexanoic acid	$^{13}C_5$ - PFHxA	14.96	3	96.20	99.03	89.78	1.14	1.10	2.20
Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid	$^{13}C_4$ - PFHpA	8.16	3	92.90	97	89.11	0.66	0.62	1.23
Perfluoro-n-[$^{13}C_8$]octanoic acid	$^{13}C_8$ - PFOA	8.17	3	96.99	100.81	94.56	0.57	0.55	1.10
Perfluoro-n-[$^{13}C_9$]nonanoic acid	$^{13}C_9$ - PFNA	7.55	3	96.64	100.07	94.52	0.33	0.32	0.63
Perfluoro-n-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid	$^{13}C_6$ - PFDA	4.08	3	96.75	98.36	95.63	0.24	0.23	0.46
Perfluoro-n-[1,2,3,4,5,6,7-$^{13}C_7$]undecanoic acid	$^{13}C_7$ - PFUnA	4.08	3	97.58	106.06	73.38	11.19	10.92	21.83
Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid	$^{13}C_2$ - PFDoA	4.08	3	81.24	96.33	37.19	26.36	21.42	42.84
Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid	$^{13}C_2$ - PFTeDA	4.08	3	72.38	94.09	27.13	40.24	29.12	58.25
Perfluoro-1-[2,3,4-$^{13}C_3$]butanesulfonic acid	$^{13}C_3$ -PFBS	4.76	3	101.95	107.08	98.15	1.61	1.64	3.28
Perfluoro-1-[1,2,3-$^{13}C_3$]hexanesulfonic acid	$^{13}C_3$ - PFHxS	8.17	3	98.21	102.33	93.8	1.94	1.90	3.80
Perfluoro-1-[$^{13}C_8$]octanesulfonic acid	$^{13}C_8$ -PFOS	8.24	3	100.46	104.09	92.85	1.57	1.57	3.15
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]hexanesulfonic acid	$^{13}C_2$ - 4:2FTS	9.60	3	138.91	195.92	120.42	12.84	17.84	35.68
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]octanesulfonic acid	$^{13}C_2$ - 6:2FTS	16.38	3	105.03	113.58	91.63	4.96	5.21	10.42

Table 11-2. All Solid Sample EIS Spike Recoveries (soils, sediment, biosolids)

Compound	Acronym	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	Highest % Recovery	Lowest % Recovery	RSD[1]	SD[2]	2x SD[3]
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]decanesulfonic acid	$^{13}C_2$ - 8:2FTS	16.32	3	116.11	153.33	104.65	12.64	14.67	29.35
Perfluoro-1-[$^{13}C_8$]octanesulfonamide	$^{13}C_8$ - PFOSA	14.96	3	102.90	110.77	95.52	4.34	4.47	8.94
N-methyl-d_3-perfluoro-1-octanesulfonamide	D_3-NMeFOSAA	8.16	3	56.81	71.3	44.19	10.82	6.15	12.29
N-ethyl-d_5-perfluoro-1-octanesulfonamide	D_5-NEtFOSAA	8.16	3	44.80	60.13	30.87	15.04	6.74	13.47
N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	D_3-NMeFOSA	9.52	3	104.39	125.75	76.55	4.18	4.36	8.72
N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	D_5-NEtFOSA	16.32	3	110.14	136.08	80	6.74	7.43	14.85
N-methyl-d_7-perfluorooctanesulfonamidoethanol	D_7-NMeFOSE	27.20	3	69.51	93.19	20.71	31.14	21.64	43.29
N-ethyl-d_9-perfluorooctane sulfonamidoethanol	D_9-NEtFOSE	81.60	3	60.28	84.64	24.27	38.17	23.01	46.03
Tetrafluoro-2-heptafluoropropoxy-$^{13}C_3$-propanoic acid	$^{13}C_3$-HFPO-DA	73.44	3	98.38	106.17	89.36	2.78	2.73	5.47

Percent Recovery < 50%
Percent Recovery > 200%
RSD = Relative Standard Deviation
SD = Standard Deviation
2x SD = two times Standard Deviation

Table 11-3. All Tissue Sample EIS Spike Recoveries (fish and bivalves)

Compound	Acronym	Mean Concentration Spike (µg/kg wet weight)	Number EIS Spikes (n =)	Mean % Recovery	Highest % Recovery	Lowest % Recovery	RSD	SD	2x SD
Perfluoro-n-[$^{13}C_4$]butanoic acid	$^{13}C_4$ -PFBA	20.10	3	91.96	98.52	84.01	8.0	7.36	14.71
Perfluoro-n-[$^{13}C_5$]pentanoic acid	$^{13}C_5$ - PFPeA	10.00	3	95.88	107.34	86.44	11.05	10.59	21.19
Perfluoro-n-[1,2,3,4,6-$^{13}C_5$]hexanoic acid	$^{13}C_5$ - PFHxA	5.02	3	94.04	94.94	92.31	1.60	1.50	3.01
Perfluoro-n-[1,2,3,4-$^{13}C_4$]heptanoic acid	$^{13}C_4$ - PFHpA	5.02	3	85.12	93.01	79.69	8.21	6.99	13.98
Perfluoro-n-[$^{13}C_8$]octanoic acid	$^{13}C_8$ - PFOA	5.07	3	93.48	95.25	90.46	2.81	2.63	5.25
Perfluoro-n-[$^{13}C_9$]nonanoic acid	$^{13}C_9$ - PFNA	2.51	3	93.48	98.02	90.3	4.32	4.04	8.08
Perfluoro-n-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid	$^{13}C_6$ - PFDA	2.51	3	90.39	97.19	83.28	7.70	6.96	13.92
Perfluoro-n-[1,2,3,4,5,6,7-$^{13}C_7$]undecanoic acid	$^{13}C_7$ - PFUnA	2.51	3	83.57	90.94	71.19	12.90	10.78	21.56
Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid	$^{13}C_2$ - PFDoA	2.51	3	73.14	95.83	53.52	29.15	21.32	42.64
Perfluoro-n-[1,2-$^{13}C_2$]tetradecanoic acid	$^{13}C_2$ - PFTeDA	2.51	3	57.13	101.68	31.36	67.81	38.74	77.48
Perfluoro-1-[2,3,4-$^{13}C_3$]butanesulfonic acid	$^{13}C_3$ -PFBS	5.02	3	92.54	97.87	88.83	5.12	4.74	9.48
Perfluoro-1-[1,2,3-$^{13}C_3$]hexanesulfonic acid	$^{13}C_3$ - PFHxS	5.07	3	98.50	98.64	98.39	0.13	0.13	0.26
Perfluoro-1-[$^{13}C_8$]octanesulfonic acid	$^{13}C_8$ -PFOS	5.07	3	97.12	103.37	91.9	5.97	5.80	11.60
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]hexanesulfonic acid	$^{13}C_2$ - 4:2FTS	10.10	3	200.51	214.83	192.33	6.21	12.45	24.90

Table 11-3. All Tissue Sample EIS Spike Recoveries (fish and bivalves)

Compound	Acronym	Mean Concentration Spike (µg/kg wet weight)	Number EIS Spikes (n =)	Mean % Recovery	Highest % Recovery	Lowest % Recovery	RSD	SD	2x SD
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]octanesulfonic acid	$^{13}C_2$ - 6:2FTS	10.00	3	174.83	229.75	145.25	27.23	47.61	95.21
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-$^{13}C_2$]decanesulfonic acid	$^{13}C_2$ - 8:2FTS	10.00	3	173.78	220.25	136.17	24.59	42.73	85.47
Perfluoro-1-[$^{13}C_8$]octanesulfonamide	$^{13}C_8$ - PFOSA	5.02	3	91.87	95.52	87.44	4.46	4.10	8.19
N-methyl-d_3-perfluoro-1-octanesulfonamide	D_3-NMeFOSAA	5.02	3	24.18	37.65	8.08	61.86	14.96	29.92
N-ethyl-d_5-perfluoro-1-octanesulfonamide	D_5-NEtFOSAA	5.02	3	19.75	30.11	7.51	57.81	11.42	22.83
N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	D_3-NMeFOSA	10.00	3	123.59	138.53	106.41	13.09	16.18	32.36
N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	D_5-NEtFOSA	10.00	3	123.62	151.09	78.52	31.84	39.36	78.72
N-methyl-d_7-perfluorooctanesulfonamidoethanol	D_7-NMeFOSE	50.20	3	15.93	30.27	5.2	81.09	12.92	25.84
N-ethyl-d_9-perfluorooctane sulfonamidoethanol	D_9-NEtFOSE	50.20	3	14.05	29.23	0.37	103.11	14.49	28.97
Tetrafluoro-2-heptafluoropropoxy-$^{13}C_3$-propanoic acid	$^{13}C_3$ - HFPO-DA	20.10	3	94.44	96.31	92.52	5.13	4.84	9.70

Percent Recovery < 50%
Percent Recovery > 200%
RSD = Relative Standard Deviation
SD = Standard Deviation
2x SD = two times Standard Deviation

Table 11-4. All Aqueous Sample IIS Spike Recoveries (groundwater, surface water, wastewater, leachate)

Matrices	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD	2x SD
Aqueous	$^{13}C_3$-PFBA	4	75.77	43.68	111.3	10.25	7.77	15.53
Aqueous	$^{13}C_2$-PFHXA	4	68.3	22.24	106.3	18.60	12.70	25.41
Aqueous	$^{13}C_4$-PFOA	4	72.95	47.16	108.6	9.68	7.07	14.13
Aqueous	$^{13}C_5$-PFNA	4	75.36	50.33	114.15	7.47	5.63	11.25
Aqueous	$^{13}C_2$-PFDA	4	71.75	15.84	112.2	9.96	7.15	14.30
Aqueous	$^{18}O_2$-PFHXS	4	73.24	46.51	108.9	9.58	7.02	14.03
Aqueous	$^{13}C_4$-PFOS	4	71.02	46.17	105.5	7.52	5.34	10.67

Percent Recovery < 50%
Percent Recovery > 150%
RSD = Relative Standard Deviation
SD = Standard Deviation
2x SD = two times Standard Deviation

Table 11-5. All Solid Sample IIS Spike Recoveries (soils, sediment, biosolids)

Matrices	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD	2x SD
Solid	$^{13}C_3$-PFBA	3	67.72	54.24	89.13	6.43	4.36	8.71
Solid	$^{13}C_2$-PFHXA	3	66.49	51.73	89.66	7.42	4.93	9.87
Solid	$^{13}C_4$-PFOA	3	68.13	54.46	89.21	6.76	4.61	9.21
Solid	$^{13}C_5$-PFNA	3	70.26	58.62	94.26	7.11	5.00	9.99
Solid	$^{13}C_2$-PFDA	3	70.28	54.92	90.97	8.55	6.01	12.02
Solid	$^{18}O_2$-PFHXS	3	67.79	53.23	86.63	7.06	4.79	9.57
Solid	$^{13}C_4$-PFOS	3	67.97	57.51	86.24	6.96	4.73	9.46

Percent Recovery < 50%
Percent Recovery > 150%
RSD = Relative Standard Deviation
SD = Standard Deviation
2x SD = two times Standard Deviation

Table 11-6. All Tissue Sample IIS Spike Recoveries (fish and bivalves)

Matrices	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	Lowest % Recovery	Highest % Recovery	RSD[1]	SD[2]	2x SD[3]
Tissue	$^{13}C_3$-PFBA	36	70.26	50.84	81.52	7.89	5.54	11.09
Tissue	$^{13}C_2$-PFHXA	36	58.74	40.73	79.97	19.35	11.37	22.73
Tissue	$^{13}C_4$-PFOA	36	69.45	50.71	81.74	9.53	6.62	13.24
Tissue	$^{13}C_5$-PFNA	36	71.97	52.08	88.15	11.10	7.99	15.97
Tissue	$^{13}C_2$-PFDA	36	67.39	46.61	85.21	19.36	13.04	26.09
Tissue	$^{18}O_2$-PFHXS	36	70.08	51.45	80.03	8.12	5.69	11.39
Tissue	$^{13}C_4$-PFOS	36	69.52	51.87	84.51	10.30	7.16	14.33

Percent Recovery < 50%
Percent Recovery > 150%
RSD = Relative Standard Deviation
SD = Standard Deviation
2x SD = two times Standard Deviation

12 HOLDING TIME STUDY

The EPA provided this summary and the holding time report found in Appendix K. The holding time study was designed and carried out cooperatively by the EPA and the DoD. The goal was to provide EPA with results to establish the holding times and preservation conditions for PFAS analytes that may be added to Table II at 40 CFR Part 136 in conjunction with the expected proposal and promulgation of EPA Method 1633.

The 2020 holding time study was performed by SGS AXYS. The goal of the study was to assess how long environmental samples and extracts of samples can be held, at various temperatures, before PFAS degrade, become unextractable, or otherwise change concentrations so that the samples are no longer representative of the matrices from which they are derived. The study included one groundwater, one soil, one sediment, and one biosolids sample matrix, as well as extracts of each of these matrices. The matrices were spiked at the mid-level concentrations and analyzed with three replicate samples for each time increment. The samples were put into the appropriate sample containers as though they were field samples.

SGS AXYS performed a separate holding time in 2018, which included reagent water, one river surface water and two wastewater effluents. That study was designed to cover different sample containers, three storage temperatures, a holding time of up to 180 days and tested 29 of the 40 PFAS evaluated with the SLVS. EPA and DoD decided to incorporate the 2018 SGS AXYS holding time study data into the data acquired under the SLVS DoD study.

Appendix K includes the results of both studies and the evaluation of compound stability. Based on their analysis of the data gathered, EPA determined that aqueous samples may be held for up to 28 days when stored at 4ºC, with the caveat that the study data showed signs of transformation of some precursors to other PFAS when the sample is stored beyond 7 days. This is likely to increase the observed concentrations of some PFAS if precursors are present in the sample. For greater stability of PFAS, the aqueous samples can be stored at -20ºC for up to 90 days.

For biosolids, all PFAS were stable for up to 90 days when stored at either 4ºC or -20ºC. Because microbiological activity in biosolids samples at 0–6 ºC may lead to production of gases which may cause the sample to be expelled from the container when it is opened, as well as producing noxious odors, EPA strongly recommends that samples be frozen if they need to be stored for more than a few days before extraction.

For solids samples, EPA found that samples could be held for 90 days when stored at either 4ºC or -20ºC, except when analyzing for NFDHA, which was not stable at either of the temperatures tested and must be analyzed as soon as possible after collection if it is an analyte of concern.

Once the PFAS are extracted, they are stable in the extracts for up to 90 days when stored at 4ºC, with the exception of NFDHA in solids and the ether sulfonates in aqueous samples, which are only stable for 28 days. (Although the extracts were analyzed at 29 days, EPA has decided to maintain consistency with holding times for other analytes. The holding times for each matrix and storage temperature are summarized in Table 12-1.

Table 12-1. Summary of Holding Times per Matrix and Storage Temperature

Matrices	Stored at 4 ºC		Stored at -20 ºC	
	Holding Time	**Exceptions**	**Holding Time**	**Exceptions**
Samples				
Aqueous	28 days	Precursor degradation after 7 days	90 days	None
Solids	90 days	NFDHA - analyze as soon as possible	90 days	NFDHA - analyze as soon as possible
Biosolid	90 days		90 days	None
Extracts				
Aqueous	90 days	28 days for ether sulfonates	NA	None
Solids	90 days	28 days for NFDHA	NA	None
Biosolid	90 days		NA	None

NA = not applicable. Extract holding time was not tested at -20ºC.

13 CONCLUSIONS

The data generated during the SLVS demonstrated that the requirements contained in the Study Plan for the mass calibration, mass calibration verification, initial calibration, and initial demonstration of capability can be routinely achieved. It is appropriate that these criteria are included in the draft published method. The study method requirements for method blanks, calibration verifications, ISCs, and qualitative standard analysis were consistently met with few exceptions, therefore, these criteria are recommended for use in the MLVS. The failure of some of these standards to meet the EIS recovery criterion proved to be inconsequential with respect to analyte recoveries. Because of this, it is recommended that the acceptance criteria for standards be adjusted accordingly. Criteria for EIS recoveries in instrument blanks, calibration verifications, ISCs, and qualitative standards will be determined in the MLVS.

The IPR, MDL, LOQVER, OPR, unspiked sample, and matrix spike results were evaluated to determine if any target analytes should be eliminated for a particular matrix type. Overall, the method performance was very good. Aqueous and solid matrix results indicate that the method is appropriate for the determination of all 40 target analytes using the 24 EIS and 7 IIS compounds utilized in the study for quantification. Tissue matrix sample results indicate that use of this method for the determination of NMeFOSE and NEtFOSE consistently yields inaccurate results. This is demonstrated through the consistently low recoveries of their associated EIS compounds and the low target analyte responses. These analytes, in particular, demonstrated that there is a limitation for the EIS compound's ability to appropriately adjust for target analyte recoveries. When EIS compound recoveries fell below 10%, target analyte recoveries were over adjusted, resulting in recoveries well over 150%. This suggests that the lower limit of acceptable EIS recoveries should be set near or at 10%. With respect to IIS recoveries, study data indicates IIS recoveries routinely can meet a 50–150% criterion for most compounds, but not all. EIS and IIS recoveries limits will be determined in the MLVS.

The method was tested on a wide variety of matrices at varying concentrations and performed better than most EPA non-drinking water methods for organic molecules. EPA will develop method performance criteria from the results of the MLVS.

14 REFERENCES

Anderson RH, Thompson T, Stroo HF, Leeson A. 2020. U.S. Department of Defense–funded fate and transport research on per- and polyfluoroalkyl substances at aqueous film-forming foam–impacted sites. Environ Toxicol Chem 40:37–43.

ITRC. 2020. *Per- and Polyfluoroalkyl Substances (PFAS)*. The Interstate Technology and Regulatory Council (ITRC) Per-and Polyfluoroalkyl Substances (PFAS) Team. April 2020. https://pfas-1.itrcweb.org.

Leeson A, Thompson T, Stroo HF, Anderson RH, Speicher J, Mills MA, Willey J, Coyle C, Ghosh R, Lebrón C, Patton C. 2020. Identifying and managing aqueous film-forming foam-derived per- and polyfluoroalkyl substances in the environment. Environ Toxicol Chem 40:24-36.

U.S. Environmental Protection Agency (EPA). 2016a. Drinking Water Health Advisory for Perfluorooctanoic Acid (PFOA). USEPA, Office of Water, Health and Ecological Criteria Division, Washington, DC. EPA 822-R-16-005. 2016.

U.S. Environmental Protection Agency (EPA). 2016b. Drinking Water Health Advisory for Perfluorooctane Sulfonate (PFOS). USEPA, Office of Water, Health and Ecological Criteria Division, Washington, DC. EPA 822-R-16-004. 2016.

U.S. Environmental Protection Agency (EPA). 2018. Method 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS) U.S. Environmental Protection Agency, Office of Research and Development, National Center for Environmental Assessment, Washington, DC, 2018.

U.S. Environmental Protection Agency (EPA). 2018b. *Protocol for Review and Validation of New Methods for Regulated Organic and Inorganic Analytes in Wastewater Under EPA's Alternate Test Procedure Program*. U.S. Environmental Protection Agency. Office of Water, Engineering and Analysis Division. EPA 821-B-18-001. February. Available online at https://www.epa.gov/sites/default/files/2018-03/documents/chemical-new-method-protocol_feb-2018.pdf.

U.S. Environmental Protection Agency (EPA). 2020. Method 533: Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry. USEPA Office of Water, Office of Groundwater and Drinking Water, Standards and Risk Management Division, Cincinnati, OH. EPA 815-B-19-020

U.S. Environmental Protection Agency (EPA). 2021. *Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS*. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Engineering and Analysis Division, Washington, DC 20460. EPA 821-D-21-001. August. Available online at https://www.epa.gov/newsreleases/epa-announces-first-validated-laboratory-method-test-pfas-wastewater-surface-water.

U.S. Department of Defense (DoD). 2021. *Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories. Based on ISO/IEC 17025:2005(E), ISO/IEC 17025:2017(E), and the NELAC Institute (TNI) Standards, Volume 1, (September 2009)*. DoD Quality Systems Manual Version 5.4. October. https://www.denix.osd.mil/edqw/home/.

Code of Federal Regulations. Appendix B to Title 40, Part 136 - Definition and Procedure for the Determination of the Method Detection Limit - Revision 2.

Appendix A

Single-Laboratory Study Plan and Analytical Method Standard Operating Procedure

3000018243
Attachment B

Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

Contract: W912DY-17-D-0004
Delivery Order: W912DY19F1365

Prepared for:

Dr. Andrea Leeson
Program Manager for Environmental Restoration
Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-3605
Phone: 571-372-6398
E-Mail: Andrea.Leeson.civ@mail.mil

Prepared by:

SERDP/ESTCP PFAS Method Validation Team
Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-3605
Phone: 571-372-6398

Prepared Under Contract:
U.S. Army Engineering and Support Center
Huntsville Engineering & Support Center
U.S. Army Corps of Engineers
Contract No.: W912DY-17-D-0004
Task Order No.: W912DY19F1365
By
HydroGeoLogic, Inc.
11107 Sunset Hills Road, Suite 400
Reston, Virginia 20190-5375

This page was intentionally left blank.

LEESON.ANDREA.1291529050 Digitally signed by LEESON.ANDREA.1291529050 Date: 2020.05.26 14:54:51 -04'00'

ESTCP Study Supervisor — Date

Charles Coyle — 05/01/2020

USACE EM-CX Study Manager — Date

MARC MILLS Digitally signed by MARC MILLS Date: 2020.05.13 11:48:28 -04'00'

EPA Technical Manager — Date

Janice Willey — 05/04/2020

NAVSEA LQAO QC Manager — Date

05/04/2020

AFCEC Study Evaluation Manager — Date

Powell, John Digitally signed by Powell, John Date: 2020.05.06 19:17:30 -05'00' — 05/06/2020

HydroGeoLogic, Inc. Project Chemist — Date

Derek R. Anderson Digitally signed by Derek R. Anderson Date: 2020.05.06 11:40:02 -04'00' — 05/06/2020

HydroGeoLogic, Inc. Program Manager — Date

DISCLAIMER

Mention of company names, trade names, or commercial products does not constitute endorsement or recommendation for use.

DISTRIBUTION LIST

Study Plan Recipients	Title	Organization	Telephone Number (optional)	E-mail Address or Mailing Address
Dr. Andrea Leeson	Study Supervisor	ESTCP/SERDP	(720) 491-8149	andrea.leeson.civ@mail.mil
Rebecca Terry	Contracting Officer Representative/Project Manager	USACE CEHNC		Rebecca.K.Terry@usace.army.mil
Charles Coyle	Study Manager	USACE EM-CX	(402) 697-2578	Charles.G.Coyle@usace.army.mil
Dr. Marc Mills	EPA Technical Manager	EPA ORD	(513) 569-7322	mills.marc@epa.gov
Dr. Richard (Hunter) Anderson	Study Evaluation Manager	AFCEC	(210) 395-0625	Richard.anderson.55@us.af.mil
Janice Willey	QA Manager	NAVSEA LQAO	(843) 737-1574	Janice.willey@navy.mil
Adrian Hanley	EPA OW Senior Chemist	EPA OW	(202) 564-1564	Hanley.Adrian@epa.gov
Schatzi Fitz-James	EPA OLEM Senior Chemist	EPA OLEM	(703) 603-8778	fitzjames.schatzi@epa.gov
Dr. Coreen Hamilton	SGS AXYS Project Manager	SGS AXYS	(250) 655-5802	Coreen.Hamilton@sgs.com
Dale Hoover	SGS AXYS QA Director	SGS AXYS	(250) 655-5805	Dale.hoover@sgs.com
John Powell	Contractor Project Chemist	HydroGeoLogic, Inc.	(913) 378-2315	jpowell@hgl.com
Dr. Mindy Vanderford	Contractor Project Manager	HydroGeoLogic, Inc.	(713) 865-2223	mvanderford@hgl.com
Derek Anderson	Contractor Deputy Program Manager	HydroGeoLogic, Inc.	(706) 372-5138	danderson@hgl.com
Lynn Kessler	Contractor PFAS Business Line Manger	HydroGeoLogic, Inc.	(703) 736-4518	lkessler@hgl.com

This page was intentionally left blank.

TABLE OF CONTENTS

LIST OF ATTACHMENTS

LIST OF TABLES

LIST OF ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
%	percent
µg/kg	micrograms per kilogram
AFCEC	Air Force Civil Engineer Center
ASTM	American Society for Testing and Materials
CEHNC	USACE Huntsville Engineering and Support Center
CFR	Code of Federal Regulations
DL	detection limit
DoD	U.S. Department of Defense
DQO	data quality objectives
EDD	electronic data deliverable
EIS	Extracted Internal Standard
ELAP	Environmental Laboratory Accreditation Program
EM-CX	Environmental and Munitions Center of Expertise
EPA	U.S. Environmental Protection Agency
ESTCP	Environmental Security Technology Certification Program
FOSE	perfluorooctanesulfonamidoethanol
g	gram
HGL	HydroGeoLogic, Inc.
ICAL	initial calibration
IDC	initial demonstration of capability
IIS	Injection Internal Standard
IPR	initial precision and recovery
LC-MS/MS	liquid chromatography mass spectroscopy/mass spectroscopy
LCS	laboratory control sample
LLOQ	lower limit of quantitation
LOD	limit of detection
LOQ	limit of quantitation
LQAO	Laboratory Quality and Accreditation Office

MDL	method detection limit
mg/L	milligram per liter
mL	milliliter
ML	multi-laboratory
MVT	SERDP/ESTCP PFAS Method Validation Team
NAPT	North American Proficiency Testing Program
NAVSEA	Naval Sea Systems Command
NPDES	National Pollutant Discharge Elimination System
OLEM	Office of Land and Emergency Management
OPR	on-going precision and recovery
ORD	Office of Research and Development
OW	Office of Water
PDF	portable document format
PFAS	per- and polyfluoroalkyl substances
PFOA	perfluorooctanoic acid
PFOS	perfluorooctanesulfonic acid
ppb	parts per billion
ppt	parts per trillion
r^2	correlation coefficient
%R	percent recovery
%RSD	percent relative standard deviation
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality Systems Manual
SERDP	Strategic Environmental Research and Development Program
SL	single laboratory
SOP	standard operating procedure
SOW	statement of work
SPE	solid-phase extraction
SS	suspended solids
TDS	total dissolved solids
TSS	total suspended solids
USACE	U.S. Army Corps of Engineers

This page was intentionally left blank.

Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

413 BACKGROUND

Per- and polyfluoroalkyl substances (PFAS) have been widely used for decades in industrial processes and consumer products. Due to their environmental persistence and mobility, the presence of some PFAS has been routinely detected at trace levels in various environmental media. U.S. Environmental Protection Agency (EPA) has published a recommended Health Advisory Level for two PFAS, perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS), and several states have established or promulgated standards for these and other PFAS. Currently there are no published methods for the scope of media proposed for this study. There exists an immediate need to evaluate these media (i.e., groundwater, wastewater, surface water, soil, sediment, biosolids, landfill leachate, and tissue) using a published method that is single- and multi-laboratory validated that can achieve the sensitivity and defensibility needed to support environmental decision making. The goal of this study is to single-laboratory (SL) validate a method for the analysis of PFAS in these media using isotope dilution liquid chromatography mass spectroscopy/mass spectroscopy (LC-MS/MS). The SL validation study will be used to make any necessary adjustments to the method prior to a multi-laboratory (ML) validation effort, that is proposed to follow. The end goal is to submit a method and supporting data package to the EPA Office of Water (OW) for consideration as a 1600-Series method, including the information that EPA Office of Land and Emergency Management (OLEM) needs to consider an EPA SW-846 method. OW will distribute the method/data package to OLEM.

The SL study will be conducted in the following three phases, as described in Sections 4.1 through 4.3 below.

- Phase 1: Calibration, Initial Demonstration of Capability (IDC), Method Detection Limit (MDL) Study, and Limit of Detection (LOD) and Limit of Quantitation (LOQ) Verifications
- Phase 2: Method Evaluation in Wastewaters, Surface Water, Landfill Leachate, and Groundwater Matrices
- Phase 3: Method Evaluation in Biosolid, Soil, Sediment, and Fish Tissue Matrices

A Holding Time Study will be conducted using matrices designated for Phases 2 and 3, either concurrent with or after Phase 2 and 3 analyses have been performed.

The SL validation study is funded through a contract with the U.S. Army Engineering and Support Center, Huntsville Engineering and Support Center (CEHNC), U.S. Army Corps of Engineers (USACE) under Contract Number W912DY-17-D-0004, Task Order Number W912DY19F1365, with HydroGeoLogic, Inc. (HGL) as the oversight Contractor for the project. Funding is also proved by OW and OLEM. The Study Plan for Single-Laboratory Validation of PFAS was developed by the Strategic Environmental Research and Development Program (SERDP)/Environmental Security Technology Certification Program (ESTCP) PFAS Method Validation Team (MVT).

This page was intentionally left blank.

513 STUDY OBJECTIVES

The main objective of this study is to develop a laboratory analysis method that:

1. Identifies and quantifies PFAS compounds (Table 1) in aqueous media (groundwater, surface water, landfill leachate, and wastewater), solids (soil, sediment, and biosolids), and tissues using isotope dilution LC-MS/MS (method). Aqueous samples will be prepared for analysis by solid-phase extraction (SPE) and solids and tissue will be prepared using solvent extraction techniques.
2. Achieves low parts per trillion (ppt) LOQ in aqueous media and parts per billion (ppb) in solid and tissue media.
3. Is capable of being implemented at a typical mid-sized, full-service, environmental laboratory.

All data submitted by the Contract Laboratory (SGS AXYS) as part of this study will be submitted to the OW for review as part of validating the LC-MS/MS method. Laboratory packages will be submitted, along with the supporting information, to the OW for consideration as a 1600-Series method. OW will distribute the method and validation package to OLEM for consideration as an SW-846 method.

The following assumptions are made in developing the method:

1. Compounds of interest will include, at a minimum, those listed in Table 1.
2. Branched and linear isomers will be included in quantitation of analytes, when applicable.
3. Isotope dilution technique will be used for the quantitation of compounds of interest for which isotopically labeled analogs are available (at a minimum, Table 1). Where not available, the concentration of any compound of interest will be determined using the isotopically labeled analog that is most chemically similar to the compound of interest and closest in retention time (as listed in Table 1).
4. Test sample matrices (e.g. groundwater, surface water, landfill leachate, wastewater, soil, sediment, biosolids, and tissues) used for spiking will be provided to the laboratory by the EPA OW and Department of Defense (DoD). Some sample matrices will be pre-screened by EPA and DoD, where possible, and by SGS AXYS before inclusion in each study phase to confirm characteristics consistent with the goals of the project and to determine appropriate spike concentrations. For sample matrices that have not been pre-screened by EPA and DoD, the laboratory will communicate results of pre-screening to determine appropriate spiking levels.
5. Aqueous samples will be prepared for analysis using a whole sample, SPE technique. Sample matrices will be screened for Quality Assurance (QA) by the Technical Manager's EPA Office of Research and Development (ORD) laboratory to ensure concentrations do not exceed the capacity of the SPE method.
6. Solid and tissue samples will be prepared for analysis using solvent extraction techniques, including sample cleanup clean up steps.

7. All matrices of interest – wastewater effluent, landfill leachate, groundwater, surface water, biosolids, soil, sediment, and tissue – will be used to evaluate the method during the SL validation Study, using in a phased approach.
8. The techniques and instrumentation for sample preparation and analysis will be limited to those that are within the capabilities of typical mid-sized full-service environmental laboratory.

In addition to the overall objective described above, SERDP/ESTCP has three general quality objectives for this study:

1. Except where otherwise directed, all samples and data must be generated according to the analytical and QA/quality control (QC) procedures specified in this study plan and the PFAS by Isotope Dilution LC-MS/MS method. Alternatively, the data must be the result of pre-approved and documented changes to these procedures. This will allow SERDP/ESTCP and the MVT to collect data that accurately reflect the performance capabilities of the proposed methodology and to use study results to identify the need for any further revisions to the method.
2. All data submitted by the laboratory will be submitted to the OW for review as part of the SL validation package. OW will distribute the data to OLEM.
3. All data produced must be capable of verification by an independent party reviewing the analytical data package.

To meet these quality objectives, SERDP/ESTCP and the MVT will employ the following QA/QC strategies:

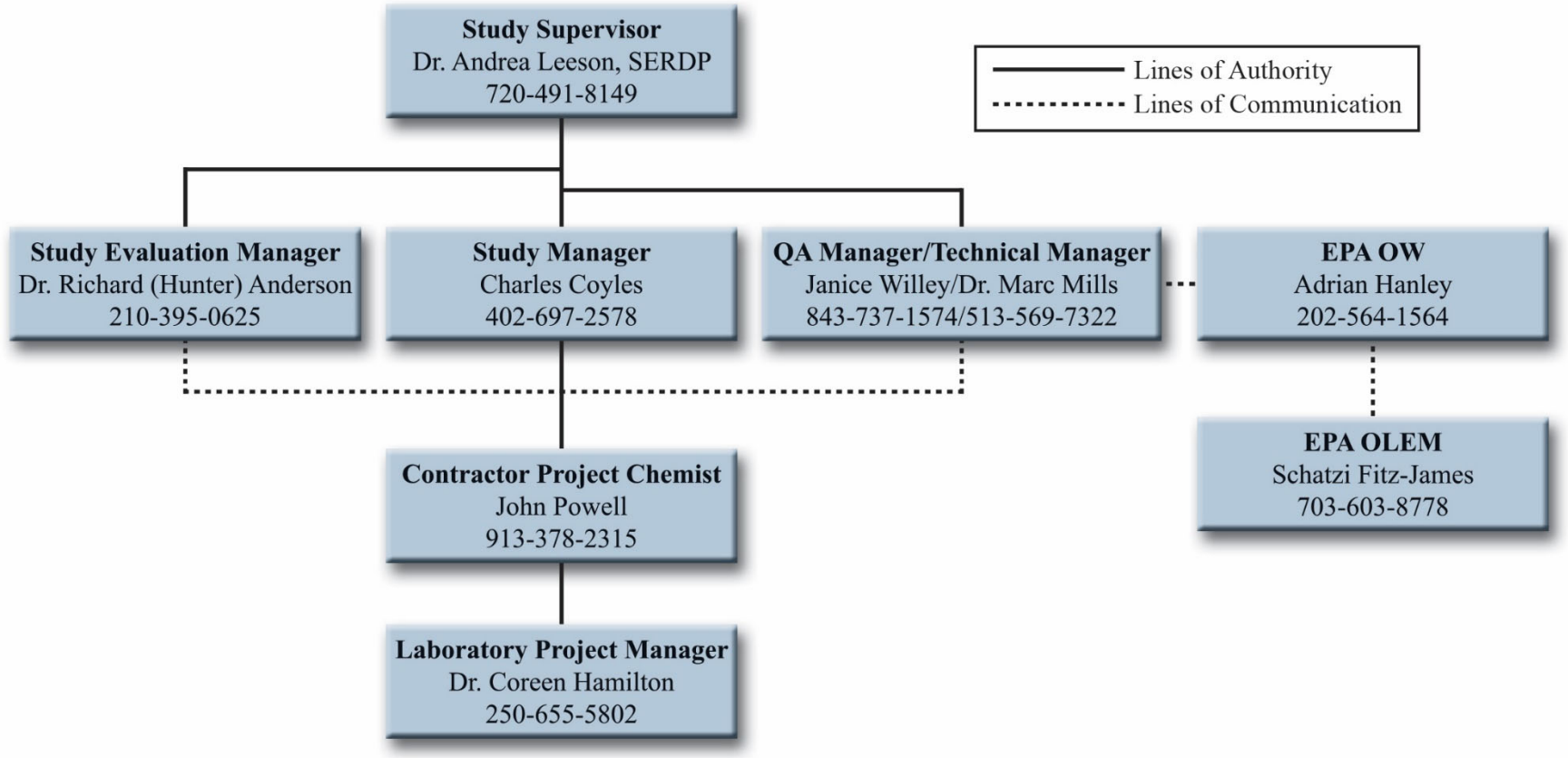
1. All Contractor activities will be performed in accordance with this study plan and with Contractor Programmatic Quality Assurance Project Plan (QAPP).
2. The Contract Laboratory is responsible for preparing and analyzing study samples (including pre-screening, in some cases, and spiking of sample matrices supplied by the MVT) and must have demonstrated experience in performing work of a similar nature. The Contract Laboratory must have a comprehensive QA program in place and implemented throughout their study operations. The Contract Laboratory will be required to follow all QC procedures defined in the PFAS by Isotope Dilution LC-MS/MS method (Attachment 1) and in Section 5 of this Study Plan. Because the purpose of the study is to develop method performance criteria, there may be instances where the QC review results do not meet the acceptance criteria specified in the LC-MS/MS method. Such instances are not failures, per se, but evidence of real-world method performance issues that are of interest in this study.
3. The developed method will be reviewed by the MVT, DoD, OW, and laboratory QA/QC officers to ensure the QC requirements meet data quality objectives (DQOs). DQOs are discussed in more detail in Section 4 and Attachment 1. The OW will submit the developed method and data from all phases of the SL validation study to OLEM for review and

comment. EPA OW and OLEM comments will be consolidated prior to sending to the QA Manager.

Cumulatively, these requirements are intended to ensure that the data produced in this study are of appropriate and documented quality.

This page was intentionally left blank.

613 STUDY MANAGEMENT



Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Coordination of Shipping of Samples	Contractor Project Chemist	John Powell	(913) 378-2315	Contractor will coordinate acquisition of study samples with EPA representatives and the Study Manager.
Work Plan Changes prior to Laboratory work	Contractor Project Manager	Mindy Vanderford	(713) 865 2223	Project Manager will gain approval for any changes in the Work Plan from USACE CEHNC and the MVT
Confirmation of Shipment of Samples	SGS AXYS	Coreen Hamilton	(250) 655-5802	Contract Lab will confirm shipment of samples to Contractor the same day received via email. Contractor will notify Study Manager of receipt via email on the same day notified.
Sample Receipt Variances	SGS AXYS	Coreen Hamilton	(250) 655-5802	SGS AXYS will notify Contractor of variances to sample receipt requirements via email on the day of receipt. Contractor will notify Study Manager of receipt via email on the same day notified.
Reporting Lab Work Plan Variances	SGS AXYS	Coreen Hamilton	(250) 655-5802	SGS AXYS will report any Work Plan variances to Contractor within one day of occurrence. Contractor will notify the QA Managers on the same day.
Acceptance of Variances and Analytical Corrective Actions	Study QA Manager/ EPATechnical Manager	Janice Willey/ Marc Mills	(843) 737-1574/ (513) 569-7322	QA Manager and Technical Manager will provide the Contractor with acceptance of variances/direction on acceptable corrective action. Contractor will notify SGS AXYS on the same day.
Reporting of Results	SGS AXYS/ Contractor	Coreen Hamilton/ John Powell	(250) 655-5802 / (913) 378-2315	SGS AXYS will provide data packages to Contractor upon completion. The Contractor will transfer the data to the QA Manager.
Reporting Data Validation Issues and Validated Data Report/Spreadsheet	Study QA Manager	Janice Willey	(843) 737-1574	QA Manager will notify Study Evaluation Manager of data validation issues and provide a validated data report/spreadsheet.
Study Evaluation Report	Study Evaluation Manager	Richard (Hunter) Anderson	(210) 395-0695	Study Evaluation Manager will provide the Study Supervisor with a draft Study Evaluation Report.
Final Study Package	Contractor Project Chemist	John Powell	(913) 378-2315	Contractor will coordinate submission of the final study package to EPA OW.

The Contract Laboratory performing the SL validation study is SGS AXYS. (Contact information: Coreen.Hamilton@sgs.com.)

SGS AXYS Analytical
2045 Mills Rd
Sidney, BC
Canada V8L 5X2

Representatives from EPA and DoD will assist the Contractor in obtaining and shipping sample matrices, representative of media of interest, to SGS AXYS. Sample matrices will be evaluated

for physical and chemical characteristics deemed important to the study by EPA or any other sample provider. Physical and chemical characteristics of interest include parameters such as total dissolved solids (TDS), total suspended solids (TSS), conductivity, and alkalinity for aqueous wastewater media and percent organic matter or lipid content for solid media and biological matrices. PFAS concentrations in sample matrices will be analyzed by some sample providers before shipment to SGS AXYS to estimate appropriate spiking levels. Additional PFAS screening by the Contract Laboratory may be required for some sample matrices. Anticipated characteristics of study sample matrices are listed in Table 4. Detailed descriptions of sample matrices are presented in Attachment 2

SGS AXYS is currently DoD Environmental Laboratory Accreditation Program (ELAP) accredited to perform analysis of PFAS under the requirements of DoD Quality Systems Manual (QSM) version 5.3, including the PFAS-specific requirements of QSM Table B-15.

SGS AXYS will submit PFAS analytical results and associated QA/QC documentation to Contractor. Contractor will review the results for completeness and submit the results to the Study QA Manager. The Study QA Manager will review and evaluate all analytical data. The Study Evaluation Manager will then analyze and draw conclusions from the results and prepare a draft study report that summarizes these results and conclusions for MVT review. As appropriate, the Study Supervisor, Study Manager, QA Managers, and Study Evaluation Manager will revise the LC-MS/MS method to optimize its performance based on the SL study findings before initiation of the ML study.

The SERDP/ESTCP MVT will submit the SL report, revised method, and all laboratory data packages to OW for review. Due to time constraints, the 90-day hold point of the Holding Time Study (see Section 4.4) may not be available at the time the majority of the data package is ready for submittal. If this is the case, any data collected beyond the period of performance of the SL study will be submitted once available.

- DoD will communicate with and coordinate the project through OW. Project progress and any potential changes in scope of cost will be communicated to the Contractor and approved by CEHNC.
- OLEM's primary role is to make sure DoD collects all data needed to support development of an SW-846 method.
- DoD will send OW the following deliverables: Study plans, draft for review and final.
    - Contractor QAPP, draft for review and final – EPA will only review the portions relevant to EPA.
    - Data packages (electronic data deliverables [EDDs] and portable document format [PDFs] lab packages. The requested EDD format is defined in Attachment 3.)
    - Data review/validation review materials and reports.
    - The SL study report, draft for review, and final report will include the statistical analysis of the data (performed by the Study Evaluation Manager). Statistical analysis of the SL Study is intended to serve as a proof of concept for the laboratory method. Results of the statistical analysis of SL data will be used to optimize the method for

ML validation. The statistical analysis of the SL study will be shared with the MVT, which includes a representative of EPA.

- A draft method in the OW Method format that does not contain any EPA logos or a method number.

713 TECHNICAL APPROACH

The study will be performed in three phases. Phase 1 (Section 4.1) involves the initial steps (initial calibration (ICAL), IDCs, MDLs, LOD and LOQ verifications, and extraction technique evaluations) in demonstrating laboratory capability with standards and clean matrices using procedures based on the LC-MS/MS method (Attachment 1). Phases 2 (Section 4.2) and 3 (Section 4.3) involve using the LC-MS/MS procedure to determine PFAS in more complex environmental matrices. Sample matrices to be included in the SL validation study is provided in Attachment 2 with a summary presented in Table 4.

4.1 PHASE 1

The focus of Phase 1 is twofold:

1. Evaluate the LC-MS/MS method for spiked, clean matrices.
2. Generate method performance criteria for QC purposes.

The initial phase of the project will involve performing ICALs, IDCs, MDL studies, and LOD and LOQ verifications. Multiple extraction methods are being tested, including one for aqueous media, one for solids, and one for tissue. The analyte list, list of Extracted Internal Standard [EIS] compounds (isotope dilution standard compounds), and Injection Internal Standards [IIS] compounds (recovery internal standard compounds) are contained in Tables 1 and 2. All internal standards will be acquired or provided by the laboratory for the SL study. Table 1 contains the minimum PFAS analytes to be included in the study. If the laboratory is able to include additional analytes, those analytes will be evaluated in the same manner as those listed in Table 1. Calibration standards will contain all analytes of interest, EIS compounds, and IIS compounds. Table 1 correlates the target analytes to the corresponding EIS compounds and Table 2 correlates the IIS compounds to the corresponding EIS compounds. The laboratory should alert the Contractor Project Chemist of any deviations from the laboratory standard operating procedure (SOP) (see Attachment 1). Any significant deviation from the methods outline in the Study Plan or Attachment 1 must be approved by the MVT and CEHNC.

Specifically, the laboratory will be required to perform the following tasks during Phase 1:

4.1.1 Initial Calibration (ICAL)

The laboratory will perform (or compile) at least three initial calibrations to demonstrate the applicable range of the procedure. Calibration QC specs and PFAS identification requirements are:

1. Standards containing both branched and linear isomers must be used when commercially available.
2. Each target analyte will be calibrated. The typical calibration range is provided in Attachment 1. However, each calibration should contain at least 5 (preferably 6) calibration levels and cover the range specified in the procedure. The lowest concentration calibration standard must be at or below the LOQ.

3. The EIS compound and IIS compound concentrations will be the same in each calibration standard and at or near the mid-level calibration concentration.
4. The maximum allowable percent relative standard deviation (%RSD) of the relative response factors for each calibration of target compound will be at or below 20 percent (%) to establish linearity. Linear or non- linear calibrations must have correlation coefficient (r^2) ≥ 0.99 for each target compound. Alternate calibration models that are already allowed for analyses that support National Pollutant Discharge Elimination System (NPDES) permits are discussed in 40 Code of Federal Regulations (CFR) 136.6(b)(4)(x).
5. Each target compound concentration must be within 70-130% of the true value for each calibration standard when back-calculated using the response-concentration relationship established by that ICAL.
6. Quantification of the target compound present in calibration standards is performed using EIS compounds. Recommended EIS compounds are listed in Table 1. EIS compound recovery is calculated using IIS compounds. Recommended IIS compounds are listed in Table 2. The laboratory should alert the Contractor Project Chemist if any deviations to the recommended labeled compounds are used for quantification.

4.1.2 Initial Demonstration of Capability (IDC)

The laboratory will perform (or compile) at least three IDCs to provide data for development of SL validation performance specifications. The IDCs will be performed using spiked reference matrices. For Phase 1, the spiked reference matrices are "clean" meaning void of target compounds at or above the MDL. The reference matrices are blank media consisting of purified or reagent water, Ottawa sand, and vegetable oil (or equivalent matrices). The laboratory will provide the clean matrices and analytical results demonstrating that the clean matrices are devoid of PFAS at concentrations above the MDL (i.e. unspiked controls). Each IDC will contain an initial precision and recovery (IPR) determination.

Initial Precision and Recovery Determination (IPR)

The IPR consists of four replicate samples of each blank medium (water, sand, oil) spiked with target and EIS compounds and carried through the entire analytical method (sample preparation and analysis). The target compounds will be spiked at the midpoint concentration of the ICAL and EIS compounds spiked at the same concentration as in the ICAL. The laboratory will calculate the percent recovery (%R) of each target compound using Equation 1:

Equation 1 Recovery

$$Recovery = \%R = \frac{C_S}{C_n} \times 100$$

where:
%R = percent recovery
C_s = Measured concentration of the spiked sample aliquot
C_n = Nominal (theoretical) concentration of the spiked aliquot

The %RSD is calculated using the results of the four replicates for each target compound using Equation 2:

Equation 2 %RSD

$$\%RSD = \frac{SD}{C_{avg}} \times 100$$

SD = Standard deviation of C_s for the four replicates
C_{avg} = Average measured concentration for the four replicates

The QA Manager will review the IPR data and work with the Study Evaluation Manager to establish performance criteria for %R and %RSD of the IPR.

MDL Studies

The laboratory will follow Revision 2 of the MDL, which was revised and codified at 40 CFR 136, Appendix B in 2017. The MDL procedure can be found at 40 CFR 136, Appendix B, or at:

https://www.epa.gov/sites/production/files/2016-12/documents/mdl-procedure_rev2_12-13-2016.pdf

Each initial MDL study will consist of seven replicate media samples spiked with target compounds and seven method blanks, prepared and analyzed over at least three days. The labeled compounds are added to each spiked sample and each method blank and carried through the entire analytical process (sample preparation and analysis). Labeled compounds will be spiked at the same levels as in the ICAL.

LOD and LOQ Verifications

LOD and LOQ verifications will be performed in each blank medium in accordance with the requirements of the DoD QSM for Environmental Laboratories, Version 5.3. The LOD and LOD are defined in the DoD QSM, Version 5.3 which can be found at:

https://www.denix.osd.mil/edqw/home/

The requirement for LOD and LOQ verification can be found in the DoD QSM, Module 4, Section 1.5.2. Each LOD and LOQ verification will consist of blank media samples spiked with target compounds and labeled compounds and carried through the entire analytical process (sample preparation and analysis). Exact spike concentrations and sample volumes will be determined by the laboratory based on the results of the MDL study or acceptable initial calibration range. Ideally, labeled compounds will be spiked around the midpoint of the calibration curve.

The MDL may be used as the detection limit (DL). The DL, as defined by the DoD QSM, is the smallest analyte concentration that can be demonstrated to be different from zero or a blank

concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

The LOD is the lowest concentration of a target analyte that must be present in a sample in order to be detected at the detection limit (DL) with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. An LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence. The LOD is verified by preparing a clean matrix at 2 to 4 times the determined DL in the same manner as field samples. If analysis of this QC sample meets all qualitative requirements of the method for analyte identification (e.g., signal to noise ratio, confirmation ion presence) then the LOD shall be established as the concentration of the spike.

The LOQ is the smallest concentration that produces a quantitative result with known and recorded precision and bias. The LOQ is set at or above the concentration of the lowest ICAL standard and within the calibration range. An LOQ verification meets all requirements of a lower limit of quantitation (LLOQ), as defined by EPA SW-846. The LOQ will be spiked between 1-2 times the corresponding lowest calibration standard and will go through the same sequence of preparation and analytical steps as used when analyzing a normal sample. It is essentially a low-level laboratory control sample (LCS). The DoD LOQ and the EPA's LLOQ are similar. The only significant difference is that the DoD uses 70-130% recovery limits and the EPA uses 50-150% recovery limits. The DoD recovery limits of 70-130% shall be used for this study.

4.1.3 Water Sample Preparation

The extraction procedure for aqueous samples is anticipated to be by SPE. Implementation of the SPE procedure may depend on the percentage of suspended solids (SS) in the sample. Aqueous samples are homogenized by the analyst using techniques indicated in Attachment 1 prior to extraction. If quantitation of the amount of particulate is required, a quantitation of SS must be carried out prior to analysis as described in the LC-MS/MS method.

Note: SS quantitation is not required for Phase 1 samples. Samples prepared in Phase 1 are blank media, not real-world samples.

Requirements for Procedures to Separate Solids and Aqueous Phases of the Sample

The volume of an aqueous sample required for analysis may depend on its SS content, due to potential losses during the cleanup procedure. SS will be quantified for all aqueous samples prior to cleanup and analysis, except for those included in Phase 1. Each sample will be categorized as either ≤ 1% SS or >1% SS with protocols for each described below.

The typical sample size is 500 milliliters (mL). Aqueous samples with SS > 1% must be separated from the solids phase by SPE prior to analysis. During aqueous sample preparation, each SPE cartridge can handle approximately 100 milligrams of solids before clogging. A high solids sample can be split among more than one SPE cartridge and the eluents combined into a single extract for analysis. The need for a second SPE cartridge can be determined once the processing of the sample has begun as %SS may not be the sole indicator of potential clogging. Samples with ≤ 1% SS are

not centrifuged (or filtered) prior to extraction. Samples with > 1% SS are centrifuged prior to extraction and the phases are extracted and analyzed separately.

Note: The full suite of QC samples typically associated with EPA analytical methods, including method blanks and LCS/on-going precision and recovery (OPR) samples will be prepared in conjunction with samples prepared for this study.

Extraction of Samples with ≤ 1% SS

Note: This extraction procedure will be used for all Phase 1 water samples and Phase 2 and 3 samples with low SS.

After homogenization, a water sample is spiked with target and EIS compounds and extracted using the procedure described in Attachment 1 for water samples with ≤ 1% SS.

Extraction of Samples with > 1% SS

Note: This extraction procedure is not needed for the Phase 1 water samples but may be needed for the Phase 2 samples.

After homogenization, a water sample is spiked with target compounds and/or labeled compounds and carried through the entire sample preparation, cleanup, and analytical process described in the Attachment 1 method for water samples with >1% SS. A separate extraction of the solids removed from the wastewater samples via filtration or centrifugation will not be conducted during Phase 2 but may be evaluated during Phase 3.

4.1.4 Solid Sample Preparation

Solid samples are to be extracted following the procedure described in Attachment 1. In summary, after homogenization, a sample is spiked with target and EIS compounds and carried through the entire sample preparation, cleanup, and analytical process described in the LC-MS/MS method for solid samples.

Note: Ottawa sand is the proxy medium for solid sample matrices for Phase 1.

4.1.5 Tissue Sample Preparation

Tissue samples are to be extracted following the procedure described in Attachment 1. In summary, a homogenized tissue sample is spiked with target and EIS compounds and carried through the entire sample preparation, cleanup, and analytical process described in the LC-MS/MS method for tissue samples.

Note: Vegetable oil is the proxy medium for tissue sample matrices for Phase 1.

4.1.6 Sample Analysis

Once extraction and cleanup are complete, all samples will be analyzed using the Attachment 1 LC-MS/MS method. SGS AXYS also will be required to analyze all the QC samples specified in the LC-MS/MS method with each batch of study samples, as described in Section 5 of this study plan.

The SERDP/ESTCP MVT will use the data from Phase 1 to develop formal performance evaluation data for the LC-MS/MS method. The data may be used to demonstrate overall precision and accuracy of the procedure for the measurement of target analytes, and also may be used to generate QC acceptance criteria for some aspects of the method.

4.2 PHASE 2

Following a brief review of the Phase 1 results by the MVT to ensure that study goals for this phase are met, the MVT will recommend that SGS AXYS proceed with the Phase 2 of the study. The focus of Phase 2 is to evaluate the sample preparation and LC-MS/MS method in various wastewater matrices, landfill leachate, groundwater, and surface water matrices. Matrices will be provided by the EPA or DoD, via existing client relationships, and/or supplemented by materials provided by other sources. A summary of sample matrices for Phase 2 is provided in Table 4 with a detailed list in Attachment 2. Based on preliminary results, the expected spiking concentrations are 5, 10, and 15 times the LOQ for aqueous samples, but these may be updated after determination of background levels of PFAS in sample matrices

The laboratory will analyze different wastewater matrices, covering matrix categories analogous to those specified for alternate test procedures. Wastewater matrices consist of effluents from publicly owned treatment works and industrial sources. If necessary, a substitute wastewater as specified in ASTM D 5905 - 98 (Reapproved 2013), Standard Specification for Substitute Wastewater (Ref. 8.4) may be provided. At least one of the wastewater sample matrix types should have at least one of the following characteristics such that each criterion below is represented by at least one wastewater:

- TSS greater than 40 milligrams per liter (mg/L)
- TDS greater than 100 mg/L
- Oil and grease greater than 20 mg/L
- Sodium chloride greater than 120 mg/L
- Calcium carbonate greater than 140 mg/L

Three replicate samples of each wastewater matrix will be evaluated at three spiked concentrations and as one unspiked control sample(s). In summary, wastewater samples will be spiked with target compounds and/or labeled compounds and carried through the entire sample preparation, cleanup, and analytical process described in the LC-MS/MS method for water samples and Section 4.1.3 of this study plan.

Spiking levels and replicates for groundwater, surface water, and landfill leachate are indicated in Attachment 2.

As stated for Phase 1, unspiked matrices (control samples) will be evaluated first for each sample matrix due to the ubiquitous nature of PFAS. Some sample matrices will have been preliminarily analyzed for background PFAS concentrations before shipment; however, unspiked sample matrices should be analyzed by the LC-MS/MS method before spiking other replicates in order to confirm the background concentrations of PFAS. High background PFAS concentrations may affect the spike amounts of target compounds to be added to the spiked samples.

The spike amounts will be determined or confirmed by the QA Manager after consulting with the laboratory on the results of the background level analyses. Analytical results of the background analysis will be included in the laboratory data package.

A total of seven (7) wastewater samples will be prepared in quadruplicate. Each sample will be analyzed unspiked and at three spiked concentrations (spike level dependent on native concentrations of target compounds) for a total of 84 wastewater analyses. Four (4) replicates of three (3) samples each of landfill leachate, groundwater, and surface water will be prepared. Each replicate will be prepared unspiked and spiked at three concentrations. The total number of groundwater, landfill leachate, and surface water samples prepared and analyzed will be 36 for each medium.

4.3 PHASE 3

The third phase of this study is designed to evaluate the sample preparation and LC-MS/MS method in biosolids, sediment, soil, and fish tissue samples. EPA's Clean Water Act Methods Program only approves methods for wastewater, not biosolids, soil, and fish tissues. However, many 40 CFR Part 136 methods contain some sample preparation information on these matrices. Analysis of solid matrices is often needed for wastewater samples with high silt or solid content. As in Phase 2, matrices will be provided to the laboratory, via existing client relationships, and/or supplemented by materials provided by the EPA and DoD.

Sample matrices to be evaluated in Phase 3 are listed in Table 4 and Attachment 2, and include sewage sludge samples, the solid fraction of a wastewater sample with > 1% SS, and fish tissue samples. The same approach for solid samples will be taken as for water samples in Phase 1 and Phase 2. Solid samples will be spiked with target and EIS compounds and carried through the entire sample preparation, cleanup, and analytical process described in the LC-MS/MS method for solid samples and Section 4.1.4 of this study plan. Tissue samples will be spiked with target and EIS compounds and carried through the entire sample preparation, cleanup, and analytical process described in the sample preparation and LC-MS/MS method for tissue samples and Section 4.1.5.

As stated for Phases 1 and 2, three replicate unspiked control sample matrices will be extracted and analyzed due to the ubiquitous nature of PFAS. The laboratory will communicate background PFAS concentrations in the sample matrices to the study QA Manager. The spiking levels will be determined by the Study Technical Manager and QA Manager in consultation with the laboratory after review of background data. Based on preliminary results spiking levels are expected to be approximately 5, 10, and 15 times the LOQ for aqueous sample matrices and 4, 6, and 10 times the LOQ for solid samples. Data from the unspiked control sample matrices can be used in the Holding Time Study to establish background

Three replicates of seven (7) soil samples and three (3) samples each for sediment, tissue, and biosolids will be prepared, and analyzed for unspiked and spiked samples at three concentrations. A total of 36 samples will be prepared and analyzed for sediment, tissue, and biosolids each and 84 samples will be prepared and analyzed for soils.

4.4 HOLDING TIME STUDY

A holding time study will be performed by SGS AXYS. The goal of the holding time study is to assess how long environmental samples and extracts of samples can be held at various temperatures before PFAS degrade, become unextractable, or otherwise change concentrations so that the samples are no longer representative of the media from which they are derived.

The holding time study can be run at any time, given the assurance that laboratory facilities are available on the precise days and times outlined in the description below. The study QA Manager needs to have assurance that their IPR, MDL, and matrix samples are spiked and then analyzed within the specified holding time.

Holding time studies previously completed by SGS AXYS for aqueous media will be submitted for review. The SGS AXYS historical holding time data package will include associated QA/QC information to confirm that the study meets required DQOs. It is assumed here that the previously conducted SGS AXYS holding time study data for reagent-grade aqueous samples have sufficient data to address method development requirements under the Clean Water Act. So, the current holding time study will not include a reagent-grade aqueous medium.

The holding time study will include one groundwater, one soil, one sediment, and one biosolid sample matrix as well as extracts of each of these matrices. A summary of matrices and holding times for the study are listed in Table 5.

The matrices will be spiked at the mid-level concentrations from Phases 2 and 3 (roughly 10 times the LOQ for aqueous and 6 times the LOQ for solid matrices) and analyzed with 3 replicate samples for each time increment. PFAS precursors, typically used by SGS AXYS (perfluorooctanesulfonamidoethanols [FOSEs], telomer alcohols) shall be included in standards added to samples for evaluation of holding times. The samples are to be preserved and put into the appropriate sample containers as though they are field samples.

Groundwater and Groundwater Extract Analysis:

- Groundwater matrix C (see Attachment 2) will be provided at sufficient volume to perform the holding time study.
- Ten sets of three replicate sample matrices (30) are to be spiked on day 0 with 5 aliquots maintained at 0-6 degrees Celsius (°C) (~ 4°C, refrigerated) and 5 aliquots frozen (~−20 °C). One set of refrigerated and one set of frozen samples (3 replicates each) are to be extracted and analyzed on days 0, 7, 14, 28, and 90 after spiking.

- Day 0 spiked groundwater will be extracted in sufficient volume to perform a holding time study on the extract. Three replicates of day 0 groundwater extracts stored at 0-6°C will be reanalyzed on days 7, 14, 28, and 90 (12 samples).
- Total aqueous sample count equals 30 samples and 12 extract analyses for a total of 42 samples.

Solids samples:

- Three solid sample matrices consisting of one soil, one sediment and one biosolid (see Attachment 2 matrix will be evaluated. The solid matrices should be hydrated via aqueous spike solutions. Then, the samples should be air dried (or wet/dried repeatedly) prior to the day 0 sample. This will account for spiking artifacts and will be far more representative of the field situation. Some water content shall be added to air-dried soils prior to spiking (suggest 10% or 20% by mass, which would translate to 4.5 grams (g) dry soil + 0.5 mL water, or 4 g dry soil + 1 mL water).
- Eight aliquots of 3 replicate samples (24) for each matrix type (72 total) are to be spiked on day 0 with 4 sets maintained at 0-6 °C and 4 sets frozen (~-20°C). One set of refrigerated and one set of frozen for each matrix type (three replicates each) are to be extracted and analyzed on days 0, 14, 28, and 90.
- Additionally, three replicates of day 0 solid extracts will be stored at 0-6°C and reanalyzed on days 14, 28, and 90. Three (3) sample types times 3 replicates times 3-time increments (day 0 will be done with the previous batch) equals 27 extract re-analyses
- Total solid sample count equals 72 samples and 27 additional extract analyses for a total of 99 samples.

Total holding time study sample count equals 30 aqueous samples, 72 solid samples, and 39 extract reanalyses for a total 141 samples.

This page was intentionally left blank.

813 QUALITY CONTROL PROCEDURES

The preparation and analytical procedures include traditional QC procedures found in EPA methods for the analysis of organic contaminants. All of the associated QC checks are summarized in Table 3 of this study plan and the SOP (Attachment 1). Given the nature and purpose of this study, some of those QC checks will not be performed separately from the analyses of the many spiked samples. Rather, the data from this study will be used to evaluate the adequacy of the method's current acceptance criteria, and as needed, to develop revised criteria that reflect the performance of this method in real-world matrices.

It is the laboratory's responsibility to maintain their instrumentation and to ensure that all study samples are analyzed on a properly calibrated instrument; therefore, if the instrument calibrations or other instrument QC (i.e. mass spectrometer tune, mass calibration check, or qualitative identification criteria) are outside the normal criteria (see Table 3), the laboratory will take standard corrective actions (e.g., cleaning the instrument, replacing column or other instrument parts) to attempt to resolve the problem before any study samples are analyzed. The laboratory is also responsible for inspecting all study samples and standards to ensure they meet all study requirements. If standard corrective actions do not resolve the problem or if study schedules will be impacted due to necessary repairs or replacement of study samples or standards, the laboratory will notify the Contractor Project Chemist to indicate the impact on study schedules, the laboratory's plans to resolve the problem(s), and if any study samples will need to be reanalyzed.

The laboratory will report the results from all procedure-specified QC operations, either in electronic format, or if necessary, in hard copy. The Contractor will compile the QC results in a database format specific to this project (See Section 6).

This page was intentionally left blank.

913 DATA REPORTING

SGS AXYS will be required to (1) provide all required information electronically, including raw data, (2) report summary-level electronic data and sufficient information for Stage 4 data validation to be performed, (3) an electronic data deliverable in the format provided in Attachment 4, and (4) maintain their raw data for a period of five (5) years and provide it upon request (at additional cost negotiated as necessary). Raw data will include all calibration data, chromatograms, quantitation reports (including quant and qualifier peaks, transition ion ratios, peak areas or heights), analytical standards preparation records (including manufacturer's Certificate of Analysis), bench sheets, and laboratory notebooks showing weights, volumes, manual calculations, and other data that will allow verification of the calculations performed and will allow the final results reported to be traced back to the raw data.

The laboratory is instructed to adhere to the following rules when reporting data:

- All reports and documentation, including instrument print-outs and other raw data, must be sequentially paginated, clearly labeled with the laboratory name, and labeled to provide sufficient identification for method blanks, calibration, interference checks, etc., necessary to link the raw data with associated summary reports.
- Results from all analyses must be reported, including calibration data and any dilutions or reanalyses performed. The laboratory also must include an explanation of any dilutions or reanalyses performed and identify which of the analyses the lab considers to be most appropriate for use.
- Concentrations of all qualitatively identifiable peaks must be reported to three significant figures in the appropriate reporting units of ppt or nanograms per liter for aqueous samples and ppb or micrograms per kilogram [µg/kg] for solid and tissue samples.
- Data qualifiers are provided in Attachment 3 must be used where appropriate.

In addition, the lab will be required to submit a written "narrative report" with each data package. The narrative report will contain detailed descriptions of any difficulties encountered in the generation of the analytical results and QC data and any attempts to resolve the difficulties. It also will contain a detailed description of any modifications to the sample preparation and analysis procedure and the date that these modifications were pre-approved by the Contractor Project Chemist.

Due to time constraints, the 90-day holding time sample points may not be available at the time the rest of the data package is ready for submittal. If this is the case, these data will be submitted as a follow on once available. The laboratory will provide reports for other phases of this study while the holding time study is in progress. This will include the data packages and electronic data deliverables.

6.1 DATA MANAGEMENT AND REPORTING

Contractor will store all submitted data (hard copy and electronic) in master files established for this study on a secure network. These master files will include the following documents and records:

- The study plan (including all draft versions, comments, and revisions)
- Documentation of the procedures used to assess the competency of laboratories participating in this study
- Documents and records associated with the solicitation and award of participant laboratories, including the statements of work (SOWs) or equivalent that describe participant laboratory requirements
- Documents and records associated with the procurement of standards and study samples, including SOWs or equivalent that describe the process used to collect and produce study samples
- The name, address, phone number and primary contact at the standards vendor and each participating laboratory
- Copies of all written correspondence (excluding non-critical emails) with laboratory staff, sampling personnel, and SERDP/ESTCP PFAS MVT staff regarding the study
- A log (or other record) that documents verbal communication with laboratory staff, sample coordinators, sampling personnel, and SERDP/ESTCP PFAS MVT staff regarding study status or problems
- Records concerning sample shipment and receipt
- All analytical data resulting from this study
- All laboratory comments on the method resulting from this study
- Records of all data review assessments and statistical analyses submitted to SERDP/ESTCP PFAS MVT
- All draft and final reports submitted to SERDP/ESTCP PFAS MVT pertaining to this study.

Contractor will develop a schedule for routine communications during the course of the study, based on the specific activities underway at the time. For example, Contractor will communicate with the Study QA Manager more frequently (e.g., daily) during those periods when samples are being shipped to the laboratories, versus less frequent communications during the periods when sample analyses are taking place. The Contractor will provide an EDD as part of the submittal, to facilitate statistical analysis of the data by the Study Evaluation Manager. The Contractor will upload the EDD and raw data packages from the laboratories to AMRDEC and notify the ESTCP Method Validation Team as soon as the materials have been uploaded to AMRDEC.

:13 EVALUATION OF METHOD PERFORMANCE

As noted earlier, DoD and the Contractor will perform brief examinations of the results of each phase of the study before authorizing the SGS AXYS to proceed with the next study phase. Those examinations will focus on completeness of the data submitted (e.g., were all required samples analyzed and results reported?) and on cursory assessments of the QC results (e.g., is there evidence of gross contamination in the blanks and do the QC results indicate that the laboratory is capable of analyzing samples at the levels of interest?). However, individual QC failures will not necessarily negate the results of a particular analysis. Rather, they will be used as indicators of performance issues that may be related to the concentrations of specific interferences in the sample matrices being tested or in the recovery procedure for extracting the sample matrices.

DoD's overall goal is to develop method performance data for the LC-MS/MS method. The results of the analyses in the three phases and holding time study will be evaluated using common statistical procedures including, but not limited to:

- Basic summary statistics of %R and %RSD metrics discussed in Section 4.1.2 above for each medium evaluated,
- Analysis of variance, to determine mean differences occurring among different "treatments" (e.g., nominal spike concentrations) and/or specific analytes and/or different media as well as any potential interactions therein.

DoD and the Contractor may use the results from the replicate samples to develop preliminary QC criteria for IPR tests, OPR tests, matrix spike/matrix spike duplicate recovery limits, relative percent difference limits, etc. The method performance criteria generated from this study will be used as a rough guideline for the ML study performance criteria.

Finally, DoD and the Contractor will develop tables of method performance data, including precision and accuracy, as a function of analyte concentration that will provide an indication of expected performance of the method under typical conditions. Such tables can be included in the revised method as further evidence of its overall capabilities or limitations.

Because this is a method development effort, there are no *a priori* QC acceptance criteria, and data from the study will not be excluded from consideration simply because they appear to fail some preconceived performance expectations. All study results will be subjected to statistical evaluations, and suspected outliers will be examined in detail by the Contractor and the laboratory before they are excluded from use in developing method performance summaries. EPA will also be given the opportunity to review the statistical evaluations and make edits before the study report is finalized.

This page was intentionally left blank.

;13 REFERENCES

8.1 International Organization for Standardization (ISO). 2009. Technical Committee (TC) 147, Water quality - Determination of perfluorooctansulfonate (PFOS) and perfluorooctanoate (PFOA) - Method for unfiltered samples using solid-phase extraction and liquid chromatography/mass spectrometry, ISO 25101:2009

8.2 AXYS Analytical Services Ltd. 2011. AXYS Method MLA-110: Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples, Solids, Tissues and Solvent Extracts by LC-MS/MS. Rev. 02, Ver. 2, April 2019

8.3 Federal Register Volume 82, Number 165. August 28, 2017). pp. 40836-40941 Final Rule, 40 CFR Part 136, Clean Water Act Methods Update Rule for the Analysis of Effluent

8.4 ASTM. 2013. Method D5095-9. Standard Practice for the Preparation of Substitute Wastewater, ASTM International, West Conshohocken, PA. http://www.astm.org

8.5 ASTM 1998. Method D2777-98, Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water, ASTM International, West Conshohocken, PA. http://www.astm.org

This page was intentionally left blank.

TABLES

This page was intentionally left blank.

Table 1
Target Analytes, CASRNs, and Quantification References
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

Method Compounds			Labeled Compounds
Compound Name	**CASRN**	**Abbreviation**	
Perfluorobutanoic acid	375-22-4	PFBA	Perfluoro-n-[13C4]butanoic acid
Perfluoropentanoic acid	2706-90-3	PFPeA	Perfluoro-n-[13C5]pentanoic acid
Perfluorohexanoic acid	307-24-4	PFHxA	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid
Perfluoroheptanoic acid	375-85-9	PFHpA	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid
Perfluorooctanoic acid	335-67-1	PFOA	Perfluoro-n-[13C8]octanoic acid
Perfluorononanoic acid	375-95-1	PFNA	Perfluoro-n-[13C9]nonanoic acid
Perfluorodecanoic acid	335-76-2	PFDA	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid
Perfluoroundecanoic acid	2058-94-8	PFUnA	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid
Perfluorododecanoic acid	307-55-1	PFDoA	Perfluoro-n-[1,2-13C2]dodecanoic acid
Perfluorotridecanoic acid	72629-94-8	PFTrDA	Perfluoro-n-[1,2-13C2]tetradecanoic acid
Perfluorotetradecanoic acid	376-06-7	PFTeA	Perfluoro-n-[1,2-13C2]tetradecanoic acid
Perfluorobutanesulfonic acid	375-73-5	PFBS	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid
Perfluoropentanesulfonic acid	2706-91-4	PFPeS	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid
Perfluorohexanesulfonic acid	355-46-4	PFHxS	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid
Perfluoroheptanesulfonic acid	375-92-8	PFHpS	Perfluoro-1-[13C8]octanesulfonic acid
Perfluorooctanesulfonic acid	1763-23-1	PFOS	Perfluoro-1-[13C8]octanesulfonic acid
Perfluorononanesulfonic acid	68259-12-1	PFNS	Perfluoro-1-[13C8]octanesulfonic acid
Perfluorodecanesulfonic acid	335-77-3	PFDS	Perfluoro-1-[13C8]octanesulfonic acid
Perfluorododecanesulfonic acid	79780-39-5	PFDoS	Perfluoro-n-[1,2-13C2]decanoic acid
4:2 fluorotelomersulfonic acid	757124-72-4	4:2FTS	1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid
6:2 fluorotelomersulfonic acid	27619-97-2	6:2FTS	1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid
8:2 fluorotelomersulfonic acid	39108-34-4	8:2FTS	1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid
Perfluorooctanesulfonamide	754-91-6	PFOSA	Perfluoro-1-[13C8]octanesulfonamide

Table 1 (Continued)
Target Analytes, CASRNs, and Quantification References
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

Method Compounds			Labeled Compounds
Compound Name	**CASRN**	**Abbreviation**	
N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid
N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid
N-ethyl perfluorooctanesulfonamide	4151.50-2	NEtFOSA	N-ethyl-d5-perfluoro-1-octanesulfonamide
N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA	N-methyl-d3-perfluoro-1-octanesulfonamide
N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtFOSE	N-methyl-d7-perfluorooctanesulfonamidoethanol
N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	MeFOSE	N-ethyl-d9-perfluorooctane sulfonamidoethanol
Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid
4,8-dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid

Notes:
CASRN = Chemical Abstracts Service Registry Number

Table 2
Isotope Dilution Standards and their associated Recovery Internal Standards
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

Quantified using Isotope Dilution Standards	
$^{13}C_4$-PFBA	$^{13}C_3$-PFBA
$^{13}C_5$-PFPeA	$^{13}C_2$-PFHxA
$^{13}C_5$-PFHxA	$^{13}C_2$-PFHxA
$^{13}C_4$-PFHpA	$^{13}C_4$-PFOA
$^{13}C_8$-PFOA	$^{13}C_4$-PFOA
$^{13}C_9$-PFNA	$^{13}C_5$-PFNA
$^{13}C_6$-PFDA	$^{13}C_2$-PFDA
$^{13}C_7$-PFUnA	$^{13}C_2$-PFDA
$^{13}C_2$-PFDoA	$^{13}C_2$-PFDA
$^{13}C_2$-PFTeDA	$^{13}C_2$-PFDA
$^{13}C_3$-PFBS	$^{18}O_2$-PFHxS
$^{13}C_3$-PFHxS	$^{18}O_2$-PFHxS
$^{13}C_8$-PFOS	$^{13}C_4$-PFOS
$^{13}C_2$-4:2 FTS	$^{18}O_2$-PFHxS
$^{13}C_2$-6:2 FTS	$^{18}O_2$-PFHxS
$^{13}C_2$-8:2 FTS	$^{18}O_2$-PFHxS
$^{13}C_8$-PFOSA	$^{13}C_4$-PFOS
D_3-N-MeFOSA	$^{13}C_4$-PFOS
D3-NMeFOSAA	$^{13}C_4$-PFOS
D_5-N-EtFOSA	$^{13}C_4$-PFOS
D5-NEtFOSAA	$^{13}C_4$-PFOS
D_7-N-MeFOSE	$^{13}C_4$-PFOS
D_9-N-EtFOSE	$^{13}C_4$-PFOS
$^{13}C_3$-HFPO-DA	$^{13}C_2$-PFHxA
Recovery Internal Standards	
$^{13}C_3$-PFBA	External
$^{13}C_2$-PFHxA	External
$^{13}C_4$-PFOA	External
$^{13}C_5$-PFNA	External
$^{13}C_2$-PFDA	External
$^{18}O_2$-PFHxS	External
$^{13}C_4$-PFOS	External

Table 3
Routine QC Checks
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

QC Check	Frequency	Acceptance Criterion	Study Requirements
Initial demonstration of capability	A minimum of three during Phase 1	Initial precision and recovery, percent recovery (%R), and percent relative standard deviation (%RSD) will be established after review of Phase 1 data	Not required for this study. Study data will be used to establish criteria.
Initial calibration (ICAL), 5-point minimum	A minimum of three during Phase 1	%RSD of the relative response factors should be ≤20% or should have coefficient of determination (r^2) >0.99 for each analyte. Target compounds should be within 70-130% of their true value for each calibration standard. Reports all results as generated.	ICAL data will be provided by lab and compared to typical calibration criteria.
Instrument Sensitivity Check	Prior to analysis and at least once every 12 hours.	Analyte concentrations should be at limit of quantitation (LOQ); concentrations must be within ±30% of their true values. Reports all results as generated.	Perform as specified in procedure.
Calibration verification	Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence.	Concentration of target analytes should range from the LOQ to the mid-level calibration concentration. Target analyte concentrations should be within ±30% of their true value. Reports all results as generated.	Perform as specified in procedure. U.S. Department of Defense (DoD)/Contractor will evaluate internal standard responses, since "recovery" cannot be determined directly.
Initial Calibration Verification	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Analyte concentrations should be within ±30% of their true value. Reports all results as generated.	Perform as specified in procedure.
Method blank	One per batch of 20 field samples or fewer	No analytes should be detected >½ LOQ or >1/10th the amount measured in field samples in the batch, whichever is greater. Reports all results as generated.	Perform as specified in procedure.
Laboratory control sample	One per preparation batch of 20 field samples or fewer	Target analytes should be spiked at a concentration ≥ LOQ and ≤ mid-level calibration standard. To be determined based on study data. Reports all results as generated.	Perform as specified in procedure to demonstrate performance at the method-specified laboratory control sample concentration. Study data will be used to test existing criterion.
EPA lower limit of quantitation (LLOQ)/DoD LOQ Verification	On per batch of 20 field sample or fewer	Target analytes should be spiked at a concentration ≥ LLOQ and ≤2x LLOQ.	Perform as specified in procedure.
Matrix spike/matrix spike duplicate	One per preparation batch of 20 field samples or fewer	Target analytes should be spiked at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. To be determined based on study data. Reports all results as generated.	Not required for this study because of the many spiked samples already being analyzed. Study data will be used to test existing criterion.
Analysis Duplicate	One per 20 field samples or fewer	To be determined based on study data. Reports all results as generated.	Perform as specified in procedure. Study data will be used to test existing criterion.

Table 3 (Continued)
Routine QC Checks
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

QC Check	Frequency	Acceptance Criterion	Study Requirements
Mass calibration check	Instrument must have a valid mass calibration prior to any sample analysis. Mass calibration is verified after each mass calibration, prior to ICAL.	A minimum of 10 spectra scans are acquired across each chromatographic peak. Mass calibration is verified after each mass calibration, prior to initial ICAL. Mass calibration must be verified to be ±0.5 atomic mass unit (amu) of true value by following the instrument manufacturer's instructions for performing a mass calibration verification and using the instrument manufacturer's recommended standards as long as these standards cover the mass range of the per- and polyfluoroalkyl substances (PFAS) ions of interest. calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ±0.5 amu of the true value.	Perform as specified in procedure.
Qualitative Identification Criteria	All peaks	A minimum of 10 spectra scans are acquired across each chromatographic peak. The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFBA, PFPeA, N-EtFOSE, and N-MeFOSE, PFOSA). Ion ratios must be used and must not exceed 50-150%. Signal to Noise Ratio must be ≥ 3 for all ions used for quantification and confirmation. Retention time of each target compound and labeled compound must fall within 0.4 minutes of the predicted retention times from the daily calibration verification or the midpoint standard of the ICAL. Analytes must elute within 0.1 minutes of the associated labeled compound. This criterion applies only to analyte and labeled analog pairs.	Perform as specified in procedure.

Notes:
QC = quality control

Table 4
Summary Sample Matrices for Inclusion in the Study
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

Project Phase	Medium	Characteristics	Number of Sample Matrices	Sample Matrix[1]
Phase 1	Purified or reagent water	Aqueous medium, no detectable PFAS contamination	1	Provided by laboratory
	Ottawa sand	Solid medium, no detectable PFAS contamination	1	
	Vegetable oil	Lipid medium, no detectable PFAS contamination	1	
Phase 2	Groundwater	No special characteristics, collected from field sites with PFAS in groundwater	3	Sample matrices A, B, and C
	Surface water	River, reservoir, and marine (salt) water, not further characterized	3	Sample matrices D, E, and F
	Wastewater	One or more of the following: TSS > 40 mg/L, TDS > 100 mg/L, Oil and grease > 20 mg/L, CaCO3 > 140 mg/L	7	Sample matrices J, K, L, M, N, O, and P
	Landfill Leachate	Municipal solid waste and construction debris landfill leachate	3	Sample matrices G, H, and I
Phase 3	Sediment	Marine, freshwater low TOC, freshwater high TOC	3	Sample matrices Q, R and S
	Fish/shellfish tissue	Low lipid and high lipid samples	3	Sample matrices T, U, and V
	Biosolids	Municipal biosolids, SRM sludge	3	Sample matrices W, X, and Y
	Soil	NAPT soils, low and high-level contamination	7	Sample matrices Z, AA, BB, CC, DD, EE, and FF

***Notes**:*

1. *Sample matrix letter designation refers to matrices listed in Attachment 2.*
2. *Detailed sample matrix descriptions provided in Attachment 2.*
3. *TSS = total suspended solids; TDS = total dissolved solids; CaCO3 = calcium carbonate; TOC = total organic carbon; SRM = standard reference materials; NAPT= North American Proficiency Testing Program*

Table 5
Holding Time Study Summary
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

Matrix	Incubation Temperatures	Incubation Intervals (days after spiking)	Replicates	Total Samples
Groundwater (Matrix C)	0-6 °C and -20°C	0, 7, 14, 28, and 90	3 for each condition	30
Groundwater extract	0-6 °C	7, 14, 28, and 90	3 for each time point	12
Soil (Matrix Z)	0-6 °C and -20°C	0, 14, 28, and 90	3 for each condition	24
Soil Extract	0-6 °C	14, 28, and 90	3 for each time point	9
Sediment (Matrix S)	0-6 °C and -20°C	0, 14, 28, and 90	3 for each condition	24
Sediment Extract	0-6 °C	14, 28, and 90	3 for each time point	9
Biosolid (Matrix W)	0-6 °C and -20°C	0, 14, 28, and 90	3 for each condition	24
Biosolid Extract	0-6 °C	14, 28, and 90	3 for each time point	9
Total Samples				**141**

Notes:

1. *Matrix letter refers to sample matrix designated in Attachment 2.*
2. *Unspiked controls for background PFAS will be analyzed during Phases 2 and 3.*

This page was intentionally left blank.

ATTACHMENT 1

LC-MS/MS METHOD

Note: The document attached here is the Standard Operating Procedure PFAS Analysis by Isotope Dilution LC-MS/MS, developed by SGS AXYS for the Single Lab Validation Study.

Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples, Solids, Tissues, and Solvent Extracts by LC-MS/MS

SCOPE

This method describes the analysis of per- and polyfluoroalkyl substances (PFAS) in aqueous, solid, tissue, and biosolid samples, determined as the total of linear and branched isomers. Method analytes and their CAS numbers can be found in Table 1. After spiking with isotopically labeled surrogate standards and cleanup on SPE cartridges aqueous samples are analyzed by liquid chromatography/mass spectrometry (LC-MS/MS). Solid samples are spiked with isotopically labelled surrogate standards, extracted in basic methanol, and cleaned up by carbon and SPE cartridges before analysis by LC-MS/MS. Tissue samples are spiked with isotopically labelled surrogate standards, extracted in acetonitrile followed by basic methanol, and cleaned up by SPE cartridges before analysis by LC-MS/MS. Final sample concentrations are determined by isotope dilution/internal standard quantification. All method analytes are quantified and reported in their neutral acid form.

Table 1. List of method analytes and CAS numbers

Abbreviation	***Name – Acid/Neutral Form***	***CAS#***
PFBA	Perfluorobutanoic acid	375-22-4
PFPeA	Perfluoropentanoic acid	2706-90-3
PFHxA	Perfluorohexanoic acid	307-24-4
PFHpA	Perfluoroheptanoic acid	375-85-9
PFOA	Perfluorooctanoic acid	335-67-1
PFNA	Perfluorononanoic acid	375-95-1
PFDA	Perfluorodecanoic acid	335-76-2
PFUnA	Perfluoroundecanoic acid	2058-94-8
PFDoA	Perfluorododecanoic acid	307-55-1
PFTrDA	Perfluorotridecanoic acid	72629-94-8
PFTA	Perfluorotetradecanoic acid	376-06-7
PFBS	Perfluorobutanesulfonic acid	375-73-5
PFPeS	Perfluoropentanesulfonic acid	2706-91-4
PFHxS	Perfluorohexanesulfonic acid	355-46-4
PFHpS	Perfluoroheptanesulfonic acid	375-92-8
PFOS	Perfluorooctanesulfonic acid	1763-23-1

PFNS	Perfluorononanesulfonic acid	68259-12-1
PFDS	Perfluorodecanesulfonic acid	335-77-3
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
4:2FTS	4:2 fluorotelomersulfonic acid	757124-72-4
6:2FTS	6:2 fluorotelomersulfonic acid	27619-97-2
8:2FTS	8:2 fluorotelomersulfonic acid	39108-34-4
NMeFOSAA	N-Methylperfluorooctanesulfonamidoacetic acid	2355-31-9
NEtFOSAA	N-Ethylperfluorooctanesulfonamidoacetic acid	2991-50-6
PFOSA	Perfluorooctanesulfonamide	754-91-6
NMeFOSA	N-Methylperfluorooctanesulfonamide	31506-32-8
NEtFOSA	N-Ethylperfluorooctanesulfonamide	4151-50-2
NMeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
NEtFOSE	N-Ethylperfluorooctanesulfonamidoethanol	1691-99-2
HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6
ADONA	4,8-dioxa-3H-perfluorononanoic acid	919005-14-4
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
3:3 FTCA	2H, 2H, 3H, 3H-perfluorohexanoic acid	356-02-5
5:3 FTCA	2H, 2H, 3H, 3H-perfluorooctanoic acid	914637-49-3
7:3 FTCA	2H, 2H, 3H, 3H-perfluorodecanoic acid	812-70-4
NFDHA	Perfluoro-3,6-dioxaheptanoic acid	151772-58-6
PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7

PFMPA	Perfluoro-4-methoxybutanoic acid	377-73-1
PFMBA	Perfluoro-4-methoxybutanoic acid	863090-89-5

REFERENCES

1. Analysis of fluorotelomer alcohols, fluorotelomer acids, and short- and long-chain perfluorinated acids in water and biota. 2005. J. Chrom. A. 1093, 89-97.
2. Recent developments in trace analysis of poly- and perfluoroalkyl substances. Analytical and Bioanalytical Chemistry 2011, 400, (6), 1625-1635. Analytical Chemistry 2007, 79, (11), 39663973.
3. Challenges in perfluorocarboxylic acid measurements. Analytical Chemistry 2007, 79, (11), 3966-3973.
4. EPA Method 537.1, Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/tandem Mass Spectrometry (LC/MS/MS), EPA Document #: EPA/600/R-18/352 Version 1.0, November 2018 National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency**.**
5. DoD QSM (US Department of Defense Quality Systems Manual for Environmental Laboratories, version 5.3, 2019)
6. Perfluoroalkyl Substances in the Environment: Theory, Practice and Innovation. 1st edition. 2018 Boca Raton, FL: CRC Press (Environment and Occupational Health, 2).
7. Effects of Sample Storage on the Quantitative Determination of 29 PFAS: Observation of Analyte Interconversions during Storage, Environ. Sci. Technol. 2019, 53, 21, 12576-12585

TABLE OF CONTENTS

LIST OF TABLES

A. ANALYTES AND NOMINAL REPORTING LIMITS

Typical reporting limits for the following 40 specific per- and polyfluoroalkyl substances (PFAS) (Table 2) are shown in Table 2 below, for the method default sample sizes:

Table 2. Target Analytes and Nominal Reporting Limits

Analyte Groups	Aqueous Sample	Leachate Sample	Solid[2]	Biosolid[3]	Tissues[4]
Typical Sample Size	0.5 L	0.1 L	5 g	2.5 g	2.0 g
Units	ng/L	ng/L	ng/g	ng/g	ng/g
PFBA	6.4	32	0.8	1.6	2.0
PFPeA	3.2	16	0.4	0.8	1.0
PFHxA	1.6	8	0.2	0.4	0.5
PFHpA	1.6	8	0.2	0.4	0.5
PFOA	1.6	8	0.2	0.4	0.5
PFNA	1.6	8	0.2	0.4	0.5
PFDA	1.6	8	0.2	0.4	0.5
PFUnA	1.6	8	0.2	0.4	0.5
PFDoA	1.6	8	0.2	0.4	0.5
PFTrDA	1.6	8	0.2	0.4	0.5
PFTA	1.6	8	0.2	0.4	0.5
PFBS	1.6	8	0.2	0.4	0.5
PFPeS	1.6	8	0.2	0.4	0.5
PFHxS[1]	1.6	8	0.2	0.4	0.5
PFHpS	1.6	8	0.2	0.4	0.5
PFOS[1]	1.6	8	0.2	0.4	0.5
PFNS	1.6	8	0.2	0.4	0.5
PFDS	1.6	8	0.2	0.4	0.5
PFDoS	1.6	8	0.2	0.4	0.5
4:2FTS	6.4	32	0.8	1.6	2.0

6:2FTS	6.4	32	0.8	1.6	2.0
8:2FTS	6.4	32	0.8	1.6	2.0
PFOSA	1.6	8	0.2	0.4	0.5
NMeFOSA	1.6	8	0.2	0.4	0.5
NEtFOSA	1.6	8	0.2	0.4	0.5
NMeFOSAA[1]	1.6	8	0.2	0.4	0.5
NEtFOSAA[1]	1.6	8	0.2	0.4	0.5
NMeFOSE	16	80	2.0	4.0	5.0
NEtFOSE	16	80	2.0	4.0	5.0
HFPO-DA	6.4	32	0.8	1.6	2.0
ADONA	6.4	32	0.8	1.6	2.0
9Cl-PF3ONS	6.4	32	0.8	1.6	2.0
11Cl-PF3OUdS	6.4	32	0.8	1.6	2.0
NFDHA	3.2	16	0.4	0.8	1.0
PFMBA	3.2	16	0.4	0.8	1.0
PFMPA	3.2	16	0.4	0.8	1.0
PFEESA	3.2	16	0.4	0.8	1.0
3:3 FTCA	8.0	40	1.0	2.0	2.5
5:3 FTCA	40	200	5.0	10.0	12.5
7:3 FTCA	40	200	5.0	10.0	12.5

[1] A standard containing a mixture of branched and linear isomer of suitable quality to be used for quantitation is currently available and required to be used for all calibration, calibration verifications, and QC samples. If more become commercially available for other target analytes, they must be utilized in the same manner.

[2] A maximum of 10 g wet, or 5 g dry, solid may be analyzed.

[3] A maximum of 5 g wet, or 0.5 g dry, biosolid may be analyzed.

[4] A maximum of 2 g of tissue may be analyzed.

B. CONTAMINATION AND INTERFERENCES

Samples and reagents should not come into contact with fluorpolymers, which may contain target analytes. Samples in aqueous media should not come into contact with polypropylene to avoid any adsorption effects. Polypropylene is allowed only for organic solutions, for aqeous solutions HDPE is used. The use of glassware for tranferral procedures and standard preparation is allowed, but standards and sample extracts are stored in HDPE or polypropylene containers.

C. SAFETY

Refer to laboratory Safety Manual and associated Standard Operating Procedures (SOPs).

D. POLLUTION PREVENTION AND WASTE MANAGEMENT

Application of this method must be compliant with all federal, provincial/state and municipal regulations governing waste management, including land disposal restrictions and sewage discharge regulations. All standards are prepared in volumes consistent with volumes required by the method to minimize the disposal of standards. The laboratory safety manual governs the safe storage, labeling and disposal of laboratory wastes.

E. DEFINITIONS AND GLOSSARY OF TERMS

- Method Analytes – Target analytes, List of PFAS that can be reported using this method.
- US DoD: Department of Defense, USA.
- Extracted Internal Standards (EIS): Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution quantitation and for internal standard quantitation. They may also be referred to as surrogate standards or quantification standards.
- Injection Internal Standards (IIS): Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction/cleanup and prior to instrumental analysis. They may also be referred to as recovery standards or quantification standards.

F. METHOD PERFORMANCE

This method must be validated to demonstrate that it is fit for the intended use. Validation of the method must meet the requirements specified by the DoD Quality Systems Manual for Environmental Laboratories [5] (current version).

Method performance is measured on a continual basis through the analysis of QC samples (blanks, laboratory control samples, and sample duplicates) run in conjunction with field samples. Sample specific recoveries are monitored by determination of EIS recovery.

In addition to specific method performance criteria, this method is restricted to use by, or under the supervision of personnel appropriately experienced and trained in the use of high performance liquid chromatography using tandem mass spectrometry and PFAS analysis. Analysts should also be skilled in the interpretation of chromatograms and mass spectra.

G. METHOD FLEXIBILITY

The procedures detailed in this method have been validated as fit for purpose to achieve the reporting limits and all quality criteria. However, a laboratory performing this method has flexibility on the following parts of the method in addition to general flexibility on reagents and standard laboratory equipment as long as method specifications are met or exceeded.

1) Instrumentation: Any HPLC/UPLC-MS/MS system is acceptable if all method requirements are met.
2) Analytical column: Any column is acceptable if all method requirements are met.
3) Weak Anion Exchange: Any manufacturer of WAX cartridge can be used. WAX cleanup is mandatory. Cleaning, rinsing, preconditioning and elution procedures are provided as guidelines. Specific procedures must be developed and validated in the laboratory prior to routine use.
4) Standards: The laboratory has flexibility in calibration design as long as minimum method requirements on reporting limits, isotope dilution coverage and number of calibration standards are met. All analytes with commercially available stable isotope analogues must be quantified using isotope dilution.

ANALYSIS PROCEDURES

1. PRESERVATION AND STORAGE

Samples are shipped at 0 - 4°C. Sample containers must be HDPE containers that have been verified as PFAS-free. All containers should be tightly sealed with screw cap lids. Sample extracts must be stored at 0 - 4°C and have a hold time of 30 days.

Table 3. Sample Storage Requirements

Matrix	Sample Size (per analysis)	Sample Container[1]	Sample Condition Upon Receipt	Storage Condition[2]	Sample Hold Time[3]	Extract Hold Time[4]	Preservation
Aqueous	Up to 1000 mL, but typically 500 mL or less (max. 50 mg solids)	High density polyethylene (HDPE)	0 - 4°C, dark	≤ -20°C, dark	90 days	30 days	None Required

Solid	Up to 5 g dry but not more than 10 g wet	High density polyethylene (HDPE)	0 - 4°C, dark	≤ -20°C, dark	1 year	30 days	None Required
Biosolid	Up to 0.5 g dry but no more than 5 g wet	High density polyethylene (HDPE)	0 - 4°C, dark	≤ -20°C, dark	1 year	30 days	None Required
Tissue	Up to 2 g (wet)	High density polyethylene (HDPE) or amber glass jar	0 - 4°C, dark	≤ -20°C, dark	1 year	30 days	None Required

[1] HDPE containers are required, amber glass containers are acceptable for tissue samples

[2] Storage temperatures quoted are nominal temperatures

[3] Hold times are from time of sampling. Project negotiated requests for specific holding times or other method-specific holding times are adhered to. This 90-day holding time on freezing of aqueous samples is based on the SGS AXYS Storage Stability Studies (Reference 7).

[4] Hold times for sample extracts are from time of extraction with storage at 4°C. This 30-day holding time is a guideline, longer hold times may be accepted based on professional judgement.

2. SAMPLE PRETREATMENT AND PREPARATION

2.1 Aqueous Samples

Homogenize the sample by shaking and allow the sample to settle. Do not filter the sample. This method is applicable to samples containing up to 50 mg of suspended solids. The standard procedure is to analyze the entire sample including basic methanol rinse of the container.

Some analytes in high-level PFAS containing samples, surfactant aqueous solutions, or AFFF affected samples may be stratified. Subsampling is done on a project specific basis only. IF prescreening indicates that subsampling for these samples is required, and, if subsampling is approved by the project manager, subsamples are collected about ½ cm below the top surface of the aqueous sample. If foam is present in a sample the subsample should be taken ½ cm below the top surface excluding any foam. Subsampling is only allowable with project manager specific agreement of protocol.

2.2 Solid/Biosolid Samples

Where solid samples are identified as an agricultural hazard or a biohazard, follow sample handling protocols specified in the Safety Manual and associated SOPs.

Following project instructions, standing water is either decanted and discarded before homogenization or mixed into the sample during homogenization. Only clear water can be decanted – water containing visible suspended solids in considered part of the sample.

Following project instructions vegetation can either be removed from the sample before homogenization or cut into small pieces with a scalpel or scissors and included in the sample.

If possible (500 mL jar or less and no more then ¾ full) use a stainless spoon to mix the sample in its original jar. If it is impractical to mix the sample within its container transfer the sample to a larger container. Remove rocks, invertebrates and foreign objects. Mix the sample thoroughly with a disposable spoon, stirring from the bottom to the top and in a circular motion along the sides of the jar. Make a reasonable effort to break particles to less than 1 mm by pressing against the side of the container. The homogenized sample should be even in colour with no layers present.

Store the homogenized material in its original container or in multiple smaller containers.

2.2.1 Pre-screening of Known or Suspected High Level Solid Samples

Samples that are known or suspected to contain high levels of analytes are subject to the following pre-screening procedure. Weigh 1 (±0.1) g sample into a 50 mL polypropylene centrifuge tube. Use an empty tube for the blank.

1. Add 20 mL of methanolic ammonium hydroxide (0.3%). Vortex and mix on a shaker table for 10 min. Allow to settle and/or centrifuge to produce a clear extract.
2. Filter using a single step filter vial:
   a. Add 20 µL of routine extracted internal standard (EIS, surrogate) to the chamber part.
   b. Add 400 µL of clear extract (i.e. by adding extract until it reaches the fill line), carefully vortex to mix.
   c. Insert filter/plunger part and filter by pushing all the way down.
3. Using a pipettor, transfer 30 µL of filtered extract to a ~300 µL polypropylene microvial and dilute to 300 µL by adding methanolic ammonium hydroxide (0.3%) using a disposable glass pipet.
4. Label this porting as 10X dilution.
5. Submit the diluted microvial for instrumental analysis.

Calculate results assuming that exactly 1 g sample was used, which is equivalent to a sample weight of 0.02 g (1 g X 0.4 mL/20 mL) and spiked with 25 µL of EIS.

Note that The EIS concentration in the undiluted portion is 5X greater than in regular sediment samples, but in the diluted portion it is only 0.5X the regular level.

2.3 Tissue Samples

All equipment used in the filleting, dissecting, shucking, compositing and homogenization of tissue is cleaned by washing with lab detergent and hot tap water, rinsing with tap water followed by ultra-pure water and then rinsing with acetone, toluene and dichloromethane (three times with each). All scalpels, spatulas and scissors are stainless steel. All solvents and containers used have been proofed and approved for use. The analyst wears only polyethylene gloves that have been approved for use for homogenization procedures, minimizes contact between glove and sample, and ensures

solvents do not come in contact with the gloves.

Several layers of clean aluminum foil (dull side up) are used as the work surface for preparing tissues for homogenization. New foil and clean equipment are used for each sample and the analyst ensures the samples are physically isolated from one another. Samples are handled in a semi-thawed state and are prepared for compositing and /or homogenization as required by the project. Each prepared tissue is placed into a clean stainless steel bowl for compositing.

All tissue comprising a sample is collected in a stainless steel bowl as it is prepared. The tissue is composited by first mixing it in the bowl using a stainless steel spoon. Then the entire sample is processed using the appropriate equipment to complete compositing and homogenization.

Blender: Use a homogenization jar appropriate for the amount of material to be processed. (between 60 mL and 500 mL). The jar should be no more than 2/3 full during homogenization. Run the blender until the sample is completely pureed. Stop and mix in the jar with a spoon and continue blending for 30 seconds. Repeat.

Grinder: After the entire sample has gone through the grinder, mix the ground tissue with a spoon and transfer back to the grinder to and repeat the grinding two more times.

Transfer the homogenized tissues to labeled clean jars as directed by the Project Plan. Single or multiple amber glass, HDPE or polypropylene containers can be used, depending on the project plan.

3. MATERIALS AND REAGENTS

3.1 Equipment

Syringes and pipettes must be validated and deemed fit for use. Note that brand names are provided as an example,

- 5 mL Norm-Ject syringe, polypropylene/HDPE
- Class A volumetric flasks
- Disposable glass pipets
- Calibrated mechanical pipettes or Hamilton graduated syringes
- HDPE bottles, 60 mL
- Disposable polypropylene centrifuge tubes (50 mL)
- Disposable polypropylene collection tubes (13 x 100 mm, 8 mL)
- Balance – Top loading and analytical (2, 3, & 4 decimal-place)
- Solid-phase extraction (SPE) cartridges (Waters Oasis WAX 150 mg, catalogue # 186002493 or equivalent)
- Balance – Top loading and analytical (2, 3, & 4 decimal-place)
- Vortex mixer
- Ultrasonic mixer
- Variable volume pipettes with disposable HDPE or polypropylene tips (10 µL to 5 mL) – used for preparation of calibration standards and spiked samples. Bias and

precision performance checks shall be done daily before use (refer to SLA-088 "Use and Maintenance of Syringes and Pipettes").
- pH Paper, range 0-14 - (Sigma-Aldrich, Panpeha), 0.5 unit readability.
- Centrifuge (Thermo Scientific Legend RT+, 16 cm rotor, or equivalent), capable of at least 3000 rpm
- Vacuum manifold for SPE Cartridges
- 300 µL polypropylene snap cap/crimp top vials (12 x 32 mm)
- 1 mL polypropylene crimp/snap vials
- 11 mm clear snap cap, PVDC film/white silicone.
- Single step filter vials (Thomson SINGLE STEP Standard Filter Vials, 0.2 µm Nylon membrane, with Preslit PTFE/silicone caps).
- Silanized glass wool – solvent rinsed, in accordance with documented procedure, is used to aid in filtration through the SPE cartridges.
- 25 mm disposable syringe filter, 0.2 µm Nylon membrane, PALL/Acrodisc
- TurboVap LV Automated Solvent Evaporation System or equivalent
- TurboVap tubes: 60 mL clear glass vial, 24 mm Solid Teflon Lined Cap, Precleaned from Proclean Environmental. Discard cap and do not use for this method. Cap with foil if required.
- 10 mg scoops, polypropylene or stainless steel

3.2 Instruments

- Ultra high performance liquid chromatograph (UPLC also called UHPLC) or high performance liquid chromatograph (HPLC) equipped with a Waters Xevo TQ-S Micro mass spectrometer, running TargetLynx/MassLynx software, or equivalent.
- Waters Acquity UPLC ® BEH C18 column, 1.7 µm, 50 x 2.1 mm
- Phenomenex Evo C18 guard column
- Trap/delay column

3.3 Cleaning of equipment and disposable ware

Clean all equipment prior to, and after each use to avoid PFAS cross-contamination. Typical cleaning solvents used include water, methanol, and methanolic ammonium hydroxide. The residual PFAS content of disposable plasticware must be verified by batch/lot number and used without cleaning if PFAS levels are less than half the reporting limit established. Cleaning procedures must be documented in the laboratory SOP/referenced documents.

3.3.1 Cleaning of Glassware

All glass equipment that is used in the preparation of reagents is cleaned by baking and washing using standard operating procedures. Prior to use, baked glassware must be solvent rinsed with methanolic ammonium hydroxide (1%), toluene and methanol, and then air dried (or rinsed with reagent water if required immediately).

3.3.2 Cleaning of Vacuum Manifold for SPE Cartridges

The top part of the SPE manifold (the head) is cleaned between samples by sonicating each side for 10 minutes in methanolic ammonium hydroxide (1%). The solvent is discarded. The piece is air dried prior to use.

The needles, red adapters and stopcocks associated with the manifold are cleaned by rinsing them with tap water and after draining the water completely, sonicating in methanolic ammonium hydroxide (1%) for 20 minutes. The solvent is discarded. The needles, red adapters and stopcocks are air dried prior to use.

The manifold reservoirs are rinsed with tap water and after draining the water completely, rinsed with methanolic ammonium hydroxide (1%). The narrow parts (bottom) are cleaned by sonicating in methanolic ammonium hydroxide (1%) for 20 minutes. The solvent is discarded. The reservoirs are air dried prior to use.

The glass chamber is cleaned by rinsing with methanolic ammonium hydroxide (1%) between analysis batches. When in use, after loading the samples but prior to elution procedures, the chamber is removed and rinsed with methanolic ammonium hydroxide (1%)

3.4 Preparation of Reagents

Acetic acid - (ACS grade or equivalent), used as received, store at room temperature, indefinite shelf life.

Acetic acid (0.1%) - is prepared by dissolving acetic acid (1 mL) in reagent water (1 L), store at room temperature, shelf life 3 months.

Acetonitrile – (UPLC grade or equivalent) is verified before use, store at room temperature, indefinite shelf life.

Ammonium acetate - (reagent grade, Sigma or equivalent) is used as received, store at 2-8° C, shelf life 2 years after opening.

Ammonium hydroxide - (Fisher, certified ACS+ grade or equivalent, 30% in water) is used as received, store at room temperature, indefinite shelf life.

Aqueous ammonium hydroxide (0.3%) - is prepared by adding ammonium hydroxide (1 mL, 30%) to reagent water (99 mL), store at room temperature, shelf life 3 months.

Aqueous ammonium hydroxide (3%) - is prepared by adding ammonium hydroxide (10 mL, 30%) to reagent water (90 mL), store at room temperature, shelf life 3 months.

Methanolic ammonium hydroxide (0.3%) - is prepared by adding ammonium hydroxide (1 mL, 30%) to methanol (99 mL), store at room temperature, shelf life 1 month.

Methanolic ammonium hydroxide (1%) - is prepared by adding ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, shelf life 1 month.

Methanolic ammonium hydroxide (2%) - is prepared by adding ammonium hydroxide (6.6 mL, 30%) to methanol (93.4 mL), store at room temperature, shelf life 1 month.

Methanolic potassium hydroxide (0.05 M) – is prepared by adding 3.3 g of potassium hydroxide to 1 L of methanol, store at room temperature, shelf life 3 months.

Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - is prepared by adding ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92mL), store at room temperature, shelf life 1 month.

Eluent A - Acetonitrile (Ultra LCMS grade, or equivalent), indefinite shelf life.

Eluent B - 2 mM ammonium acetate in 95:5 water:acetonitrile. Prepared by dissolving 0.154 g of ammonium acetate in 950 mL of water (Caledon Ultra LC/MS grade, or equivalent) and 50 mL of acetonitrile (Caledon Ultra LCMS grade, or equivalent). Store at room temperature, shelf life 2 months.

Formic acid - (greater than 96% purity or equivalent) is used as received, verified by lot number before use, store at room temperature, indefinite shelf life.

Formic acid (aqueous, 0.1 M) - is prepared by dissolving formic acid (4.6 g) in reagent water (1 L), store at room temperature, shelf life 2 years.

Formic acid (aqueous, 0.3 M) - is prepared by dissolving formic acid (13.8 g) in reagent water (1 L), store at room temperature, shelf life 2 years.

Formic acid (aqueous, 5% v/v) - is prepared by mixing 5 mL formic acid with 95 mL reagent water, store at room temperature, shelf life 2 years.

Formic acid (aqueous, 50% v/v) - is prepared by mixing 50 mL formic acid with 50 mL reagent water, store at room temperature, shelf life 2 years.

Formic acid (methanolic, 1:1, 0.1 M formic acid:methanol) - is prepared by mixing equal volumes of methanol and 0.1 M formic acid, store at room temperature, shelf life 2 years.

Methanol - (HPLC grade or better, 99.9% purity) is used as received, verified by lot number before use, store at room temperature, indefinite shelf life.

Reagent water – Laboratory reagent water: Test by lot/batch number for residual PFAS content

Carbon - EnviCarb®, or equivalent) is used as received, verified by lot number before use, store at room temperature, indefinite shelf life.

3.5 Preparation of Standard Solutions

Prepare stocks and working standard solutions according to laboratory standard operating procedures. Working standard concentrations are provided as one working example. It is acceptable to use different concentrations as defined in the laboratory SOP. However, the standards must result in equivalent, or better method performance.

3.5.1 Extracted Internal Standard (EIS, Surrogate Standard)

Prepare the EIS solution containing the EIS compounds listed in Table 4 in methanol from prime stocks. Base is not added to the standard solutions, but some prime standards have a small amount of base added by the manufacturer. An aliquot of EIS solution, typically 50 µL, is added to each sample prior to extraction. Table 5 presents the nominal amounts of EIS compounds added to each sample.

3.5.2 Injected Internal Standard (IIS, Recovery Internal Standard)

The IIS solution containing the injection internal standard compounds listed in Table 4 is prepared in methanol from prime stock. Base is not added to the standard solutions, but some prime standards have a small amount of base added by the manufacturer. An aliquot of IIS solution, typically 50 µL, is added to each sample prior to instrumental analysis. Table 6 presents the nominal amounts of IIS compounds added to each sample.

3.5.3 Method Analyte Solutions

Prepare a spiking solution, containing the method analytes listed in Table 4, in methanol from prime stocks. Base is not added to the standard solutions, but some prime standards have a small amount of base added by the manufacturer. The solution is used to prepare the calibration standards and to spike the known reference QC sample that is analyzed with every batch. Quantitative standards containing a mixture of branched and linear isomers must be used for method analytes if they are commercially available. Currently, these include PFOS, PFHxS, NEtFOSAA, and NMeFOSAA.

3.5.4 Calibration Standards

A series of calibration solutions containing method analytes and ^{13}C, ^{18}O and deuterium labeled EIS and IIS are used to establish the initial calibration of the analytical instrument. The concentration of the method analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the EIS and IIS remain constant. The calibration solutions are prepared using methanol, methanolic ammonium hydroxide (2.0%), water, acetic acid and the method analyte and labelled compound standard solutions. They are made to match the solvent mix of sample extracts which contain methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid, and do not undergo solid phase extraction/cleanup. Concentrations for eight calibration solutions are presented in Table 4. A minimum of 5 contiguous calibrations standards is required for valid analysis. The lowest level calibration solution used for initial calibration that meet a signal:noise ratio of 3:1 and must be at a concentration less than or equal to the Limit of Quantitation (LOQ). All initial calibration requirement listed in Table 7 must be met.

3.5.5 Qualitative Standards

If qualitative standards (technical grade) that contain a mixture of branched and linear isomers standards are available (for those quantitative standards are not available for), a standard is made

for comparison to suspected branched isomer peaks in field samples. Qualitiative standards that are currently commercially available include PFOA, PFOSA, NMeFOSE, and NEtFOSE

3.6 Validation of Standard Solutions

Prepare standard solutions in uniquely named batches and validate for accuracy by comparative analysis against second source standards prior to use. A record of the validation and approval for use must be retained on file with the preparation records.

Table 4. Nominal Concentrations of Calibration Solutions (ng/mL)

	CAL A	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I
Perfluoroalkyl carboxylates									
PFBA	0.2	0.4	0.8	2	5.0	10	20	50	250
PFPeA	0.1	0.2	0.4	1	2.5	5	10	25	125
PFHxA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFHpA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFOA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFNA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFDA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFUnA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFDoA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFTrDA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFTA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Perfluoroalkyl sulfonates									
PFBS	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFPeS	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFHxS	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFHpS	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFOS	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFNS	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFDS	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
PFDoS	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5

	CAL A	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I
Flurorotelomer sulfonates									
4:2FTS	0.2	0.4	0.8	2	5	10	20	50	250
6:2FTS	0.18	0.36	0.72	1.8	4.51	9.01	18.03	45.07	225.4
8:2FTS	0.2	0.4	0.8	2	5	10	20	50	250
Perfluorooctane sulfonamides									
PFOSA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
NMeFOSA	0.0575	0.115	0.23	0.575	1.4375	2.875	5.75	12.5	62.5
NEtFOSA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Perfluorooctane sulfonamidoacetic acids									
NMeFOSAA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
NEtFOSAA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Perfluorooctane sulfonamide ethanols									
NMeFOSE	0.5	1.0	2.0	5	12.5	25	50	125	625
NEtFOSE	0.375	0.75	1.5	3.75	9.37	18.74	37.49	93.75	468.75
Per- and Polyfluoroether carboxylates									
HFPO-DA	0.19	0.38	0.76	1.9	4.75	9.5	19	47.5	237.6
ADONA	0.2	0.4	0.8	2	5.0	10	20	50	250
PFMPA	0.1	0.2	0.4	1	2.5	5	10	25	215
PFMBA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
NFDHA	0.1	0.2	0.4	1	2.5	5	10	25	125
Ether sulfonates									
9Cl-PF3ONS	0.2	0.4	0.8	2	5.0	10	20	50	250
11-Cl-PF3OUdS	0.2	0.4	0.8	2	5.0	10	20	50	250
PFEESA	0.05	0.1	0.2	0.5	1.25	2.5	5.0	12.5	62.5
Fluorotelomer carboxylates									
3:3 FTCA	0.2	0.4	0.8	2	5	10	20	50	250
5:3 FTCA	1.25	2.5	5	12.5	31.3	62.5	125	315	1560

	CAL A	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I
7:3 FTCA	1.25	2.5	5	12.5	31.3	62.5	125	315	1560
Extracted Internal Standards (EIS)									
$^{13}C_4$-PFBA	10	10	10	10	10	10	10	10	10
$^{13}C_5$-PFPeA	5	5	5	5	5	5	5	5	5
$^{13}C_5$-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_4$-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_8$-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_9$-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_6$-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_7$-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_2$-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_2$-PFTA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_3$-PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_3$-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_8$-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_2$-4:2FTS	5	5	5	5	5	5	5	5	5
$^{13}C_2$-6:2FTS	5	5	5	5	5	5	5	5	5
$^{13}C_2$-8:2FTS	5	5	5	5	5	5	5	5	5
$^{13}C_8$-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_3-NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_3-NMeFOSAA	5	5	5	5	5	5	5	5	5
D_5-NEtFOSAA	5	5	5	5	5	5	5	5	5
D_7-NMeFOSE	25	25	25	25	25	25	25	25	25
D_9-NEtFOSE	25	25	25	25	25	25	25	25	25
$^{13}C_3$-HFPO-DA	10	10	10	10	10	10	10	10	10
Injection Internal Standards (IIS)									
$^{13}C_3$-PFBA	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
$^{13}C_2$-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5

	CAL A	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I
$^{13}C_4$-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_5$-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_2$-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{18}O_2$-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_4$-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5

Table 5. Nominal Amounts of EIS Analytes Added to Sample

Compound	Solution Type	Amount Spiked, ng
$^{13}C_4$-PFBA	EIS	40
$^{13}C_5$-PFPeA	EIS	20
$^{13}C_5$-PFHxA	EIS	10
$^{13}C_4$-PFHpA	EIS	10
$^{13}C_8$-PFOA	EIS	10
$^{13}C_9$-PFNA	EIS	5
$^{13}C_6$-PFDA	EIS	5
$^{13}C_7$-PFUnA	EIS	5
$^{13}C_2$-PFDoA	EIS	5
$^{13}C_2$-PFTA	EIS	5
$^{13}C_3$-PFBS	EIS	10
$^{13}C_3$-PFHxS	EIS	10
$^{13}C_8$-PFOS	EIS	10
$^{13}C_2$-4:2FTS	EIS	20
$^{13}C_2$-6:2FTS	EIS	20
$^{13}C_2$-8:2FTS	EIS	20

$^{13}C_8$-PFOSA	EIS	10
D_3-NMeFOSA	EIS	10
D_5-NEtFOSA	EIS	10
D_3-NMeFOSAA	EIS	20
D_5-NEtFOSAA	EIS	20
D_7-NMeFOSE	EIS	100
D_9-NEtFOSE	EIS	100
$^{13}C_3$-HFPO-DA	EIS	40

Table 6. Nominal Amounts of IIS Added to Sample

Compound	Solution Type	Amount Spiked, ng
$^{13}C_3$-PFBA	IIS	20
$^{13}C_2$-PFHXA	IIS	10
$^{13}C_4$-PFOA	IIS	10
$^{13}C_5$-PFNA	IIS	5
$^{13}C_2$-PFDA	IIS	5
$^{18}O_2$-PFHXS	IIS	10
$^{13}C_4$-PFOS	IIS	10

4. QUALITY ASSURANCE/QUALITY CONTROL

All samples are analyzed in batches. The composition of a batch is detailed on a batch sheet. Each batch has the following composition:

- Preparatory Batch Size - Each sample preparatory batch consists of up to 20 field samples and additional QC samples.
- Method Blank (MB) – One blank must be prepared with every preparatory batch. For aqueous field samples, the associated MB are prepared using a volume of reagent water that is

matched to the typical volume of field samples to be prepared in the preparatory batch. For soil and sediment field samples, the associated MB is 5 g of an approved clean solid matrix (sand) wetted with 2.5 g of reagent water. For tissue sample, the associated MB is 2 g of an approved clean tissue matrix.

- Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – One Laboratory Control Sample (LCS) must be prepared with every preparatory batch. If an MS/MSD cannot be performed on a sample in the preparatory batch due to insufficient sample volume received, a Laboratory Control Sample Duplicate (LCSD) must be included in the preparatory batch. For aqueous field samples, associated LCS and LCSDs are prepared by adding an aliquot of native standard to reagent water. The LCS/LCSD water volume is matched to the typical volume of field samples to be prepared in the preparatory batch. For soil and sediment field samples, associated LCS and LCSDs are an aliquot of method analyte standards added to 5 g of an approved clean solid matrix (sand) wetted with 2.5 g of reagent water. For tissue sample, associated LCS and LCSDs are an aliquot of method analyte standards added to 2 g of an approved clean tissue matrix.
- Limit of Quantitation (LOQ or EPA LLOQ) Verification Sample - One Limit of Quantitation (LOQ or EPA LLOQ) Verification Sample must be prepared with every preparatory batch. For aqueous field samples, associated LOQ Verification is prepared by adding an aliquot of native standard to reagent water. The LOQ Verification water volume is matched to the typical volume of field samples to be prepared in the preparatory batch. For soil and sediment field samples, the associated LOQ Verification is an aliquot of method analyte standards added to 5 g of an approved clean solid matrix (sand) wetted with 2.5 g of reagent water. For tissue sample, the associated LOQ Verification is an aliquot of method analyte standards added to 2 g of an approved clean tissue matrix.
- Matrix Spike/Matrix Spike Duplicate (MS/MSD) – A minimum of one Matrix Spike (MS) and one Matrix Spike Duplicate (MSD) must be prepared with every preparatory batch.
- Extracted Internal Standards (EIS) – EIS analytes are spiked into every standard, QC sample, blank, and field sample. EIS analytes are added to solid field sample and solid QC sample prior to extraction. For aqueous field samples, EIS analytes must be added directly into the original field sample container, prior to extraction.
- Injection Internal Standards (IIS) – IIS analytes are spiked into every standard, QC sample, blank, and field sample prior to instrumental analysis. IIS analytes are added to an aliquot of each prepared standard, instrument blank, prepared QC sample, and field sample prior to instrumental analysis.

All of the specifications in Table 7 must be met in order to report data.

Table 7. QC Specification Table

QC Parameter	Specification
MS Acquisition Rate	Minimum rate for every method analyte, EIS analyte, and IIS analyte of at least 10 spectral scans acquired across each peak
Instrument Sensitivity	All method analytes detected in field samples, QC samples, and standards must have a S:N ≥ 3:1 for transition product ion used for quantitation and for confirmation.
Mass Calibration	Instrument must have a valid mass calibration following the manufacturer specified procedure prior to any sample analysis. The mass calibration is updated on an as-needed basis (e.g. QC failures, ion masses fall outside of the instrument required mass window, major instrument maintenance, or if the instrument is move).
Mass Calibration Verification	Mass calibration must be verified after each mass calibration, prior to any sample analysis. Mass calibration must be performed per the instrument manufacturer's instructions. A mass calibration verification must be performed using standards whose mass range brackets the masses of interest (quantitative and qualitative ions).
Initial Calibration (ICAL)	Run initially, and as required to maintain compliance with calibration verification and instrument sensitivity. The isotopically labeled analog of an analyte (EIS analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation). If a labeled analog is not commercially available, the EIS Analyte with the closest retention time or chemical similarity to the method analyte must be used for quantitation. Method Analytes must be within 70-130% of their true value for each calibration standard. ICAL must meet one of the three options below: Option 1: The RSD of the RFs for all method analytes must be ≤20%. Option 2: Linear or non- linear calibrations must have r^2 ≥ 0.99 for each method analyte. Option 3: The% RSE for all method analytes must be ≤20%.
Retention Time (RT) window position establishment	Once per ICAL and at the beginning of the analytical sequence. Position of method analyte, EIS analyte, and IIS analyte peaks shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV retention times or the midpoint standard of the ICAL curve can be used to establish the RT window position.
Retention Time (RT) window width	Method analyte, EIS analyte, and IIS analyte RTs must fall within 0.4 minutes of the predicted retention times from the midpoint standard of the ICAL or initial daily CCV, whichever was used to establish the RT window position for the analytical batch. All branched isomer peaks identified in

QC Parameter	Specification
	either the calibration standard or the qualitative (technical grade) standard must fall within in the retention time window for that analyte. For all method analytes with exact corresponding isotopically labeled analogs, method analytes must elute within 0.1 minutes of the associated EIS.
Extracted Internal Standard (EIS) Analytes	Must be added to every field sample, standard, blank, and QC sample. Recoveries of the EIS analytes are calculated by internal standard quantification against the IIS using an average RRF. Recovery criteria for EIS analytes in instrument blanks and standards is 70% to 130%. Interim recovery criteria for EIS analytes in field samples and preparatory QC samples is 50% to 200%. This criteria is subject to change pending results from the multi-laboratory method validation study.
Injection Internal Standards (IIS)	Must be added to every prepared field sample, standard, blank, and QC sample prior to instrumental analysis. IIS analyte recovery is calculated determined against the average IIS analyte area of the ICAL standards. Acceptance criteria is pending results from the multi-laboratory method validation study.
Initial Calibration Verification (ICV):	After each Initial Calibration (ICAL), prior to sample analysis; analyze a second source standard; calculated method analyte and EIS analyte concentrations must be within ±30% of their true value.
Instrument Sensitivity Check (ISC)	Daily, prior to analysis and every 12 hours thereafter. Method analyte concentration must be spiked at LOQ and calculated method analyte and EIS analyte concentration must be within ±30% of their true values.
Continuing Calibration Verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence. Concentration of analytes must range from the LOQ to the mid-level calibration concentration. If at the LOQ; this standard can serve as the ISC as well. Method analyte and EIS analyte concentrations must be within ±30% of their true value.
Instrumental Blanks	Immediately following the highest standard analyzed and daily prior to sample analysis. Concentration of each method analyte detected must be ≤ ½ the LOQ. If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest

QC Parameter	Specification
	standard until acceptance criteria is met. If sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria (>1/2 LOQ), they must be reanalyzed. No samples shall be analyzed until instrument blank has met acceptance criteria. When the highest standard analyzed is not part of the calibration curve, it cannot be used to extend out the calibration range, it is used only to document a higher concentration at which carry over still does not occur.
Method Blank (MB)	One per preparatory batch. No method analytes can be detected > ½ LOQ or >1/10th the amount measured in field samples in the batch, whichever is greater.
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	Method analyte concentration must be spiked at concentrations ≥LOQ (or LLOQ) and ≤the mid-level calibration concentration. Interim recovery criteria for method analytes in the LCS/LCSD are set at 70-130%. This criteria is subject to change pending results from the multi-laboratory method validation study. LCS/LCSD RPD for each method analyte must be ≤ 30%.
Limit of Quantitation (LOQ) (or EPA LLOQ) Verification	Method analyte concentration must be spiked at concentrations one to two times the LOQ. Interim recovery criteria for method analytes in the LCS/LCSD are set at 70-130%. This criteria is subject to change pending results from the multi-laboratory method validation study.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Method analyte concentration must be spiked at concentrations ≥ LOQ and ≤ the mid-level calibration concentration. Interim recovery criteria for method analytes in the MS/MSD are set at 70-130%. This criteria is subject to change pending results from the multi-laboratory method validation study. MS/MSD RPD for each method analyte must be ≤ 30%.

5. EXTRACTION PROCEDURES

5.1 Aqueous Samples

5.1.1 Sample Size

Typical sample size is 500 mL, however sample size may be up to 1000 mL. The MB and LCS must be of the same volume as a typical sample in the batch. This method is applicable to aqueous samples containing up to 50 mg of suspended solids per sample. The default procedure is to analyze the entire sample. Smaller sample volumes may be analyzed for samples containing solids greater than specified for this method, or when unavoidable due to high level or high-volume samples.

5.1.2 Subsampling

Subsampling of the submitted samples is done on a project specific basis and requires project manager approval. For visual guidance on solids content see Figures 5 and 6. Chemists must visually inspect each sample prior to spiking or otherwise processing the sample and compare with Figures 5 and 6. If in the judgment of the chemist the sample is likely to pose issues including clogging of the cartridge, the chemist must stop processing the sample and project manager must be notified for approval to procedure with a reduced sample size unless project manager approval has already been granted via the project contract and instructions. The sample data report must state when a sample has been prepared using a reduced sample size is processed instead of the entire sample received.

If a reduced sample size is required, transfer a weighed subsample using the following subsampling procedure to a 60 mL HDPE bottle and dilute to approximately 60 mL using reagent water. This is now the “sample bottle”.

Subsampling has been shown to increase uncertainty in PFAS analysis. For subsampling:

1. Refer to the project instructions to find out the volume prescribed for the workup of each sample.
2. Gently invert sample 3-4 times being careful to avoid foam formation.
3. Subsample immediately (do not let stand).
4. If suds form and a larger volume (more than 5 mL) is required – pour, avoid any suds. SUBSAMPLING BIAS MAY OCCUR.
5. If suds form and a smaller volume (less than 5 mL) is required – pipette below suds.
6. SUBSAMPLING BIAS MAY OCCUR
7. If no suds form – pour or pipette based on volume required.

5.1.3 Sample Processing

Do not use any fluoropolymer articles or *Kim-wipes* in these extraction procedures. Use only HDPE or polypropylene squeeze bottles and centrifuge tubes. Reagents and solvents for cleaning syringes may be kept in glass containers.

1. The volume of sample analyzed is determined by weighing the full and then the empty sample bottle. Weigh each sample (with lid) to 0.1 g.

2. Prepare the MB, LOQ Verification, LCS, and LCSD (if required) using PFAS-free water in HDPE sample bottles. Select a volume of water that is typical of the samples in the batch. Spike the LOQ Verification and LCS/LCSD samples with method analyte solutions (Section 3.5.3). Spike the field sample bottles designated for use as MS and MSD samples with method analyte solutions (Section 3.5.3).

3. Spike an aliquot of EIS solution (Section 3.5.1) directly into the sample bottle (either original bottle or subsampled bottle) and QC samples. Mix by swirling the sample container.

4. Check that pH is 6.5 ± 0.5. If necessary, adjust pH with 50% formic acid or ammonium hydroxide (or with 5% formic acid and aqueous ammonium hydroxide (3%)).

5. Collect the required number of Waters Oasis 150 mg WAX SPE cartridges and pack clean silanized glass wool to half the height of the available space in the SPE barrel.

6. Set up the vacuum manifold with pre-labeled SPE cartridges to which clean glass wool has been added. Add a reservoir and reservoir adaptor to each cartridge.

7. Prepare and pre-condition the cartridges. Without using vacuum, pass 2 full SPE cartridge volumes (~7.5 mL, each) of methanolic ammonium hydroxide (1%) and then 5 mL of 0.3 M formic acid through each cartridge. Do not allow the WAX SPE to go dry. Discard the wash solvents.

8. Load each sample onto an SPE cartridge, using a reservoir, and emptying the sample bottle as much as possible. Pour the sample into the reservoir (don't use a pipette) taking care to avoid splashing while loading. Adjust the vacuum to pull the sample through the cartridge at 5 mL/min. Retain the emptied sample bottle and allow it to air dry for later rinsing. Discard the sample being pulled through the cartridge.

9. Leaving the reservoir in place until after the sample has been eluted, rinse the walls of the reservoir thoroughly with wash solutions and pass washes through the cartridge using vacuum. The wash solutions are 2 x 5 mL reagent water (10 mL total), and 5 mL of 1:1 0.1M formic acid:methanol. Dry the cartridge by pulling air through for 15 seconds. The wash solutions are discarded.

10. Prepare the collection tubes (13 x 100 mm polypropylene) by pre-labeling them and placing them into the manifold rack, ensuring that the extract delivery needles are lined up into the collection tubes but not touching the walls of the tubes. DO NOT add IIS to the collection vials.

11. Use a total of 5 mL of elution solvent (methanolic ammonium hydroxide (1%)). First rinse the sample bottle with the cartridge elution solvent and then, using a glass pipette, transfer the bottle rinse to the reservoir, washing the walls of the reservoir. Use vacuum to pull the elution solvent through the cartridge and into the collection tubes.

12. Allow sample bottle to air dry and then weigh the empty bottle and lid to 0.1 g. Determine the mass of sample analyzed by difference and convert to volume using a nominal density of 1.0 g/mL. This is the sample volume analyzed.

13. Add 25 µL of acetic acid to each sample extract and vortex to mix. Using a 10 mg scoop, add 10 mg of carbon to teach sample and batch QC extract. Hand-shake occasionally for 5 minutes (not longer). It is important to minimize the time the sample extract is in contact with the carbon. Immediately vortex (30 seconds) and centrifuge ~2800 rpm for 10 minutes.

14. Prepare a new set of collection tubes by adding an aliquot of IIS solution (Section 3.5.2) to each tube. Do a visual check to confirm that IIS solution has been added to each tube. Use a 5 mL polypropylene syringe equipped with a syringe filter (25 mm filter, 0.2 µm nylon membrane) to filter the entire extract into the prepared collection tubes. Vortex to mix and transfer a portion into a 1 mL polypropylene microvial for LC-MS/MS analysis. Cap the collection tube containing the remaining extract and store at 4 °C as backup.

Note: If the cartridge begins to clog during sample loading, record the volume of sample that was processed and notify the project manager. If requested, load the remaining sample onto a second cartridge. To do this, carefully remove the first cartridge from the reservoir and put aside for washing and elution. Put a second pre-conditioned cartridge in place and, using the same reservoir, continue loading the sample. Wash each cartridge and elute as normal but do not use a reservoir for the first (clogged cartridge) and do not add an aliquot of IIS to its collection tube. Use the elution solvent for the second cartridge to rinse the sample bottle and reservoir as normal. If two cartridges are used, combine the elutions and mix thoroughly. Filter ~ 1 mL portion of the combined extract into a polypropylene container (13 x 100 mm). Using a pipettor, transfer a 350 µL portion of the filtered extract into a 1 mL autosampler vial and mark the level. Add another 350 µL portion and using a gentle stream of nitrogen (water bath at 40 ºC), concentrate to the 350 µL marker and submit for LC-MS/MS analysis. This concentration step is applicable to situations where 2 SPE cartridges were eluted, each with 5 mL of elution solvent.

If the second cartridge clogs as well, the sample is not appropriate for a whole water treatment at the selected sample size. Stop and contact the project manager for guidance on remedial action. Management options include proceeding with the fraction of the sample as loaded with expectation of lowered recoveries and uncertain particle loading, repeating the analysis using a smaller sample size, or SPE Cartridge Pre-Treatment and Sample Loading Summary (Aqueous samples).

5.2 Solid Samples

5.2.1 Sample Size

Weigh an aliquot of homogenized sample into a 50 mL polypropylene centrifuge tube. The maximum sample weight for sediment or soil is 5 g dry weight (up to 10 g wet weight). The maximum sample weight for biosolids is 0.5 g dry weight (up to 5 g wet).

5.2.2 Sample Processing

Do not use any fluoropolymer articles or *Kim-wipes* in these extraction procedures. Use only HDPE or polypropylene squeeze bottles and centrifuge tubes. Reagents and solvents for cleaning syringes may be kept in glass containers

1. Prepare batch QC samples using 5 g of approved clean reference solid (sand) wetted with 2.5 g of reagent water for the MB, LOQ Verification, LCS and LCSD (if required). Spike the LOQ Verification, and LCS/LCSD samples with method analyte solutions (Section 3.5.3). Spike the MS and MSD samples with method analyte solutions (Section 3.5.3).

2. Spike an aliquot of EIS solution (Section 3.5.1) directly into each sample and QC sample. Vortex and allow to equilibrate for 30 minutes.

3. Add 10 mL of methanolic ammonium hydroxide (0.3%) to each sample. Vortex to disperse and then shake for 30 minutes on a mechanical shaker table. Centrifuge at 2800 rpm for 10 minutes and collect the supernatant in a 50 mL polypropylene centrifuge tube.

4. Add 15 mL of methanolic ammonium hydroxide (0.3%) to the remaining solid sample in each centrifuge tube. Vortex to disperse and shake for 30 minutes on a mechanical shaker table. Centrifuge at 2800 rpm for 10 minutes and decant the supernatant into the centrifuge tube with supernatant.

5. Add another 5 mL of methanolic ammonium hydroxide (0.3%) to the remaining sample in each centrifuge tube. Shake by hand to disperse, centrifuge at 2800 rpm for 10 minutes and decant the supernatant into the centrifuge tube with supernatant.

6. Using a 10 mg scoop, add 10 mg of Carbon to the combined extract, mix by occasional hand shaking over a five-minute period (not longer) and then centrifuge at ~2800 rpm for 10 minutes. Immediately decant the extract into a 60 mL glass TurboVap tube.

7. Add reagent water to each TurboVap tube to dilute to 35 mL. Samples containing more than 50% water may yield extracts that are greater than 35 mL in volume. Do not add further water to these.

8. The extract is concentrated in order to remove the methanol only, not water. Concentrating extracts to different final volumes in 1 mL increments in ungraduated vials is necessary as excessive concentration at this step will cause the loss of the more volatile analytes and must be avoided. If too much methanol is present in the extract during SPE then poor recovery of C13 and C14 carboxylic acids and C10 and C12 sulfonates is observed. If the extract is concentrated too far then loss of the neutral compounds (methyl and ethyl FOSEs and FOSAs) is observed, presumably due to their volatility. Batch QC samples and all project samples that contain less than 5 mL of water are concentrated to 7 mL. Samples containing to 5 to 8 mL of water are concentrated to 8 mL, samples containing 8 to 9 mL of water are concentrated to 9 mL and samples containing 9 to 10 mL of water are concentrated to 10 mL. (This information is summarized on the flow chart). The amount of water in a sample is determined by multiplying the sample weight by the percent moisture and dividing by 100. Using the TurboVap, concentrate each extract at 55 ºC with a N_2 flow of 1.2 L/min to a final volume that depends on the water content of the sample. Allow extracts to concentrate for 25 minutes, then mix (by vortex if volume

is < 20 mL or using a glass pipette if volume is > 20 mL). Then continue concentrating, stopping and mixing every 10 minutes until volume has been reduced to the required volume. If the extract volume appears to stop reducing, the concentrating must be stopped and a note made of the volume when it was stopped. Use pre-marked empty TurboVap tubes as guides to reading the final volume.

9. Add 40-50 mL of reagent water to the extract and vortex. Check that the pH is 6.5 ±0.5. If necessary, adjust pH with 50% formic acid or ammonium hydroxide (or with 5% formic acid and aqueous ammonium hydroxide (3%)).The extracts are ready for SPE cleanup.

10. Collect and label the required number of Waters Oasis 150 mg WAX SPE cartridges. Place on the manifold and add a reservoir and adaptor to each SPE cartridge. Optional: The SPE cartridges for the entire batch may be packed with glass wool. Do not allow the WAX SPE cartridge to go dry between conditioning and sample loading.

11. Without using vacuum, pass 2 full SPE cartridge volumes (~7.5 mL, each) of methanolic ammonium hydroxide (1%) and then 5 mL of 0.3 M formic acid through each cartridge. Discard the wash solvents.

12. Load each sample onto an SPE cartridge using a reservoir. Pour the sample from the TurboVap tube. Don’t use a pipette and take care to avoid splashing while loading. Adjust the vacuum to load the extract at 5 mL/min, retaining the emptied TurboVap tube and keeping the reservoir in place for rinsing. Discard the sample eluting from the cartridge.

13. Leaving the reservoir in place until after the sample has been eluted, using a pipette, rinse the walls of the reservoir thoroughly with wash solutions and pass washes through the cartridge using vacuum. The wash solutions are 2 x 5 mL reagent water (10 mL total), and 5 mL of 1:1 0.1M formic acid:methanol. Dry the cartridge by pulling air through for 15 seconds. The wash solutions are discarded.

14. Prepare pre-labeled collection tubes (13 x 100 mm polypropylene) by adding an aliquot of IIS solution (Section 3.5.2) to each tube. Do a visual check of the tubes to confirm that IIS solution has been added to each one. Place collection tubes into the manifold rack and ensure that the extract delivery needles are lined up into the collection tubes but not touching the walls of the tubes.

15. Use a total of 5 mL of elution solvent (methanolic ammonium hydroxide (1%)). Using a glass pipette, first rinse the walls of the TurboVap tube with the cartridge elution solvent, and then use the same solution and pipette to rinse the walls of the reservoir. Use vacuum to pull the elution solvent through the cartridge and into the collection tubes that have been spiked with IIS solution.

16. Add 25 µL of acetic acid to each sample extract, swirl to mix. Use a 5 mL polypropylene syringe equipped with a syringe filter (25 mm filter, 0.2 µm nylon membrane) to filter a portion (~1 mL) of the extract into a microvial for LC-MS/MS analysis. Cap the collection tube containing the remaining extract and store at 4 °C as backup.

5.3 Tissue Samples

5.3.1 Sample Size

Sample size may be up to 2 grams (wet weight). The entire sample can be processed without splitting the extract. The LOQ Verification, LCS and LCSD (if required) are 2 g of approved clean reference tissue and the MB is 2 g of reference tissue. The project may recommend a reduced sample size for the project samples but 1 gram is the minimum required sample size (less sample will mean less water in the extract and will affect recovery of analytes after the TurboVap concentration).

5.3.2 Sample Processing

Do not use any fluoropolymer articles or Kim-wipes in these extraction procedures. Use only HDPE or polypropylene bottles and centrifuge tubes. Reagents and solvents for cleaning syringes may be kept in glass containers.

1. Prepare the batch QC samples using reference tissue for the MB (2 g) and 2 g for the LCS and LCSD (if required). Spike the LOQ Verification and LCS/LCSD samples with method analyte solutions (Section 3.5.3).

2. For each sample weigh a 2 g aliquot of homogenized tissue into a 15 mL polypropylene centrifuge tube. Spike the MS and MSD samples with method analyte solutions (Section 3.5.3).

3. Spike an aliquot of EIS solution (Section 3.5.1) directly into each sample and QC sample. Vortex and allow to equilibrate for 30 minutes.

4. Add 10 mL of 0.05M KOH in methanol to each sample. Vortex to disperse the tissue and extract on the tipper table for 16 hours. Centrifuge at 2800 rpm for 10 minutes and collect the supernatant in a 50 mL polypropylene centrifuge tube. Cover the tube when not in use.

5. Add 10 mL of acetonitrile to remaining tissue in the 15 mL centrifuge tube, vortex to mix and disperse the tissue. Sonicate for 30 minutes. Centrifuge at 2800 rpm for 10 minutes and collect the supernatant, adding it to the extract in the 50 mL centrifuge tube containing the extract.

6. Add 5 mL of 0.05M KOH in methanol to the remaining sample in each centrifuge tube. Vortex to disperse the tissue and hand mix briefly. Centrifuge at 2800 rpm for 10 minutes and collect the supernatant, adding it to the extract in the 50 mL centrifuge tube.

7. Using a 10 mg scoop, add 10 mg of Carbon to the combined extract, mix by occasional hand shaking over a five-minute period (not longer) and then centrifuge at ~2800 rpm for 10 minutes. Immediately decant the extract into a 60 mL glass TurboVap tube.

8. The extract is concentrated in order to remove the methanol only, not water. Add 1 mL of reagent water to each TurboVap tube and set the TurboVap to 55 ºC with a N_2 flow of 1.2 L/min to concentrate to the extract to 2.5 mL (only ~1 mL methanol should be left). Add reagent water to each TurboVap tube to dilute the extracts to 50 mL. Check that pH = 6.5 ± 0.5, if adjustment is needed use 50% formic acid or ammonium hydroxide (or with 5% formic acid and aqueous ammonium hydroxide (3%)). The extracts are ready for SPE cleanup.

9. Collect and label the required number of Waters Oasis 150 mg WAX SPE cartridges, place on the manifold and add a reservoir and adaptor to each SPE cartridge. Optional: The SPE cartridges for the entire batch may be packed with glass wool.

10. Without using vacuum, pass two full SPE cartridge volumes (~7.5 mL, each) of methanolic ammonium hydroxide (1%) followed by 5 mL of 0.3 M formic acid through each cartridge. Discard the wash solvents.

11. Pour the sample from the TurboVap tube. Don't use a pipette and take care to avoid splashing while loading. Adjust the vacuum to load the extract at 5 mL/min, retaining the emptied TurboVap tube and keeping the reservoir in place for rinsing. Discard the sample eluting from the cartridge.

12. Leaving the reservoir in place until after the sample has been eluted, using a pipette, rinse the walls of the reservoir thoroughly with wash solutions and pass washes through the cartridge using vacuum. The wash solutions are 2 x 5 mL reagent water (10 mL total), and 5 mL of 1:1 0.1M formic acid:methanol. Dry by pulling air through under vacuum for 15 seconds. The wash solutions are discarded.

13. Prepare pre-labeled collection tubes (13 x 100 mm, polypropylene) by adding an aliquot of IIS (Section 3.5.2) to each tube. Do a visual check of the tubes to confirm that IIS has been added to each one. Place the collection tubes into the manifold rack and ensure that the extract delivery needles are lined up into the collection tubes but not touching the walls of the tubes.

14. Use a total of 5 mL of elution solvent (methanolic ammonium hydroxide (1%)). Using a glass pipette, first rinse the walls of the TurboVap tube with the cartridge elution solvent, and then use the same solution and pipette to rinse the walls of the reservoir. Use vacuum to pull the elution solvent through the cartridge and into the collection tubes that have been spiked with IIS.

15. Add 25 µL of acetic acid to each sample extract. Use a 5 mL polypropylene syringe equipped with a syringe filter (25 mm filter, 0.2 µm nylon membrane) to filter a portion (~1 mL) of the extract into a microvial for LC-MS/MS analysis. Cap the collection tube containing the remaining extract and store at 4 °C as back-up.

6 CLEAN UP PROCEDURES

Clean up occurs concurrently with the extraction procedures of sections 5.1, 5.2, and 5.3

7 INSTRUMENTAL ANALYSIS

7.1 General

Analysis of sample extracts for PFAS by UPLC-MS/MS is performed on an ultrahigh performance liquid chromatograph coupled to a triple quadrupole mass spectrometer, running manufacturer's

MassLynx v.4.1 software. The mass spectrometer is run with unit mass resolution in the multiple reaction monitoring (MRM) mode. The instrument specifications are listed in Table 8 below.

Table 8. Instrumentation

Instrument	Waters Acquity UPLC, TQ-S Xevo MS/MS or TQ-S Xevo Micro MS/MS, (or equivalent)
LC column	Waters Acquity UPLC ® BEH C18 column, 1.7 µm, 50 x 2.1 mm (or equivalent)
Guard column	Guard column: Phenomenex Evo C18 Guard (or equivalent)
Acquisition	MRM mode, negative ESI, unit resolution
Injection volume	2.0 µL (Note, will vary with instrument, this volume is provided for the default method only)

The UPLC-MS/MS operating conditions are presented in Tables 9 and 10.

Table 9. Typical UPLC-MS/MS Operating Conditions

UPLC Gradient Program			**General UPLC Conditions**	
Time (min)	**Flow mixture [2]**	**Flow Rate Program**		
0.0	2% eluent A 98% eluent B	0.35 mL/min	Column Temp (°C)	40
0.2	2% eluent A 98% eluent B	0.35 mL/min	Max Pressure (bar)	1100.0
4.0	30% eluent A 70% eluent B	0.40 mL/min	Source Temp (°C)	140
7	55% eluent A 45% eluent B	0.40 mL/min	**MS/MS Conditions**	
9	75% eluent A 25% eluent B	0.40 mL/min	Desolvation Temp (°C)	500
10	95% eluent A 5% eluent B	0.40 mL/min	Capillary Voltage (kV)	0.70
10.4	2% eluent A 98% eluent B	0.40 mL/min	Cone Gas (L/hr)	~70

11.8	2% eluent A 98% eluent B	0.40 mL/min	Desolvation gas (L/hr)	~800
12.0	2% eluent A 98% eluent B	0.35 mL/min		

[1] Eluent A = Acetonitrile
Eluent B = 2 mM ammonium acetate in 95:5 water:acetonitrile

The ions monitored are presented in Table 10. Specific tuning parameters will vary with instrument and laboratory and must be developed and validated in the laboratory prior to use.

7.2 Calibration Procedures

Perform initial calibration of the UPLC-MS/MS instrument by the analysis of five or more calibration solutions. The product ion response of the primary (quantitative) and secondary (qualitative) transition must have a signal to noise (S/N) ratio of 3:1 or greater. For qualitative identification of each target analyte, the ratio of the primary product ion response (total of responses for all isomers detected for the analyte) to the secondary ion response (total of responses for all isomers detected for the analyte) is used.

To verify the initial calibration, analyze a mid-level calibration standard prepared from a second source and results must meet the method calibration verification requirements.

7.3 Sample Analysis

Samples are injected in the following order:

Instrument Blank
Instrument Sensitivity Check[1, 2]
Continuing Calibration Verification Standard[1]
Qualitative Standards
Instrument Blank
Method Blank
LOQ Verification
LCS
LCSD
Samples(10 or fewer)
Calibration Verification Standard[1]
Instrument Blank
Samples (10 or fewer)

……continued cycle

[1]If CCV concentration is at the LOQ, it can serve as the ISC as well.

[2] Instrument Sensitivity Check to be analyzed, at a minimum every 12 hours

8. QUALITATIVE AND QUANTITATIVE DETERMINATION

8.1 Peak Identification

Positive identification of target PFAS, EIS analytes, and IIS analytes require:

- ≥ 3:1 signal:noise for precursor ion to product quantitative and confirmation ion transitions.
- A qualitative standard containing all available isomers (branched and linear) is analyzed every 12 hours to confirm the retention time of each linear and known branched isomer or isomer group.
- Method analyte, EIS analyte, and IIS analyte RTs must fall within 0.4 minutes of the predicted retention times from the midpoint standard of the ICAL or initial daily CCV, whichever was used to establish the RT window position for the analytical batch. The retention time window used must be of sufficient width to detect earlier eluting branched isomers. For all method analytes with exact corresponding isotopically labeled analogs, method analytes must elute within 0.1 minutes of the associated EIS.
- For concentrations at or above the method LOQ, the total (branched and linear isomer) quantification ion response to the total (branched and linear isomer) confirmation ion response ratio must fall within ±50% of the ratio observed in the mid-point initial calibration standard The response of all isomers in the quantitative standards should be used to define ratio. In samples, the total response should include only branched isomer peaks which have been identified in either the quantitative or qualitative standard. If standards (either quantitative or qualitiative) are not available for purchase, on the linear isomer can be identified and quantitated in samples. If ratio exceeds this criteria, the peak is identified, and the analyte concentration is reported with a “Q” qualifier, indicating the sample result is potentially biased and is further discussed in the sample case narrative. The ratio requirement does not apply for PFBA, PFPeA, NMeFOSE, and NEtFOSE where suitable (not detectable or inadequate S/N) secondary transitions are unavailable.

8.2 Analyte Quantification

Target compounds are quantified using the isotope dilution/internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labeled or deuterium labeled standard and correcting for response factors. Relative response factors are determined from the initial calibration (I-CAL, see Table 6) and are confirmed at least every 12 hours (CAL/VER).

$$\text{Concentration of Target} = \frac{\text{area of target}}{\text{area of EIS}} \times \frac{\text{weight of EIS (ng)}}{\text{RRF}} \times \frac{1}{\text{weight of sample (g)}}$$

$$\text{where RRF} = \frac{\text{area of target}}{\text{area of EIS}} \times \frac{\text{Concentration of EIS}}{\text{Concentration of Target}}$$

The recovery of the EIS is calculated **(by internal standard quantification against the IIS using an average RRF)** and monitored as an indication of overall data quality. Final target concentrations are recovery corrected by this method of quantification. EIS recovery is calculated as:

$$\%\ \text{EIS recovery} = \frac{\text{area of EIS}}{\text{area of IIS}} \times \frac{\text{weight of IIS (ng)}}{\text{weight of EIS (ng)}} \times \frac{100}{\text{average RRF}_s} \quad \text{in the sample}$$

And

$$\text{RRF}_s = \frac{\text{area of EIS}}{\text{area of IIS}} \times \frac{\text{weight of IIS (ng)}}{\text{weight of EIS (ng)}} \quad \text{for each calibration level}$$

The isotopically labeled analog of an analyte (EIS) is used for quantitation (Isotope Dilution Quantitation). If a labeled analog is not commercially available, an EIS with chemical similarity and close retention time is used for quantitation (internal standard quantitation).

Table 10. Analytes, Transition Ions and Quantification References

	Typical Retention Time [1]	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass	Typical Ion Ratio Quant./Conf.[2]	Quantified using
Method Analytes						
PFBA	1.96	212.8	168.9	n.a.	n.a.	$^{13}C_4$-PFBA
PFPeA	4.18	263.0	219.0	68.9	n.a.	$^{13}C_5$-PFPeA
PFHxA	4.81	313.0	269.0	118.9	13	$^{13}C_5$-PFHxA
PFHpA	5.32	363.1	319.0	169.0	3.5	$^{13}C_4$-PFHpA
PFOA	6.16	413.0	369.0	169.0	3.0	$^{13}C_8$-PFOA
PFNA	6.99	463.0	419.0	219.0	4.9	$^{13}C_9$-PFNA
PFDA	7.47	512.9	469.0	219.0	5.5	$^{13}C_6$-PFDA
PFUnA	7.81	563.1	519.0	269.1	6.9	$^{13}C_7$-PFUnA
PFDoA	8.13	613.1	569.0	319.0	10	$^{13}C_2$-PFDoA
PFTrDA	8.53	663.0	619.0	168.9	6.7	avg. $^{13}C_2$-PFTA and $^{13}C_2$-PFDoA
PFTA	8.96	713.1	669.0	168.9	6.0	$^{13}C_2$-PFTA
PFBS	4.79	298.7	79.9	98.8	2.1	$^{13}C_3$-PFBS
PFPeS	5.38	349.1	79.9	98.9	1.8	$^{13}C_3$-PFHxS
PFHxS	6.31	398.7	98.9	79.9	1.9	$^{13}C_3$-PFHxS
PFHpS	7.11	449.0	79.9	98.8	1.7	$^{13}C_8$-PFOS

	Typical Retention Time [1]	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass	Typical Ion Ratio Quant./Conf.[2]	Quantified using
PFOS	7.59	498.9	79.9	98.8	2.3	$^{13}C_8$-PFOS
PFNS	7.92	548.8	79.9	98.8	1.9	$^{13}C_8$-PFOS
PFDS	8.28	599.0	79.9	98.8	1.9	$^{13}C_8$-PFOS
PFDoS	9.14	699.1	79.9	98.8	1.9	$^{13}C_8$-PFOS
4:2FTS	4.67	327.1	307.0	80.9	1.7	$^{13}C_2$-4:2 FTS
6:2FTS	5.81	427.1	407.0	80.9	1.9	$^{13}C_2$-6:2FTS
8:2FTS	7.28	527.1	507.0	80.8	3.0	$^{13}C_2$-8:2FTS
PFOSA	8.41	498.1	77.9	478.0	47	$^{13}C_8$-PFOSA
NMeFOSA	9.70	511.9	219.0	169.0	0.66	D_3-NMeFOSA
NEtFOSA	9.94	526.0	219.0	169.0	0.63	D_5-NEtFOSA
NMeFOSAA	7.51	570.1	419.0	483.0	2.0	D_3-NMeFOSAA
NEtFOSAA	7.65	584.2	419.1	526.0	1.2	D_5-N-EtFOSAA
NMeFOSE	9.57	616.1	58.9	n.a.	n.a.	D_7-NMeFOSE
NEtFOSE	9.85	630.0	58.9	n.a.	n.a.	D_9-NEtFOSE
HFPO-DA	4.97	284.9	168.9	184.9	1.95	$^{13}C_3$-HFPO-DA
ADONA	5.79	376.9	250.9	84.8	2.8	$^{13}C_3$-HFPO-DA
9Cl-PF3ONS	7.82	530.8	351.0	532.8 → 353.0	3.2	$^{13}C_3$-HFPO-DA

	Typical Retention Time [1]	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass	Typical Ion Ratio Quant./Conf.[2]	Quantified using
11Cl-PF3OUdS	8.62	630.9	450.9	632.9 → 452.9	3.0	$^{13}C_3$-HFPO-DA
PFEESA	5.08	315	135	82.9		$^{13}C_5$-PFPeA
PFMPA	3.21	229	84.9	n.a.		$^{13}C_5$-PFPeA
PFMBA	4.53	279	85.1	n.a.		$^{13}C_5$-PFHxA
NFDHA	4.84	295	201	84.9		$^{13}C_5$-PFHxA
3:3 FTCA	3.89	241	177	117		$^{13}C_5$-PFPeA
5:3 FTCA	5.14	341	237	217		$^{13}C_5$-PFHxA
7:3 FTCA	6.76	441	317	337		$^{13}C_5$-PFHxA
Extracted Internal Standard Analytes						
$^{13}C_4$-PFBA	1.95	216.8	171.9	n.a.		$^{13}C_3$-PFBA
$^{13}C_5$-PFPeA	4.18	268.3	223.0	n.a.		$^{13}C_2$-PFHxA
$^{13}C_5$-PFHxA	4.80	318.0	273.0	120.3		$^{13}C_2$-PFHxA
$^{13}C_4$-PFHpA	5.32	367.1	322.0	n.a.		$^{13}C_2$-PFHxA
$^{13}C_8$-PFOA	6.16	421.1	376.0	n.a.		$^{13}C_4$-PFOA
$^{13}C_9$-PFNA	6.99	472.1	427.0	n.a.		$^{13}C_5$-PFNA
$^{13}C_6$-PFDA	7.47	519.1	474.1	n.a.		$^{13}C_2$-PFDA

	Typical Retention Time [1]	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass	Typical Ion Ratio Quant./Conf.[2]	Quantified using
$^{13}C_7$-PFUnA	7.81	570.0	525.1	n.a.		$^{13}C_2$-PFDA
$^{13}C_2$-PFDoA	8.13	615.1	570.0	n.a.		$^{13}C_2$-PFDA
$^{13}C_2$-PFTA	8.96	715.17	670.0	n.a.		$^{13}C_2$-PFDA
$^{13}C_3$-PFBS	4.78	302.1	79.9	98.9		$^{18}O_2$-PFHxS
$^{13}C_3$-PFHxS	6.30	402.1	79.9	98.8		$^{18}O_2$-PFHxS
$^{13}C_8$-PFOS	7.59	507.1	98.9	79.9		$^{13}C_4$-PFOS
$^{13}C_2$-4:2FTS	4.67	329.1	80.9	309.0		$^{18}O_2$-PFHxS
$^{13}C_2$-6:2FTS	5.82	429.1	80.9	409.0		$^{18}O_2$-PFHxS
$^{13}C_2$-8:2FTS	7.28	529.1	80.9	509.0		$^{18}O_2$-PFHxS
$^{13}C_8$-PFOSA	8.41	506.1	77.8	n.a.		$^{13}C_4$-PFOS
D_3-NMeFOSA	9.70	515.0	219.0	n.a.		$^{13}C_4$-PFOS
D_5-NEtFOSA	9.94	531.1	219.0	n.a.		$^{13}C_4$-PFOS
D_3-NMeFOSAA	7.51	573.2	419.0	n.a.		$^{13}C_4$-PFOS
D_5-NEtFOSAA	7.65	589.2	419.0	n.a.		$^{13}C_4$-PFOS
D_7-NMeFOSE	9.56	623.2	58.9	n.a.		$^{13}C_4$-PFOS
D_9-NEtFOSE	9.83	639.2	58.9	n.a.		$^{13}C_4$-PFOS
$^{13}C_3$-HFPO-DA	4.97	284.9	168.9	184.9		$^{13}C_2$-PFHxA

	Typical Retention Time [1]	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass	Typical Ion Ratio Quant./Conf.[2]	Quantified using
Injection Internal Standard Analytes						
$^{13}C_3$-PFBA	1.95	216.0	172.0	n.a.		External
$^{13}C_2$-PFHxA	4.80	315.1	270.0	119.4		External
$^{13}C_4$-PFOA	6.16	417.1	172.0	n.a.		External
$^{13}C_5$-PFNA	6.99	468.0	423.0	n.a.		External
$^{13}C_2$-PFDA	7.47	515.1	470.1	n.a.		External
$^{18}O_2$-PFHxS	6.30	403.0	83.9	n.a.		External
$^{13}C_4$-PFOS	7.59	502.8	79.9	98.9		External

[1]Times are shown are in decimal minute units.

[2]Transistion s may vary by instrument, values shown are examples. Ion ratios applicable to qualitative identification are determined from instrumental calibration data.

9.0 REPORTING CRITERIA AND PRACTICES

Results are reported for concentrations quantitated at greater that the detection limit.

Data is qualified using the following flag scheme:

U – Analyte was not detected and is reported as less than the MDL.
J – The reported result is an estimated value (for example, below the LOQ)
B – Blank contamination. The recorded result is associated with a contaminated blank (i.e. a blank with concentrations exceeding the stated criteria)
Q – Associated quality control exceeds method criteria (e.g., sample ion ratio)
E – Reported result exceeds upper limit of the calibration range (required further dilution)
D – Result reported from dilution

Figure 1. PFAS in Whole Water - Summary Flowchart

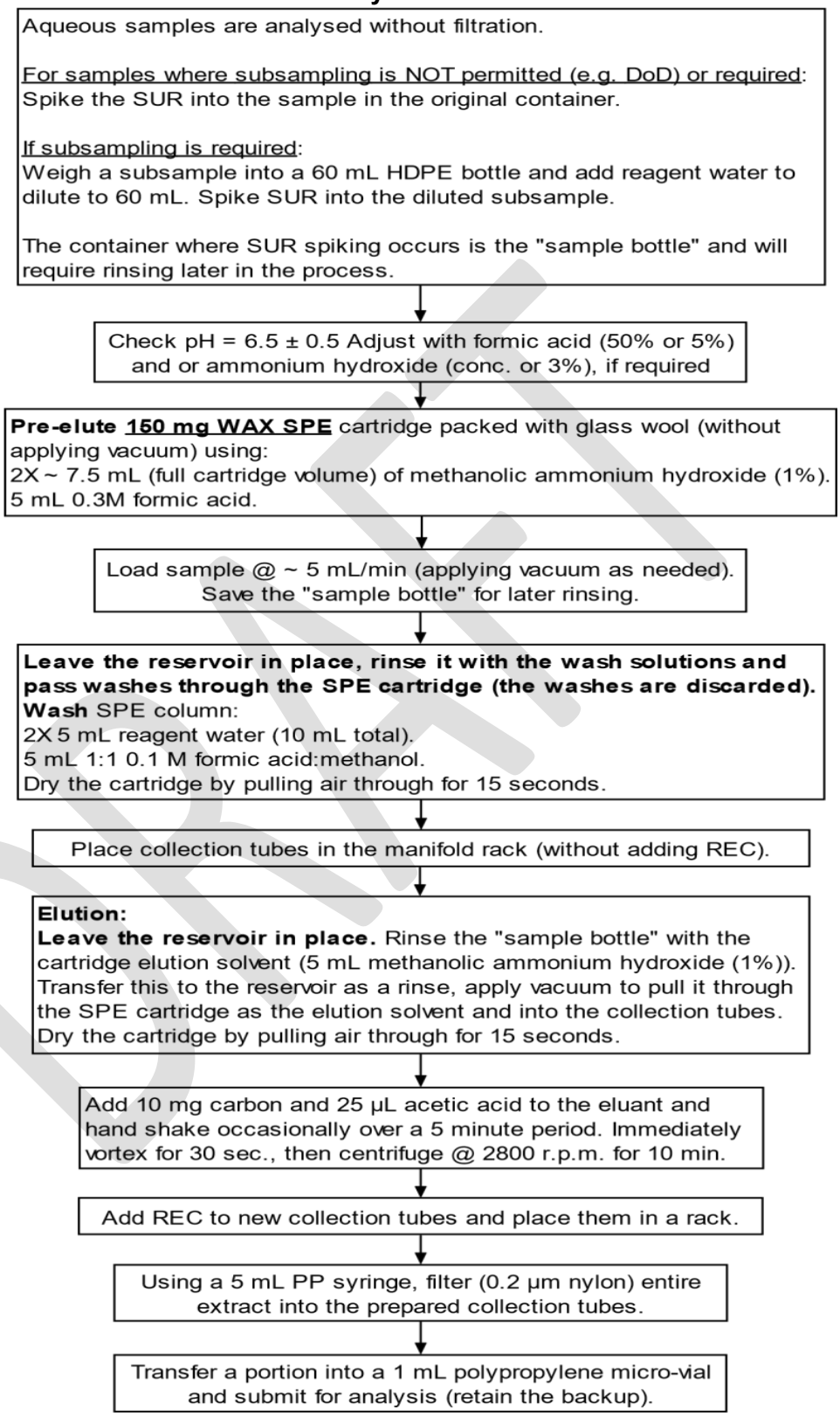
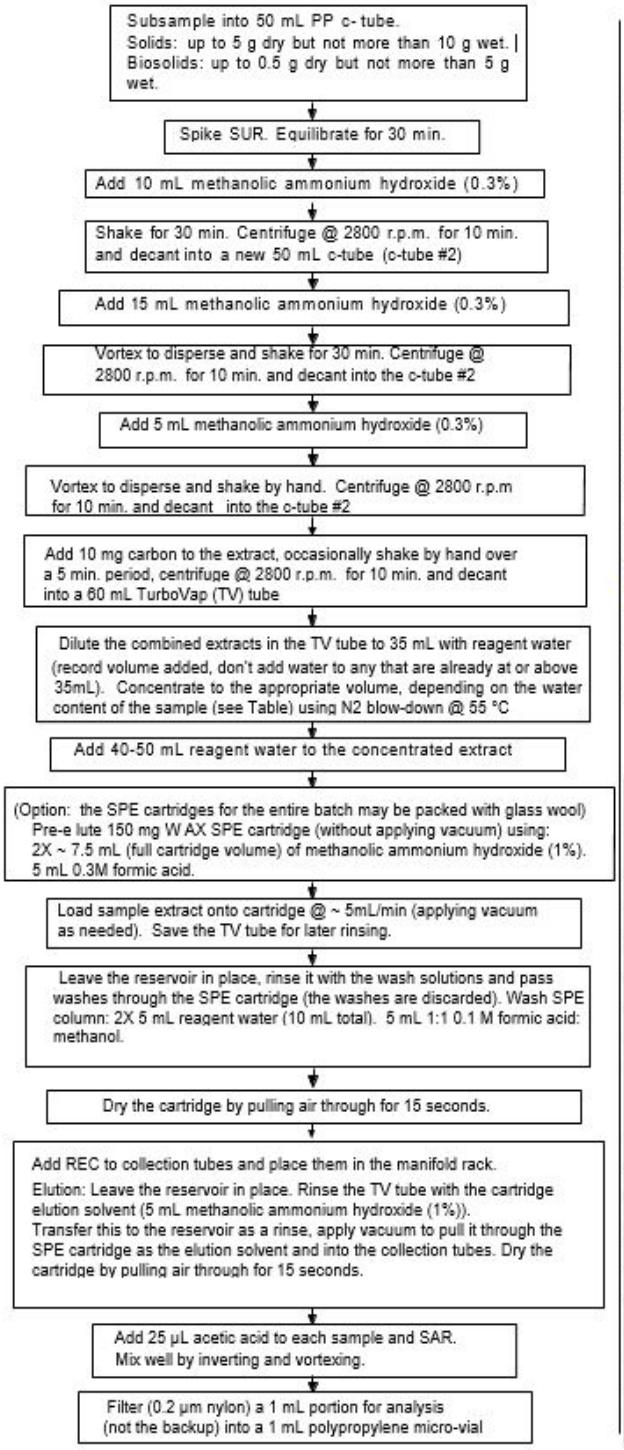


Figure 2. PFAS in Solid Samples - Summary Flowchart



PFAS Solids TurboVap use guidelines and Table of sample size, moisture content and corresponding final TurboVap volumes

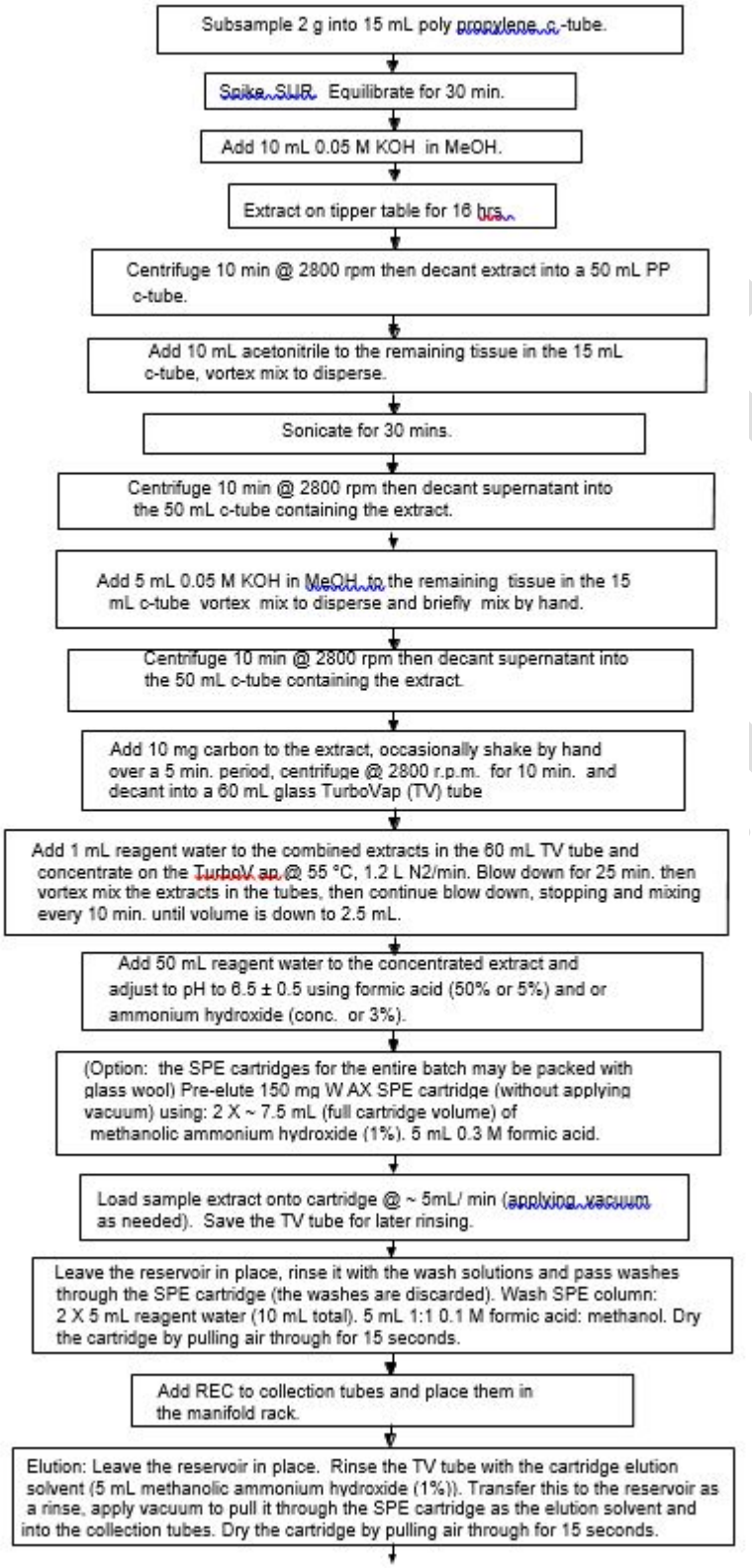
PFAS Solids extracts should be blow down for 25 minutes, then mixed (by vortex if volume is < 20 mL or using a glass pipette if volume is > 20 mL) then continue blow down, stopping and mixing every 10 min. until volume is down to the volume in the table below, or IF VOLUME IS NO LONGER REDUCING, STOP THE BLOW DOWN AND NOTE THE VOLUME.

* This table requires the sample size and % Moisture to be known before blow down can be started.
* Any spills must be recorded and corrected for in determining the blow down volume.
* The sample extract will contain the water from the sample & any water added to bring the volume to 35 mL.
* Record the volume of water added to each sample extract.
* The blow down is done to leave a few mL of water and a minimal amount of methanol (up to 5 mL in some sample extracts), but not to blow down lower than the total amount of water present before blow down.
* Do not blow down lower than the amount of water that was added

DO NOT LEAVE ON BLOW DOWN LONGER THAN NEEDED ONCE THE CORRECT VOLUME HAS BEEN REACHED. IF VOLUME IS NO LONGER REDUCING, STOP THE BLOW DOWN AND NOTE THE VOLUME.

Sample size (g)	% Moisture	Volume of water in sample (mL) {= Sample size x % Moisture/100}	Final TurboVap volume* (mL)
<5	0-100	<5	7
5	0	0	7
5	30	1.5	7
5	50	2.5	7
5	70	4	7
5	90	4.5	7
5	100	5	7
7.5	35 (e.g. BLK, OPR)	2.5	7
7.5	65	5	7
7.5	70	5.3	8
7.5	90	6.8	8
10	50	5	7
10	55	5.5	8
10	60	6	8
10	80	8	8
10	85	8.5	9
10	90	9	9
10	>90	9-10	10
>10	Custom	procedure	reqd.

Figure 3. PFAS in Tissue Samples – Summary Flowchart



PFAS in Tissue Samples – Summary Flowchart (cont.)

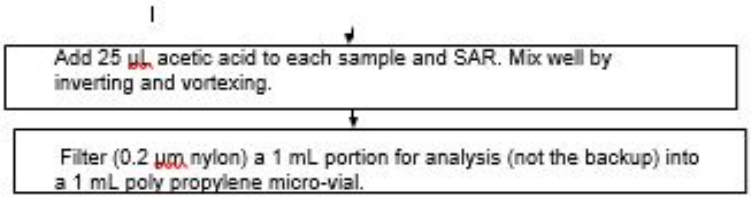


Figure 4. Appearance of a 100 mL aqueous sample containing 100, 50, 30 and 10 mg of solids from left to right, respectively.

Figure 5. Appearance of a 500 mL aqueous sample containing 100, 50, 30 and 10 mg of solids from left to right, respectively.

Figure 6. Appearance of 150 mg WAX SPE cartridges packed with glass wool to half the height of the cartridge barrel. Picture taken after passing aqueous samples containing 10, 30, 50 and 100 mg in triplicate from left to right, respectively.

This page was intentionally left blank.

ATTACHMENT 2

STUDY SAMPLE MATRIX SUMMARY

Attachment 2
Study Sample Matrix Summary
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

Project Phase	Sample Matrix Type	Sample Matrix Designation	Sample Matrix Description	SGS AXYS ID	Point of Contact	Shipping Status	PFAS Pre-Screened (yes/no)?	Sample Size to be Prepared by Lab	# of Blank Samples (unspiked)	# of Low Level Spiked Samples	# of Mid-Level Spiked Samples	# of HIgh-Level Spiked Samples	Used in Holding Time Study
Phase 2	*Groundwater*	A	Midwest region groundwater (Colorado Site)	L32934-1 and 2	Chris Bellona, CSM (cbellona@mines.edu)	Received by Lab 15-APR-20	Yes	500 mL	3	3	3	3	No
		B	Upper Midwest region groundwater (Wurtsmith AFB)	L32934-3	Chris Bellona, CSM (cbellona@mines.edu)	Received by Lab 15-APR-20	Yes	500 mL	3	3	3	3	No
		C	Arid region groundwater (WL378) (Altus AFB)	L32644-1	Hunter Anderson, Airforce (richard.anderson55@us.af.mil)	Received by Lab 4-FEB-20	No (assumed ND)	500 mL 500 mL	3 15	3	3 30	3	Yes
	Surface Water	D	Freshwater River (Willow Grove COP-SW01 to 11)	L32907-1 to -11	Chuck Coyle (Charles.G.Coyle@usace.army.mil)	Received by Lab	Yes	500 mL	3	3	3	3	No
		E	Freshwater River (Willow Grove RP-SW01 to 11)	L32907-23 to 33	Chuck Coyle (Charles.G.Coyle@usace.army.mil)		Yes	500 mL	3	3	3	3	No
		F	Marine water	L32942-1	SGS Axys sample		Assumed ND	500 mL	3	3	3	3	No
	Landfill Leachate	G	Municipal solid waste landfill leachate	L32688-1 to-3	Timothy Townsend (ttown@ufl.edu)	Received by Lab	Yes	500 mL	3	3	3	3	No
		H	Construction and Debris landfill leachate	L32708-2	Timothy Townsend (ttown@ufl.edu)		Yes	500 mL	3	3	3	3	No
		I	MSW incineration ash landfill	L32708-1	Marc Mills, EPA mills.marc@epa.gov)		Yes	500 mL	3	3	3	3	No
	Wastewater	J	WW #3 Metal finisher	L32525-2	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No	500 ml	3	3	3	3	No
		K	WW #5 Hospital	L32525-4	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	500 mL	3	3	3	3	No
		L	WW #6 POTW Influent	L32525-5	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	500 mL	3	3	3	3	No
		M	WW#7 Bus washing station	L32525-6	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	250 ml	3	3	3	3	No
		N	WW#8 Power Plant	L32525-7	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	500 mL	3	3	3	3	No
		O	WW #9 Pulp and paper effluent	L32525-9	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	500 mL	3	3	3	3	No
		P	WW# 10 POTW Effluent	L32525-8	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	500 mL	3	3	3	3	No

See notes end of table

Attachment 2
Study Sample Matrix Summary
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS

Project Phase	Sample Matrix Type	Sample Matrix Designation	Sample Matrix Description		Point of Contact	Shipping Status	PFAS Pre-Screened (yes/no)?	Standard Sample Size to be Prepared by Lab (g wet/ sample)	# of Blank Samples (unspiked)	# of Low Level Spiked Samples	# of Mid-Level Spiked Samples	# of HIgh-Level Spiked Samples	Used in Holding Time Study
Phase 3	Sediment	Q	Marine sediment	L32918-1	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No	10 g	3	3	3	3	No
		R	Sediment #3 Great Lakes, freshwater low TOC sediment	L32526-2	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No	10 g	3	3	3	3	No
		S	Sediment #2 Freshwater high TOC sediment Raisin River	L32526-1	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No	10 g 10 g	3 12	3	3 24	3	Yes
	Tissue	T	Fish Tissue #2 low lipids tissue - 1.2% lipids	L32527-2	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No	2 g	3	3	3	3	No
		U	Fish Tissue #1 high lipids tissue 4% lipids	L32527-1	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	2 g	3	3	3	3	No
		V	Shellfish tissue - clams	L32492-1	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	2 g	3	3	3	3	No
	Biosolid	W	Biosolid #1	L32524-1	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No	2.5 g	3	3	3	3	No
		X	Biosolid #2	L32524-2	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	2.5 g	3 12	3	3 24	3	Yes
		Y	Biosolid very dry pelletized biosolid product	L32524-3	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	2.5 g	3	3	3	3	No
	Soil	Z	Dehli, 2nd Quarter 2014 Fresno County CA	L32547-1	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No	10 g 10g	3 12	3	3 24	3	Yes
		AA	Musselshell, 2nd Quarter 2016, Clark County MT	L32547-2	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	10 g	3	3	3	3	No
		BB	Ivy, 3rd Quarter 2017, Cashe County UT	L32547-3	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	10 g	3	3	3	3	No
		CC	Fruitland, 1st Quarter 2018, San Juan County NM	L32547-4	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	10 g	3	3	3	3	No
		DD	Armijo 4th, Quarter 2018, Dona Ana County NM	L32547-5	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	10 g	3	3	3	3	No
		EE	Drummer, 2nd Quarter 2019, Dekalb County IL	L32547-6	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	10 g	3	3	3	3	No
		FF	Brock, 2nd Quarter 2019, Wheatley County TN	L32547-7	Adrian Hanley, EPA OW (hanley.adrian@epa.gov)		No	10 g	3	3	3	3	No
Additional Matrices	Leachate		WW #2 Wastewater Leachate		Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No						
	Wastewater		WW # 10 POTW Effluent		Adrian Hanley, EPA OW (hanley.adrian@epa.gov)	Received by Lab	No						

Notes:
Some sample matrices listed in this table are subject to availability, and alternate sample matrices may be selected as they become available. This table will be updated as sample matrices are obtained.

This page was intentionally left blank.

ATTACHMENT 3

EDD INSTRUCTIONS

Attachment 3
EDD Instructions
Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC MS/MS

Following is the description of data fields requested for electronic data deliverables (EDDs) for the PFAS Single Laboratory Validation Study. The format of data in each field is indicated in brackets (e.g. [text string string]). A sample EDD is provided after the data field description.

Data Fields:

1) **Lab_ID**: [text string] Laboratory Name
2) **Sample_No**: [text string] These are the sample identification names (IDs) from the Chain of Custody, Sample Matrix names from the Sample Matrix Summary table or other experimental identifier. Quality control (QC) samples and matrices can be identified using naming and abbreviation conventions typically used by the laboratory.
3) **Lab_Sample_ID**: [text string] The ID the laboratory assigns to the sample (which identifies the sample on the associated data files and reports).
4) **Analysis_Date**: [short date] Use format mm/dd/yyyy (e.g. 11/20/2019) – do not include time stamp.
5) **Analysis**: [text string] fill in "PFAS"
6) **Compound**: [text string] Use the names included in the example EDD. DO NOT CHANGE. Analytes not listed can be named according to typical laboratory conventions.
7) **CAS_No**: [text string] Use the Chemical Abstract Service Registration Number (CASRN) included in the example EDD. DO NOT CHANGE. For compounds with no CASRN, leave blank.
8) **PFAS_Acronym**: [text string] Use acronyms included in the example EDD. For compounds with no acronym, leave entry blank.
9) **Dilutions**: [number integer] Dilution made post extraction (e.g., extract diluted 1:10 is entered as "10"). If analyzed without dilution, enter "1".
10) **Lab_Flag**: [text string]

    "U" for Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

    "J" for the reported result is an estimated value (e.g., matrix interference was observed, or the analyte was detected at a concentration below the calibration range).

    "B" for Blank contamination. The recorded result is associated with a contaminated blank.

    'Q' when one or more quality control criteria failed (e.g., Ion ratio, LCS recovery, EIS spike recovery, or CCV recovery).

    "E" when the reported result exceeds the upper limit of the calibration range.

    "D" when the reported result is from a dilution.

If you have multiple flags assigned to a result, do not include any spacing between the flags.

11) **Conce_Found**: [number, double] Enter numeric quantitative result value only. Do NOT enter any text string strings or symbols (e.g., "ND", "<"). For analytes that are not detected, the laboratory's LOD for that analyte is entered and the "U" flag is entered in the Lab Flag column. All results are anticipated to be reported in parts per trillion or nanograms per liter [ng/L] for aqueous samples and parts per billion or micrograms per kilogram [µg/kg] for solid and tissue samples.

12) **Conc Spiked**: [number, double] For unspiked samples enter "0" for method analytes. For spiked samples, enter the spike concentration representing the estimated concentration in the final extract in ppt. For EIS and IIS, enter the spike concentration representing the concentration in the final extract in ppb.

13) **Percent Rec**: [text string] For unspiked samples, enter "NA" for method analytes. For spiked samples enter the spike percentage recovery as a whole number (e.g., 95 versus 0.95). Do NOT include "%" symbol. For EIS and IIS, enter the spike % recovery as a whole number (e.g., 95 versus 0.95). Do NOT include "%".

14) **MDL** (Method Detection Limit): [number, double] Enter the sample specific MDL (i.e. with extract dilution factor, sample volume/weight and final volume taken into account) in ppt.

15) **LOD** (Limit Of Detection): [number, double] Enter the sample specific LOD (i.e. with extract dilution factor, sample volume/weight and final volume taken into account) in ppt.

16) **LOQ (**Limit of Quantitation): [number, double] Enter the sample specific LOQ (i.e. with extract dilution factor, sample volume/weight and final volume taken into account) in ppt.

17) **Unit**: the reporting units for this project are parts per trillion (ppt) or nanograms per liter (ng/L) for aqueous samples and parts per billion micrograms per kilogram (µg/kg) for solid samples.

18) **Transition_Ratio**: [text string] Enter Transition Ratio (Quant Ion Area/Conf Ion Area)

19) **RRT**: [text string] Enter relative retention time

20) **Sample_Size**: [number, double] Enter volume (aqueous samples) or weight (solid samples) of sample extracted

21) **Sample_size_units:** [text string] Will be liters (L) for aqueous samples or kilograms (Kg) for solid samples

22) **Extract_final_vol:** [number, double] Enter in milliliters (mL)

23) **Extract_units**: [text string] Always "mL"

24) **Extraction_date**: [short date] Use format mm/dd/yyyy (e.g. 11/20/2019) – do not include time stamp.

25) **Received_date**: [short date] Date sample matrix received from Method Development Team. Use format mm/dd/yyyy (e.g. 11/20/2019) – do not include time stamp.

26) **Spike_date**: [short date] Date sample matrix spiked. Use format mm/dd/yyyy (e.g. 11/20/2019) – do not include time stamp. Blank entry for unspiked controls.

27) **Matrix**: [text string]

Sample Matrices:

- **GW** = Groundwater;
- **SW** = Surface Water;
- **SD** = Sediment;
- **SS** = Soil;
- **TS** = Tissue;
- **WW** = Wastewater;
- **LC** = Leachate;
- **BS** = Biosolids

Blank Reference Matrices:

- **RW** = Reagent water
- **OS** = Ottawa sand
- **VO** = Vegetable oil

Quality Control Samples can be labeled as is typically done by the laboratory.

28) **Method**: [text string] Laboratory SOP Name in format of "name(space)revision number"

29) **Study_Phase** – [text string]

Phase I for Initial Demonstration of Capabilities (IDC);

Phase II to include WW, LC, GW, and SW matrices;

Phase III – biosolids, fish tissue and soil

HTS – holding time study

30) QC_Sample_Type [text string]

This page was intentionally left blank.

ATTACHMENT 4

EDD FORMAT

(Electronic)

This page was intentionally left blank.

Appendix B

PFAS SLVS Data Compilation (Institute for Defense Analyses Report)

INSTITUTE FOR DEFENSE ANALYSES

Data Compilation in Support of Single Laboratory Validation of a Novel Per- and Polyfluoroalkyl Substances (PFAS) Detection Method for Environmental Matrices

Allyson M. Buytendyk
Sara C. Runkel
Shelley M. Cazares

August 2021

IDA Document D-22794
Log: H 21-000317

INSTITUTE FOR DEFENSE ANALYSES
4850 Mark Center Drive
Alexandria, Virginia 22311-1882

The Institute for Defense Analyses is a nonprofit corporation that operates three Federally Funded Research and Development Centers. Its mission is to answer the most challenging U.S. security and science policy questions with objective analysis, leveraging extraordinary scientific, technical, and analytic expertise.

About This Publication

This work was conducted by the Institute for Defense Analyses under contract HQ0034-19-D-0001, Project AM-2-1528, "Validation of PFAS Detection Methods," for the Executive Director, Strategic Environmental Research and Development Program (SERPD) and Environmental Security Technology Certification Program (ESTCP), under the Office of the Deputy Under Secretary of Defense (Installations and Environment). The views, opinions, and findings should not be construed as representing the official position of either the Department of Defense or the sponsoring organization.

For More Information

Shelley M. Cazares, Project Leader
scazares@ida.org, (703) 845-6792

Leonard J. Buckley, Director, Science and Technology Division
lbuckley@ida.org, (703) 578-2800

Copyright Notice

INSTITUTE FOR DEFENSE ANALYSES

IDA Document D-22794

Data Compilation in Support of Single Laboratory Validation of a Novel Per- and Polyfluoroalkyl Substances (PFAS) Detection Method for Environmental Matrices

Allyson M. Buytendyk
Sara C. Runkel
Shelley M. Cazares

Executive Summary

The Strategic Environmental Research and Development Program (SERDP) and the Environmental Security and Technology Certification Program (ESTCP) with the Environmental Protection Agency Office of Water, Engineering and Analysis Division developed a novel method for measuring trace contamination of per- and polyfluoroalkyl substances (PFAS), a broad category of man-made chemicals that are environmentally persistent and associated with health problems in humans. Aqueous film-forming foam (AFFF), which contain mixtures of PFAS, have been used widely by the Department of Defense (DoD) to extinguish high-hazard liquid hydrocarbon fires. The use of AFFF has resulted in the widespread occurrence of PFAS in groundwater, drinking water, soils, and sediments at many current and former military installations. The DoD has environmental management responsibilities for the release of PFAS into the environment associated with the use of AFFF. SERDP/ESTCP is sponsoring the validation of the novel PFAS measurement method for complex water matrices (e.g., wastewater, surface water, groundwater, landfill leachate), solids (e.g., soil, sediment, biosolids) and tissues. The first step in the validation process is a single laboratory validation (SLV) study.

SERDP/ESTCP asked IDA to calculate summary statistics from the data generated in the SLV study and systematically compile the statistics and data into specified tables to support subsequent analysis by Naval Sea Systems Command, the Air Force Civil Engineer Center, and SERDP/ESTCP itself. IDA automatically generated data tables in a systematic and reproducible fashion using a coded Python computer script to eliminate human error. The summary statistics (means and standard deviations) included native concentrations, spike concentrations, and percent recoveries of the PFAS analytes in aqueous and solid-type matrices and tissues. IDA performed a rigorous quality check on the summary statistics to be confident they were calculated correctly. A digital appendix with all data tables accompanies this document with the full set of tables. These data can now be used to support the government's SLV of the novel PFAS method.

Contents

1. Introduction

Per- and polyfluoroalkyl substances (PFAS) are a group of man-made chemicals that are resistant to heat, oil and water. The properties of PFAS can be attributed to short, strong bonds between carbon and fluorine atoms, but a consequence for such durability is the chemical structure does not easily break down in the environment.[1] In recent years, scientists have nicknamed PFAS "forever chemicals" because of their rise as a persistent and mobile pollutant. Some familiar consumer product brands that contain PFAS include Teflon (i.e., non-stick cookware) and Scotchgard (i.e., stain/water repellants for fabrics).[2] PFAS have been used in numerous industrial applications but most notably is the aqueous film-forming foam (AFFF) that were adopted widely across the Department of Defense (DoD) on ships and military bases to extinguish high-hazard liquid hydrocarbon fires.[3] The use of AFFF has resulted in the prevalence of PFAS in groundwater, drinking water, soils, and sediments at many current and former military installations.[4]

PFAS are present in drinking water systems and groundwater supplies around DoD military installations at levels above the Environmental Protection Agency (EPA) Lifetime Health Advisories of 70 parts per trillion (individually or combined).[5] The EPA published the Lifetime Health Advisories and Health Effects Support Documents notice for Perfluorooctanoic Acid (PFOA) and perfluorooctane sulfonate (PFOS) in 2016.[6] Concern

---

1 Reddy, Prakash. (2015), *Organofluorine Compounds in Biology and Medicine*, Elsvier, accessed September 16, 2021, https://www.sciencedirect.com/book/9780444537485/organofluorine-compounds-in-biology-and-medicine.

2 United States Environmental Protection Agency, "Basic Information on PFAS," accessed August 3, 2021, https://www.epa.gov/pfas/basic-information-pfas.

3 Strategic Environmental Research and Development Program (SERDP) and the Environmental Security Technology Certification Program (ESTCP), accessed August 3, 2021, https://www.serdp-estcp.org/Featured-Initiatives/Per-and-Polyfluoroalkyl-Substances-PFASs.

4 Leeson, A. et al. (2020),"Identifying and Managing Aqueous Film-Forming Foam-Derived Per- and Polyfluoroalkyl Substances in the Environment," Environ. Toxicol. Chem. 40, 24-36.

5 M. Sullivan, "Addressing Perfluorooctane Sulfonate (PFOS)and Perfluorooctanoic Acid (PFOA)," DoD, 2018, accessed August 11, 2021, https://www.epa.gov/sites/default/files/2018-05/documents/dod_presentation_epa_summit_pfos_pfoa_may2018_final.pptxx_.pdf.

6 Environmental Protection Agency, Lifetime Health Advisories and Health Effects Support Documents for Perfluorooctanoic Acid and Perfluorooctane Sulfonate. *Federal Register.* 18, 33250-33251, accessed August 26, 2021, https://www.federalregister.gov/documents/2016/05/25/2016-12361/lifetime-health-advisories-and-health-effects-support-documents-for-perfluorooctanoic-acid-and.

about the toxicity of PFAS and their impact on human health continues to grow. Researchers have observed noncancer effects following repeated oral exposures to PFAS, including abnormal thyroid and kidney development in animal studies. The number of human studies is too small to draw direct conclusions between PFAS exposure and health outcomes. In 2021, the EPA conducted a toxicity assessment for a specific PFAS—perfluorobutane sulfonic acid (PFBS) and its related ionic compound—to evaluate the human health hazard. After a rigorous peer review by external scientists, the EPA published human oral reference doses derived from animal models for various noncancer effects.[7] To understand the degree of a person's exposure to PFAS, scientists need to measure more of the possible thousands of PFAS in all facets of the environment (air, soil, water, tissue).

The DoD has environmental management responsibilities for the release of PFAS into the environment associated with the use of AFFF. The Strategic Environmental Research and Development Program (SERDP) and the Environmental Security and Technology Certification Program (ESTCP) with the EPA Office of Water, Engineering and Analysis Division developed a novel method for measuring trace contamination of PFAS using isotope-dilution liquid-chromatography mass spectroscopy/mass spectroscopy (LC-MS/MS).[8] SERDP/ESTCP is sponsoring the validation of the novel PFAS measurement method for complex water matrices (e.g., wastewater, surface water, groundwater, landfill leachate), solids (e.g., soil, sediment, biosolids) and tissues.

In pursuit of this effort, SERDP/ESTCP conducted a single-laboratory validation (SLV) study of the new method.[9] The SLV allows the opportunity to make any necessary adjustments to the method before a larger multi-laboratory validation effort. SERDP/ESTCP sponsored IDA to calculate summary statistics from the data collected in the SLV study and systematically compile the statistics and data into specified tables to support the SLV study. The compiled data tables will inform the government team, consisting of SERDP/ESTCP, Naval Sea Systems Command (NAVSEA), and Air Force Civil Engineer Center, as it completes the SLV, which will be documented in a later government report that will be submitted to the EPA.

---

7 EPA, *Human Health Toxicity Values for Perfluorobutane Sulfonic Acid* (CASRN 375-73-5) *and Related Compound Potassium Perfluorobutane Sulfonate* (CASRN 29420-49-3), EPA/600/R-20/345F, U.S. EPA Office of Research and Development Center for Public Health and Environmental Assessment, April 2021, accessed August 11, 2021, https://ofmpub.epa.gov/eims/eimscomm.getfile?p_download_id=542393.

8 EPA, *Draft Method 1633 Analysis of Per- and Polyfluoroalkyl (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS*, August 2021, accessed September 16, 2021, https://www.epa.gov/system/files/documents/2021-09/method_1633_draft_aug-2021.pdf

9 SERDP/ESTCP PFAS Method Validation Team, "Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS," April 2020.

The purpose of this short turnaround IDA project was to compile the data reported by the laboratory in a systematic, reproducible mode that the government team could then analyze to perform the SLV. This report documents how we compiled the data tables (including calculating summary statistics like means and standard deviations), presents examples for each set of tables generated, and discusses some high-level observations about the dataset. A digital appendix accompanies this paper with the full set of tables.

2. Method

SERDP/ESTCP provided the data collected in the SLV study. The single laboratory[10] in the study produced electronic data delivery (EDDs) files that report the results for 64 PFAS analytes from 32 environmental matrix samples measured using the experimental method. NAVSEA then reviewed the EDDs for data validation. The EDDs report values for

1. the native analyte concentration (the value of PFAS analyte native to the environmental sample measured with the method);
2. the spike analyte concentration, which is a known physical amount (mass per unit volume = concentration), or "spike," of PFAS analyte the laboratory *added* to the environmental sample);
3. the measured spike analyte concentration (the value of the spike analyte concentration measured with the method); and
4. the percent recoveries of the spiked analyte (the measured value of the spike analyte concentration measured with the method relative to the known physical amount that the laboratory added to the environmental sample; see Equation 1)

for over 30,000 subsamples. The laboratory created subsamples from the environmental matrix samples to make duplicate native and spike concentration measurements as outlined in the SLV study procedure.[11]

SERDP/ESTCP also provided direction on what specific calculations and tables to generate from the EDDs, including the table templates with specific rows and columns to populate with values. We used Python to read in the sponsor-provided EDDs; process the data to calculate percent recoveries, means, mean of means, standard deviations (SD), and percent relative standard deviations (RSD); and output the sponsor-requested tables in CSV format, using a coded script. Upon a request from SERDP/ESTCP, we reported all means for PFAS concentrations with three significant figures and all percent recoveries, means, SD (i.e. 2x SD, etc.), and percent RSD to two places after the decimal. These metrics were defined in the SLV study plan.

---

[10] SGS AXYS Analytical Services Ltd. Sidney, British Columbia, Canada

[11] SERDP/ESTCP PFAS Method Validation Team, Attachment 1 in "Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS," April 2020.

We used the equations that follow in the sponsor-requested calculations.

Equation 1: Percent recovery

$$\left(\frac{\textbf{Measured spike [ ]} - \textbf{Meausured native [ ]}}{\textbf{Known spike [ ] added}}\right) \cdot \mathbf{100};$$

$$\textbf{where [ ] = concentration}$$

Equation 1 describes how percent recovery is calculated based on the measured native concentration, and the measured and known analyte spike concentrations of a particular PFAS analyte in an environmental matrix.

Equation 2: Mean

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i ;$$

$$\textbf{where } n = \textbf{number of quantities}, x_i = i\textbf{th quantity value}$$

Equation 2 describes how the mean of a type of quantity was calculated. That quantity could be a native concentration, a known spike concentration, a measured spike concentration, or a percent recovery for a PFAS analyte.

Equation 3: Mean of means

$$\mu_G = \frac{1}{G}\sum_{g=1}^{G} \bar{x}_g;$$

$$\textbf{where } G = \textbf{number of groups}, \bar{x}_g = g\textbf{th mean}$$

Equation 3 describes how the mean was calculated from a group of means for a given quantity. For example, Equation 2 could be used to calculate the mean percent recovery of a PFAS analyte in a particular groundwater matrix, and then Equation 3 could be used to calculate the mean of the mean percent recoveries of *all* groundwater matrices. In another example, Equation 2 could be used to calculate the mean measured spike concentration of a PFAS analyte in a particular soil matrix, and then Equation 3 could be used to calculate the mean of the mean measured spike concentration of *all* soil matrices.

SERDP/ESTCP instructed us to use the mean of means as written in Equations 3 since that is how the EPA calculated the mean from a group of means in a previous method validation study. We recognize there is a risk of bias toward lower sample sizes in the calculation.

Equation 4: Standard deviation (SD)

$$\frac{\sqrt{\sum(x_i-\mu)^2}}{n-1};$$

$$\textbf{where } n = \textbf{number of quantities}, x_i = \textbf{ith quantity}, \mu = \textbf{sample mean}$$

Equation 4 describes how we calculated the standard deviation for a given quantity. That quantity could be a native concentration, a known spike concentration, a measured spike concentration, or a percent recovery for a PFAS analyte.

Equation 5: % Relative standard deviation (RSD)

$$\left(\frac{SD}{\text{Mean}}\right) \cdot 100$$

Equation 5 describes how we calculated the relative standard deviation for a given quantity. For example, Equation 2 and Equation 4 could be used to calculate the mean and standard deviation of the percent recovery of a PFAS analyte in a particular groundwater matrix, and then Equation 5 could be used to calculate the relative standard deviation for the PFAS analyte in that particular groundwater matrix using the values from the mean in Equation 2 and the standard deviation in Equation 4.

We verified the calculation in every column in each of the tables generated by the coded script by comparing those values to values manually calculated using Excel. The data in the EDDs were used in the manual calculations based on the equations outlined in this section. At least one analyte (i.e., across a row in the table) was checked for each table.

3. Results

We generated more than 100 tables grouped into categories at the request of the sponsor. This section describes the calculations we performed and provides an example of the tables produced in each category. The eight table categories were as follows:

- Sample Native Concentration Tables
- Sample Matrix Recovery Tables
- Media Type Matrix Recovery Tables
- Extracted Internal Standard (EIS) Spike Recovery Tables
- Injected Internal Standard (IIS) Spike Recovery Tables
- Ongoing Precision and Recovery (OPR) Spike Recovery Tables
- Limit of Quantitation Verification (LOQVER) Spike Recovery Tables
- Method Blank (MB) Spike Recovery Tables

All tables produced are part of the digital appendix that accompanies this paper.

A. PFAS Analytes and Compounds

The SLV study comprises of 40 PFAS analytes and 24 isotopically labeled internal standard compounds. Table 1 is a list of the chemical names of the PFAS analyte acronyms that were listed in the EDDs and their chemical abstract service (CAS) registry numbers. This table is file A-1 SLV PFAS Analytes.csv from section A of the digital appendix.

Table 1. SLV PFAS Acronyms and Chemical Names

Group	Acronym	Chemical Name	CAS
Analytes	PFBA	Perfluorobutanoic acid	375-22-4
	PFPeA	Perfluoropentanoic acid	2706-90-3
	PFHxA	Perfluorohexanoic acid	307-24-4
	PFHpA	Perfluoroheptanoic acid	375-85-9
	PFOA	Perfluorooctanoic acid	335-67-1
	PFNA	Perfluorononanoic acid	375-95-1
	PFDA	Perfluorodecanoic acid	335-76-2
	PFUnA	Perfluoroundecanoic acid	2058-94-8
	PFDoA	Perfluorododecanoic acid	307-55-1

Group	Acronym	Chemical Name	CAS
Analytes	PFTrDA	Perfluorotridecanoic acid	72629-94-8
	PFTA	Perfluorotetradecanoic acid	376-06-7
	PFBS	Perfluorobutanesulfonic acid	375-73-5
	PFPeS	Perfluoropentanesulfonic acid	2706-91-4
	PFHxS	Perfluorohexanesulfonic acid	355-46-4
	PFHpS	Perfluoroheptanesulfonic acid	375-92-8
	PFOS	Perfluorooctanesulfonic acid	1763-23-1
	PFNS	Perfluorononanesulfonic acid	68259-12-1
	PFDS	Perfluorodecanesulfonic acid	335-77-3
	PFDoS	Perfluorododecanesulfonic acid	79780-39-5
	4:2FTS	4:2 fluorotelomersulfonic acid	757124-72-4
	6:2FTS	6:2 fluorotelomersulfonic acid	27619-97-2
	8:2FTS	8:2 fluorotelomersulfonic acid	39108-34-4
	PFOSA	Perfluorooctanesulfonamide	754-91-6
	NMeFOSA	N-methyl perfluorooctanesulfonamide	31506-32-8
	NEtFOSA	N-ethyl perfluorooctanesulfonamide	4151-50-2
	NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9
	NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6
	MeFOSE	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7
	NEtFOSE	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2
	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6
	ADONA	4,8-dioxa-3H-perfluorononanoic acid	919005-14-4
	PFMPA	Perfluoro-3-methoxypropanoic acid	377-73-1
	PFMBA	Perfluoro-4-methoxybutanoic acid	863090-89-5
	NFDHA	Perfluoro-3,6-dioxaheptanoic acid	151772-58-6
	9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1
	11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7
	3:3 FTCA	2H, 2H, 3H, 3H-perfluorohexanoic acid	356-02-5
	5:3 FTCA	2H, 2H, 3H, 3H-perfluorooctanoic acid	914637-49-3
	7:3 FTCA	2H, 2H, 3H, 3H-perfluorodecanoic acid	812-70-4
Internal Standards		Perfluoro-n-[13C4]butanoic acid	
		Perfluoro-n-[13C5]pentanoic acid	
		Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	
		Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	
		Perfluoro-n-[13C8]octanoic acid	
		Perfluoro-n-[13C9]nonanoic acid	

Group	Acronym	Chemical Name	CAS
Internal Standards		Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	
		Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	
		Perfluoro-n-[1,2-13C2]dodecanoic acid	
		Perfluoro-n-[1,2-13C2]tetradecanoic acid	
		Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	
		Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	
		Perfluoro-1-[13C8]octanesulfonic acid	
		1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	
		1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	
		1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	
		Perfluoro-1-[13C8]octanesulfonamide	
		N-methyl-d3-perfluoro-1-octanesulfonamide	
		N-ethyl-d5-perfluoro-1-octanesulfonamide	
		N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	
		N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	
		N-methyl-d7-perfluorooctanesulfonamidoethanol	
		N-ethyl-d9-perfluorooctane sulfonamidoethanol	
		Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	

B. Environmental Sample Matrices

The SLV study includes 32 environmental samples from four aqueous matrices (groundwater, surface water, wastewater, and landfill leachate), three solid matrices (soil, sediment, and biosolid) and tissue.

Table 2 is a list of the environmental samples from the EDDs. We also include three-character codes for each environmental sample, which are used in later figures. This table is file B-1 SLV Environmental Samples.csv from section B of the digital appendix.

Table 2. SLV Environmental Sample Names

Environmental Matrix	Sample Name	Abbreviation
Groundwater	GW CO	GW 1
	GW WL378	GW 2
	GW Wurtsmith AFB	GW 3
Landfill Leachate	LCH MSW Landfill	LC 1
	LCH C & D landfill	LC 2
	LCH MSW incineration ash landfill	LC 3
Surface water	SW COP-SW01 to 11	SW 1
	SW RP-SW01 to 11	SW 2

Environmental Matrix	Sample Name	Abbreviation
Wastewater	SW Marine Surface water	SW 3
	WW #3	WW 1
	WW #5	WW 2
	WW #6	WW 3
	WW #7	WW 4
	WW #8	WW 5
	WW #9	WW 6
	WW #10	WW 7
Biosolid	Biosolid #1	BS 1
	Biosolid #2	BS 2
	Biosolid #3	BS 3
Sediment	Marine sediment	SD 1
	Sediment #3	SD 2
	Sediment #2	SD 3
Soil	Soil 2014-107	SS 1
	Soil 2016-106	SS 2
	Soil 2017-111	SS 3
	Soil 2018-105	SS 4
	Soil 2018-116	SS 5
	Soil 2019-107	SS 6
	Soil 2019-110	SS 7
Tissue	Fish Tissue #1	TS 1
	Fish Tissue # 2	TS 2
	Clam Tissue	TS 3

Note: Correct abbreviations are shown for SD 2 and SD 3.

C. Sample Native Concentration Tables

We generated a total of 35 sample native concentration tables, which is section C of the digital appendix. Native concentration indicates the extent to which PFAS analytes occur in the environmental matrix before sampling. We calculated the mean native concentration and RSD for the 40 PFAS analytes, from three subsamples, for each environmental sample.

In the EDDs, the laboratory flagged results for various reasons indicated by specific letters based on the sponsor's instructions in the SLV study plan.[12] A list of all the flag letters that appear in the EDDs and their corresponding explanation is as follows:

- U indicates that the analyte was not detected and is reported as less than the limit of detection or as defined by the customer.
- J indicates that the reported result is an estimated value.
- B indicates a blank contamination. The recorded result is associated with a contaminated blank.
- Q indicates when one or more quality-control criteria failed.
- E indicates when the reported result exceeds the upper limit of the calibration range.
- D indicates when the reported result is from a dilution.

The laboratory also reported in the EDD the limit of quantification (LOQ), the smallest concentration of the analyte that is reliably detected and quantified, for the analytes. The LOQ for the PFAS analytes in the aqueous samples ranged from 1.43 to 296 ng/L, in the solid samples ranged from 0.16 to 44.3 µg/kg, and tissue samples ranged from 0.396 to 10.1 µg/kg.

At the sponsor's direction, we did not include in further analysis any data that were flagged by the lab as either B or U. Furthermore, at the sponsor's request, we did not calculate the mean native concentration if all three subsamples were qualified with a B or U. We could not calculate the RSD of the native concentration if there was only one subsample.

Table 3 is from a soil sample. This table is file C-19 Soil 2017-111 Native Concentration Table.csv in section C of the digital appendix. There are 32 tables like this, 1 for each of the 32 environmental samples.

[12] SERDP/ESTCP PFAS Method Validation Team, "Study Plan for Single-Laboratory Validation of PFAS by Isotope Dilution LC-MS/MS," April 2020.

Table 3. Example Sample Native Concentration Table

	L33303-13		L33303-14		L33303-15				
Analyte	Qualifier	Concentration (µg/kg)	Qualifier	Concentration (µg/kg)	Qualifier	Concentration (µg/kg)	Number of Detections	Mean	RSD
PFBA	U	0.409	U	0.41	U	0.412	0	NA	NA
PFPeA	U	0.0214	J	0.027	U	0.0216	1	0.03	NA
PFHxA	J	0.026	U	0.0204	JQ	0.021	2	0.02	15.04
PFHpA	U	0.0296	U	0.0296	U	0.0298	0	NA	NA
PFOA	U	0.0377	J	0.057	J	0.056	2	0.06	1.25
PFNA	U	0.0877	U	0.0879	U	0.0884	0	NA	NA
PFDA	U	0.0316	U	0.0317	U	0.0319	0	NA	NA
PFUnA	U	0.0337	U	0.0337	U	0.0339	0	NA	NA
PFDoA	U	0.0602	U	0.0603	U	0.0607	0	NA	NA
PFTrDA	U	0.0388	U	0.0388	U	0.0391	0	NA	NA
PFTeDA	U	0.0326	U	0.0327	U	0.0329	0	NA	NA
PFBS	U	0.0143	U	0.0143	U	0.0144	0	NA	NA
PFPeS	U	0.0153	U	0.0153	U	0.0154	0	NA	NA
PFHxS	U	0.0184	U	0.0184	U	0.0185	0	NA	NA
PFHpS	U	0.0581	U	0.0582	U	0.0586	0	NA	NA
PFOS	U	0.0683	U	0.0685	U	0.0689	0	NA	NA
PFNS	U	0.0469	U	0.047	U	0.0473	0	NA	NA
PFDS	U	0.0408	U	0.0409	U	0.0411	0	NA	NA
PFDoS	U	0.0388	U	0.0388	U	0.0391	0	NA	NA
4:2 FTS	U	0.288	U	0.288	U	0.29	0	NA	NA
6:2 FTS	U	0.118	U	0.119		2.69	1	2.69	NA
8:2 FTS	U	0.229	U	0.23	U	0.231	0	NA	NA
PFOSA	U	0.0693	U	0.0695	U	0.0699	0	NA	NA
N-MeFOSA	U	0.05	U	0.0501	U	0.0504	0	NA	NA

Analyte	L33303-13 Qualifier	L33303-13 Concentration (µg/kg)	L33303-14 Qualifier	L33303-14 Concentration (µg/kg)	L33303-15 Qualifier	L33303-15 Concentration (µg/kg)	Number of Detections	Mean	RSD
N-EtFOSA	JQ	0.092	JQ	0.067	JQ	0.101	3	0.09	20.33
N-MeFOSAA	U	0.0306	U	0.0307	U	0.0308	0	NA	NA
N-EtFOSAA	U	0.0449	U	0.045	U	0.0452	0	NA	NA
N-MeFOSE	U	0.207	U	0.207	U	0.209	0	NA	NA
N-EtFOSE	U	0.252	U	0.252	U	0.254	0	NA	NA
HFPO-DA	U	0.139	U	0.139	U	0.14	0	NA	NA
ADONA	U	0.0581	U	0.0582	U	0.0586	0	NA	NA
PFMPA	U	0.0337	U	0.0337	U	0.0339	0	NA	NA
PFMBA	U	0.0296	U	0.0296	U	0.0298	0	NA	NA
NFDHA	U	0.0857	U	0.0858	U	0.0864	0	NA	NA
9Cl-PF3ONS	U	0.0388	U	0.0388	U	0.0391	0	NA	NA
11Cl-PF3OUdS	U	0.0724	U	0.0726	U	0.073	0	NA	NA
PFEESA	U	0.0184	U	0.0184	U	0.0185	0	NA	NA
3:3 FTCA	U	0.0612	U	0.0613	U	0.0617	0	NA	NA
5:3 FTCA	U	0.37	U	0.371	U	0.373	0	NA	NA
7:3 FTCA	U	0.314	U	0.315	U	0.317	0	NA	NA

A description of each column in Table 3:

- Analyte—Always 40 PFAS analytes.
- Qualifier—Lab flag for the native concentration of each of the three analyte subsamples from the EDD.
- Concentration—Native concentration for each of the three subsamples of the analyte from the EDD in µg/kg (or ng/L for aqueous matrices).
- Number of Detections—Number of subsamples for the analyte that do not have a U or B flag for native concentration. Could be 0, 1, 2 or 3.
- Mean—Mean native concentration of the subsamples that do not have a U or B flag. If all three subsamples have U or B, then the value is NA.
- RSD— Percent RSD of the mean native concentration. If the number of samples is less than two then NA was used.

The laboratory created additional subsamples from the groundwater samples to later spike at a higher concentration. We generated tables for the additional groundwater native concentration subsamples.

Table 4 is from a groundwater sample. This table is file C-1(b) GW CO Native HIGH 2 Concentration Table.csv from section C of the digital appendix. There are three "HIGH 2" tables like this, one for each of the three groundwater samples.

Table 4. Example Groundwater Sample Native Concentration Table

	L33877-1	
Analyte	**Qualifier**	**Concentration (ng/L)**
PFBA	J	4.86
PFPeA		5.81
PFHxA		4.96
PFHpA		1.98
PFOA		5.74
PFNA	J	0.446
PFDA	U	0.329
PFUnA	U	0.259
PFDoA	U	0.379
PFTrDA	U	0.24
PFTeDA	U	0.259
PFBS		5.05
PFPeS		1.99
PFHxS		11.1
PFHpS	J	0.261

	L33877-1	
Analyte	Qualifier	Concentration (ng/L)
PFOS		20.1
PFNS	U	0.299
PFDS	U	0.329
PFDoS	U	0.18
4:2 FTS	U	2.28
6:2 FTS	U	3.96
8:2 FTS	U	1.57
PFOSA	J	0.968
N-MeFOSA	U	0.2
N-EtFOSA	U	0.579
N-MeFOSAA	U	0.589
N-EtFOSAA	U	0.319
N-MeFOSE	U	1.19
N-EtFOSE	U	1.02
HFPO-DA	U	0.409
ADONA	U	0.778
PFMPA	U	0.18
PFMBA	U	0.12
NFDHA	U	1.38
9Cl-PF3ONS	U	0.868
11Cl-PF3OUdS	U	0.818
PFEESA	U	0.14
3:3 FTCA	U	0.719
5:3 FTCA	U	5.06
7:3 FTCA	U	5.93

A description of each column in Table 4:

- Analyte—Always 40 PFAS analytes.
- Qualifier—Lab flag for the native concentration of the analyte subsample from the EDD.
- Concentration—Native concentration for the subsample of the analyte from the EDD in ng/L.

D. Sample Matrix Recovery Tables

We generated a total of 32 sample matrix recovery tables, which is section D of the digital appendix. We calculated the mean spike concentration and mean percent recovery for the 40 PFAS analytes, from three subsamples, for each environmental sample. Spike

concentration indicates the known amount of analyte added to the native sample, while percent recovery indicates the percent of the analyte detected compared with the known amount. We subtracted the native concentration from the spike concentration before calculating the percent recovery, for each subsample. At the sponsor's request, we reported only positive percent recovery values. Upon the sponsor's direction, we also did not include spike concentrations that were flagged by the lab as either U (where it was not detected and less than the limit of detection) or B (where the results were associated with a contaminated blank in our calculations). We reported the mean spike concentration value with an asterisk when all three subsamples had B-flags. For all samples except groundwater, we also calculated the mean and percent RSD of the percent recoveries for an analyte across the three spike concentrations: low, medium, and high. At the sponsor's direction, we did not include instances where the mean native concentration (values also reported in digital appendix section C) were greater than the mean spike concentration for an analyte in the calculations of the mean and RSD of the mean percent recovery, for a sample.

Table 5 is from a soil sample (same sample in Table 3). This table is file D-19 Soil 2017-111 Matrix Recovery.csv in section D of the digital appendix. There are 29 tables like this, 1 for each solid and most aqueous matrices (except groundwater). The % Recovery column is the mean of the calculated percent recoveries for the three subsamples. At the sponsor's request, the % Mean Recovery column is the mean of the mean percent recoveries; we recognize that it is potentially biased toward means with lower sample sizes since the sample means are not composed from the same number of data.

Table 5. Example Sample Matrix Recovery Table

	Low Spike Recovery					Medium Spike Recovery				
Analyte	Number Detects	Native Mean PFAS Concentration (µg/kg)	Mean PFAS Spike Concentration (µg/kg)	% Recovery	Native> Spike (Yes/No)	Number Detects	Native Mean PFAS Concentration (µg/kg)	Mean PFAS Spike Concentration (µg/kg)	% Recovery	Native> Spike (Yes/No)
PFBA	0	NA	2.62	104.07	No	0	NA	3.94	108.55	No
PFPeA	1	0.027	1.31	112.7	No	1	0.027	1.97	111	No
PFHxA	2	0.0235	0.656	95.71	No	2	0.0235	0.984	104.02	No
PFHpA	0	NA	0.656	105.34	No	0	NA	0.984	104.38	No
PFOA	2	0.0565	0.656	99.67	No	2	0.0565	0.984	101.68	No
PFNA	0	NA	0.656	105.85	No	0	NA	0.984	109.8	No
PFDA	0	NA	0.656	102.24	No	0	NA	0.984	112.17	No
PFUnA	0	NA	0.656	104.93	No	0	NA	0.984	108.79	No
PFDoA	0	NA	0.656	105.69	No	0	NA	0.984	105.37	No
PFTrDA	0	NA	0.656	115.04	No	0	NA	0.984	116.23	No
PFTeDA	0	NA	0.656	102.65	No	0	NA	0.984	108.79	No
PFBS	0	NA	0.656	107.02	No	0	NA	0.984	109.45	No
PFPeS	0	NA	0.658	101.83	No	0	NA	0.987	107.78	No
PFHxS	0	NA	0.656	104.88	No	0	NA	0.984	114.88	No
PFHpS	0	NA	0.657	98.17	No	0	NA	0.986	103.28	No
PFOS	0	NA	0.656	113.78	No	0	NA	0.984	118.6	No
PFNS	0	NA	0.657	88.89	No	0	NA	0.986	99.76	No
PFDS	0	NA	0.656	98.37	No	0	NA	0.984	101.32	No
PFDoS	0	NA	0.657	76.7	No	0	NA	0.985	86.12	No
4:2 FTS	0	NA	2.62	96.06	No	0	NA	3.94	106.01	No
6:2 FTS	1	2.69	2.36*	NA	NA	1	2.69	3.55	37.5	No
8:2 FTS	0	NA	2.62	121.35	No	0	NA	3.94	127.01	No
PFOSA	0	NA	0.656	105.13	No	0	NA	0.984	109.79	No
N-MeFOSA	0	NA	0.754	109.85	No	0	NA	1.13	108.85	No
N-EtFOSA	3	0.0867	1.64	108.74	No	3	0.0867	2.46	115.83	No
N-MeFOSAA	0	NA	0.656	101.53	No	0	NA	0.984	97.63	No
N-EtFOSAA	0	NA	0.656	110.17	No	0	NA	0.984	112.16	No
N-MeFOSE	0	NA	6.56	104.98	No	0	NA	9.84	112.51	No
N-EtFOSE	0	NA	4.91	109.43	No	0	NA	7.37	113.56	No
HFPO-DA	0	NA	2.49	109.9	No	0	NA	3.74	119.7	No
ADONA	0	NA	2.63	112.8	No	0	NA	3.95	119.98	No
PFMPA	0	NA	1.31	116.03	No	0	NA	1.97	116.95	No
PFMBA	0	NA	0.656	122.27	No	0	NA	0.984	116.56	No
NFDHA	0	NA	1.31	97.2	No	0	NA	1.97	88.15	No
9Cl-PF3ONS	0	NA	2.63	120.53	No	0	NA	3.94	128.09	No
11Cl-PF3OUdS	0	NA	2.63	114.2	No	0	NA	3.94	121.27	No
PFEESA	0	NA	0.656	99.95	No	0	NA	0.984	94.93	No
3:3 FTCA	0	NA	2.62	106.1	No	0	NA	3.94	112.86	No
5:3 FTCA	0	NA	16.4	97.56	No	0	NA	24.6	92.44	No
7:3 FTCA	0	NA	16.4	97.36	No	0	NA	24.6	99.87	No

Analyte	Number Detects	High Spike Recovery Native Mean PFAS Concentration (µg/kg)	High Spike Recovery Mean PFAS Spike Concentration (µg/kg)	High Spike Recovery % Recovery	High Spike Recovery Native> Spike (Yes/No)	ALL SPIKE RECOVERIES n =	ALL SPIKE RECOVERIES % Mean Recovery	ALL SPIKE RECOVERIES RSD
PFBA	0	NA	6.57	104.41	No	3	105.68	2.36
PFPeA	1	0.027	3.28	109.63	No	3	111.11	1.38
PFHxA	2	0.0235	1.64	102.23	No	3	100.65	4.35
PFHpA	0	NA	1.64	105.49	No	3	105.07	0.57
PFOA	2	0.0565	1.64	97.98	No	3	99.78	1.86
PFNA	0	NA	1.64	103.46	No	3	106.37	3.01
PFDA	0	NA	1.64	106.5	No	3	106.97	4.66
PFUnA	0	NA	1.64	104.27	No	3	106	2.3
PFDoA	0	NA	1.64	106.71	No	3	105.92	0.66
PFTrDA	0	NA	1.64	108.74	No	3	113.34	3.55
PFTeDA	0	NA	1.64	105.49	No	3	105.64	2.91
PFBS	0	NA	1.64	106.3	No	3	107.59	1.53
PFPeS	0	NA	1.65	100	No	3	103.2	3.94
PFHxS	0	NA	1.64	108.74	No	3	109.5	4.61
PFHpS	0	NA	1.65	98.99	No	3	100.15	2.74
PFOS	0	NA	1.64	111.99	No	3	114.79	2.98
PFNS	0	NA	1.64	110.34	No	3	99.66	10.76
PFDS	0	NA	1.64	100.2	No	3	99.97	1.49
PFDoS	0	NA	1.64	82.35	No	3	81.73	5.8
4:2 FTS	0	NA	6.57	107.91	No	3	103.33	6.16
6:2 FTS	1	2.69	5.92	61.96	No	2	49.73	34.78
8:2 FTS	0	NA	6.57	121.4	No	3	123.25	2.64
PFOSA	0	NA	1.64	105.08	No	3	106.67	2.54
N-MeFOSA	0	NA	1.89	111.29	No	3	110	1.11
N-EtFOSA	3	0.0867	4.1	114.06	No	3	112.88	3.27
N-MeFOSAA	0	NA	1.64	103.66	No	3	100.94	3.03
N-EtFOSAA	0	NA	1.64	111.79	No	3	111.37	0.95
N-MeFOSE	0	NA	16.4	108.74	No	3	108.74	3.46
N-EtFOSE	0	NA	12.3	111.38	No	3	111.46	1.85
HFPO-DA	0	NA	6.24	100.75	No	3	110.12	8.61
ADONA	0	NA	6.59	91.91	No	3	108.23	13.48
PFMPA	0	NA	3.28	102.74	No	3	111.91	7.1

Analyte	Number Detects	High Spike Recovery: Native Mean PFAS Concentration (µg/kg)	High Spike Recovery: Mean PFAS Spike Concentration (µg/kg)	High Spike Recovery: % Recovery	High Spike Recovery: Native> Spike (Yes/No)	ALL SPIKE RECOVERIES: n =	ALL SPIKE RECOVERIES: % Mean Recovery	ALL SPIKE RECOVERIES: RSD
PFMBA	0	NA	1.64	105.69	No	3	114.84	7.33
NFDHA	0	NA	3.28	99.09	No	3	94.81	6.16
9Cl-PF3ONS	0	NA	6.58	96.26	No	3	114.96	14.47
11Cl-PF3OUdS	0	NA	6.58	93.93	No	3	109.8	12.93
PFEESA	0	NA	1.64	96.75	No	3	97.21	2.61
3:3 FTCA	0	NA	6.57	101.17	No	3	106.71	5.5
5:3 FTCA	0	NA	41	96.83	No	3	95.61	2.9
7:3 FTCA	0	NA	41	102.19	No	3	99.81	2.42

A description of each column in Table 5:

- Headings: Low Spike Recovery, Medium Spike Recovery, and High Spike Recovery
  - Analyte—Always 40 PFAS analytes.
  - Number Detects—Number of EDD entries for the analyte that do not have a U or B flag for native concentration. Could be 0, 1, 2 or 3.
  - Native Mean PFAS Concentration—Mean native concentration of the EDD entries that do not have a U or B flag. Or NA, if all EDD entries have a U or B flag. Identical to values reported in the corresponding sample native concentration table in section C of the digital appendix.
  - Mean PFAS Spike Concentration—Mean spike concentration of the EDD entries that do not have a U or B flag. If all three subsamples have B-flags, then the mean concentration value is reported with an asterisk.
  - % Recovery—Mean of the three subsamples' percent recoveries for spike concentrations that do not have a U or B flag. The native concentration is subtracted before calculating each of the three subsamples' percent recoveries. If the native concentration is NA, then a value of 0 was used instead. If mean native concentration is greater than the spike concentration, then NA was used. If three subsample values have a B-flag, then NA was used. If the mean percent recovery is less than 0, then NA was used.
  - Native> Spike (Yes/No)—Whether or not the mean native concentration is greater than the mean spike concentration. The mean spike concentration value is pulled directly from column Native Mean PFAS Concentration and compared with column % Recovery. If the mean native concentration was NA, then we used the value of 0 instead.
- Heading: ALL SPIKE RECOVERIES
  - n=—Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e., a No in columns Native> Spike (Yes/No)) and when there is not an NA in column % Recovery. Could be 0, 1, 2 or 3.
  - % Mean Recovery—Mean of the mean percent recoveries where the mean native concentration is not greater than the mean spike (i.e., a No in columns Native> Spike (Yes/No)) and when there is not an NA in column % Recovery.

– RSD—Percent RSD of the mean percent recoveries, used to calculate column % Mean Recovery. If the number of samples is less than two then NA was used.

The laboratory spiked the subsamples in the groundwater matrix with an additional concentration (i.e., “high 2”). Table 6 is from a groundwater sample (same sample in Table 4). This table is file D-1 GW CO Matrix Recovery.csv in section D of the digital appendix. There are three tables like this, one for each of the three groundwater samples. The % Recovery column is the mean of the calculated percent recoveries for the three groundwater subsamples. The % Mean Recovery column is the mean of the mean percent recoveries from all groundwater spike concentrations and is potentially biased toward means with lower sample sizes since the sample means are not composed from the same number of data.

Table 6. Example Groundwater Sample Matrix Recovery Table

	Low Spike Recovery					Medium Spike Recovery				
Analyte	Number Detects	Native Mean PFAS Concentration (ng/L)	Mean PFAS Spike Concentration (ng/L)	% Recovery	Native> Spike (Yes/No)	Number Detects	Native Mean PFAS Concentration (ng/L)	Mean PFAS Spike Concentration	% Recovery	Native> Spike (Yes/No)
PFBA	3	18.2	15	NA	Yes	3	18.2	29.9	109.89	No
PFPeA	3	22.2	7.5	NA	Yes	3	22.2	14.9	NA	Yes
PFHxA	3	19.6	3.75	NA	Yes	3	19.6	7.47	NA	Yes
PFHpA	3	7.47	3.75	NA	Yes	3	7.47	7.47	102.93	No
PFOA	3	23	3.75	NA	Yes	3	23	7.47	NA	Yes
PFNA	3	2.08	3.75	90.56	No	3	2.08	7.47	102.16	No
PFDA	3	0.556	3.75	96.23	No	3	0.556	7.47	110.57	No
PFUnA	0	NA	3.75	91.09	No	0	NA	7.47	110.87	No
PFDoA	0	NA	3.75	82.26	No	0	NA	7.47	107.12	No
PFTrDA	0	NA	3.75	89.23	No	0	NA	7.47	112.42	No
PFTeDA	0	NA	3.75	91.86	No	0	NA	7.47	110.83	No
PFBS	3	20.6	3.75	NA	Yes	3	20.6	7.47	NA	Yes
PFPeS	3	8.06	3.76	NA	Yes	3	8.06	7.49	NA	Yes
PFHxS	3	48.1	3.75	NA	Yes	3	48.1	7.47	NA	Yes
PFHpS	3	1.3	3.76	91.96	No	3	1.3	7.49	108.63	No
PFOS	3	124	3.75	NA	Yes	3	124	7.47	NA	Yes
PFNS	0	NA	3.76	83.08	No	0	NA	7.48	97.51	No
PFDS	0	NA	3.75	78.04	No	0	NA	7.47	91.76	No
PFDoS	0	NA	3.75	74.59	No	0	NA	7.48	94.04	No
4:2 FTS	0	NA	15	87.74	No	0	NA	29.9	105.43	No
6:2 FTS	0	NA	13.5	108.06	No	0	NA	26.9	120.16	No
8:2 FTS	0	NA	15	108.2	No	0	NA	29.9	125.28	No
PFOSA	3	5.6	3.75	NA	Yes	3	5.6	7.47	130.25	No
N-MeFOSA	0	NA	4.31	73.25	No	0	NA	8.59	110.98	No
N-EtFOSA	0	NA	9.38	73.76	No	0	NA	18.7	105.67	No
N-MeFOSAA	0	NA	3.75	90.25	No	0	NA	7.47	118.67	No
N-EtFOSAA	0	NA	3.75	87.55	No	0	NA	7.47	114.16	No
N-MeFOSE	0	NA	37.5	85.57	No	0	NA	74.7	109.08	No
N-EtFOSE	0	NA	28.1	87.64	No	0	NA	56	115.45	No
HFPO-DA	0	NA	14.2	92.23	No	0	NA	28.4	105.74	No
ADONA	0	NA	15	84.22	No	0	NA	29.9	99.66	No
PFMPA	0	NA	7.5	95.42	No	0	NA	14.9	112.49	No
PFMBA	0	NA	3.75	84.43	No	0	NA	7.47	110.31	No
NFDHA	0	NA	7.5	83.42	No	0	NA	14.9	105.37	No
9Cl-PF3ONS	0	NA	15	82.45	No	0	NA	29.9	102.55	No
11Cl-PF3OUdS	0	NA	15	72.26	No	0	NA	29.9	94.43	No
PFEESA	0	NA	3.75	91.34	No	0	NA	7.47	106.35	No
3:3 FTCA	0	NA	15	91.75	No	0	NA	29.9	109.8	No
5:3 FTCA	0	NA	93.8	85.84	No	0	NA	187	97.1	No

Analyte	Low Spike Recovery					Medium Spike Recovery				
	Number Detects	Native Mean PFAS Concentration (ng/L)	Mean PFAS Spike Concentration (ng/L)	% Recovery	Native> Spike (Yes/No)	Number Detects	Native Mean PFAS Concentration (ng/L)	Mean PFAS Spike Concentration	% Recovery	Native> Spike (Yes/No)
7:3 FTCA	0	NA	93.8	83.5	No	0	NA	187	96.76	No

Individual Sample Matrix Recovery Table (Part 2)

Analyte	High Spike Recovery					High 2 Spike Recovery (groundwater only)				
	Number Detects	Native Mean PFAS Concentration (ng/L)	Mean PFAS Spike Concentration (ng/L)	% Recovery	Native> Spike (Yes/No)	Number Detects	Native Mean PFAS Concentration (ng/L)	Mean PFAS Spike Concentration (ng/L)	% Recovery	Native> Spike (Yes/No)
PFBA	3	18.2	44.7	103.36	No	1	4.86	199	99.74	No
PFPeA	3	22.2	22.4	117.45	No	1	5.81	99.7	106.14	No
PFHxA	3	19.6	11.2	NA	Yes	1	4.96	49.9	97.8	No
PFHpA	3	7.47	11.2	107.45	No	1	1.98	49.9	101.12	No
PFOA	3	23	11.2	NA	Yes	1	5.74	49.9	97.05	No
PFNA	3	2.08	11.2	106.72	No	1	0.446	49.9	98.78	No
PFDA	3	0.556	11.2	109.63	No	0	NA	49.9	104.62	No
PFUnA	0	NA	11.2	108.32	No	0	NA	49.9	99.95	No
PFDoA	0	NA	11.2	107.21	No	0	NA	49.9	101.31	No
PFTrDA	0	NA	11.2	108.95	No	0	NA	49.9	96.85	No
PFTeDA	0	NA	11.2	108.06	No	0	NA	49.9	99.35	No
PFBS	3	20.6	11.2	NA	Yes	1	5.05	49.9	103.84	No
PFPeS	3	8.06	11.2	111.29	No	1	1.99	50	94.23	No
PFHxS	3	48.1	11.2	NA	Yes	1	11.1	49.9	107.14	No
PFHpS	3	1.3	11.2	105.61	No	1	0.261	50	93.22	No
PFOS	3	124	11.2	NA	Yes	1	20.1	49.9	108.59	No
PFNS	0	NA	11.2	108.03	No	0	NA	50	97.6	No
PFDS	0	NA	11.2	93.74	No	0	NA	49.9	92.26	No
PFDoS	0	NA	11.2	91.69	No	0	NA	49.9	88.49	No
4:2 FTS	0	NA	44.7	109.69	No	0	NA	199	98.19	No
6:2 FTS	0	NA	40.3	115.16	No	0	NA	180	102.43	No
8:2 FTS	0	NA	44.7	124.31	No	0	NA	199	114.93	No
PFOSA	3	5.6	11.2	147.49	No	1	0.968	49.9	102.61	No
N-MeFOSA	0	NA	12.9	102.31	No	0	NA	57.4	98.03	No
N-EtFOSA	0	NA	28	100.34	No	0	NA	125	102.21	No
N-MeFOSAA	0	NA	11.2	144.62	No	0	NA	49.9	97.82	No
N-EtFOSAA	0	NA	11.2	127.37	No	0	NA	49.9	98.29	No
N-MeFOSE	0	NA	112	106.26	No	0	NA	499	105.44	No
N-EtFOSE	0	NA	83.9	109.89	No	0	NA	374	108.82	No
HFPO-DA	0	NA	42.5	106.87	No	0	NA	190	100.14	No
ADONA	0	NA	44.9	106.91	No	0	NA	200	84.12	No
PFMPA	0	NA	22.4	100.15	No	0	NA	99.7	100.02	No
PFMBA	0	NA	11.2	100.31	No	0	NA	49.9	99.83	No

Individual Sample Matrix Recovery Table (Part 2)

	High Spike Recovery					High 2 Spike Recovery (groundwater only)				
Analyte	Number Detects	Native Mean PFAS Concentration (ng/L)	Mean PFAS Spike Concentration (ng/L)	% Recovery	Native> Spike (Yes/No)	Number Detects	Native Mean PFAS Concentration (ng/L)	Mean PFAS Spike Concentration (ng/L)	% Recovery	Native> Spike (Yes/No)
NFDHA	0	NA	22.4	92.83	No	0	NA	99.7	82.88	No
9Cl-PF3ONS	0	NA	44.8	104.81	No	0	NA	200	89.07	No
11Cl-PF3OUdS	0	NA	44.8	99.02	No	0	NA	200	88.26	No
PFEESA	0	NA	11.2	104.17	No	0	NA	49.9	97.44	No
3:3 FTCA	0	NA	44.7	102.01	No	0	NA	199	96.8	No
5:3 FTCA	0	NA	280	96.18	No	0	NA	1250	85.42	No
7:3 FTCA	0	NA	280	92.61	No	0	NA	1250	93.37	No

Individual Sample Matrix Recovery Table (Part 3)

	ALL SPIKE RECOVERIES		
Analyte	n =	% Mean Recovery	RSD
PFBA	3	104.33	4.93
PFPeA	2	111.8	7.15
PFHxA	1	97.8	NA
PFHpA	3	103.84	3.14
PFOA	1	97.05	NA
PFNA	4	99.55	6.85
PFDA	4	105.26	6.24
PFUnA	4	102.56	8.74
PFDoA	4	99.47	11.87
PFTrDA	4	101.86	10.55
PFTeDA	4	102.53	8.42
PFBS	1	103.84	NA
PFPeS	2	102.76	11.74
PFHxS	1	107.14	NA
PFHpS	4	99.86	8.51
PFOS	1	108.59	NA
PFNS	4	96.55	10.62
PFDS	4	88.95	8.23
PFDoS	4	87.2	9.99
4:2 FTS	4	100.26	9.58
6:2 FTS	4	111.46	7
8:2 FTS	4	118.18	6.88
PFOSA	3	126.78	17.86
N-MeFOSA	4	96.14	16.83
N-EtFOSA	4	95.49	15.35
N-MeFOSAA	4	112.84	21.59
N-EtFOSAA	4	106.84	16.4
N-MeFOSE	4	101.59	10.62

Individual Sample Matrix Recovery Table (Part 3)

	ALL SPIKE RECOVERIES		
Analyte	**n =**	**% Mean Recovery**	**RSD**
N-EtFOSE	4	105.45	11.59
HFPO-DA	4	101.25	6.61
ADONA	4	93.73	12.19
PFMPA	4	102.02	7.17
PFMBA	4	98.72	10.82
NFDHA	4	91.13	11.56
9Cl-PF3ONS	4	94.72	11.33
11Cl-PF3OUdS	4	88.49	13.2
PFEESA	4	99.82	6.82
3:3 FTCA	4	100.09	7.7
5:3 FTCA	4	91.14	6.99
7:3 FTCA	4	91.56	6.19

See Table 5 for a description of the columns for the spike concentrations Low Spike Recovery, Medium Spike Recovery, High Spike Recovery, and ALL SPIKE RECOVERIES. A description of the "High 2" columns in Table 6:

- Heading: High 2 Spike Recovery (groundwater only)
  - Number Detects—Number of EDD entries for the analyte that do not have a U or B flag for native concentration. Could be 0 or 1.
  - Native Mean PFAS Concentration—Mean native concentration of the EDD entry that does not have a U or B flag. Or NA, if the EDD entry has a U or B flag. Values are not labeled as means in the corresponding sample native concentration tables in section c of the digital appendix.
  - Mean PFAS Spike Concentration—Mean spike concentration of the EDD entry that does not have a U or B flag.
  - % Recovery—Mean of the three subsamples' percent recoveries for spike concentrations that do not have a U or B flag. The native concentration is subtracted before calculating each of the three subsamples' percent recoveries. If the native concentration is NA, then a value of 0 was used instead. If mean native concentration is greater than the spike concentration, then NA was used. If three subsample values have a B-flag, then NA was used. If the mean percent recovery is less than 0, then NA was used.
  - Native> Spike (Yes/No)—Whether or not the mean native concentration is greater than the mean spike concentration. The mean spike concentration is value is pulled directly from column Native Mean PFAS Concentration and compared with the column % Recovery. If the mean native concentration is NA, then we used the value of 0 instead.

E. Media Type Matrix Recovery Tables

We generated a total of 11 tables for section E of the digital appendix. We calculated the mean of the sample mean percent recovery, percent RSD, and SD for the 40 PFAS analytes, for the 8 environmental media (groundwater, surface water, wastewater, landfill leachate, soil, sediment, biosolid, and tissue). While the previous tables discussed in section D provide results for each of the 32 individual environmental samples, these tables in section E *summarize* those results for each of the 8 environmental media. At the sponsor's direction, we used the sample mean percent recovery values from tables in section D of the digital appendix for the three spike concentrations: low, medium, and high. we also identified the lowest and highest sample mean percent recovery for the matrix. We also calculated the mean of the matrix mean percent recovery, percent RSD, and SD for the 40 analytes, for aqueous and solid matrices for the three spike concentrations.

Also at request of the sponsor, we calculated the mean of the mean percent recoveries, percent RSD, and SD of the percent recoveries for an analyte across all spike concentrations, for the media type. We also identified the lowest and highest sample mean percent recovery for the media type.

Table 7 is constructed from all soil samples. This is file E-5 Soil Sample Matrix Recovery.csv in section E of the digital appendix. There are seven tables like this, one for all solid and most aqueous matrices (except groundwater). The % Mean Recovery column is the mean of the mean percent recoveries and is potentially biased toward means with lower sample sizes since the sample means are not composed from the same number of data. The Lowest % Recovery and Highest % Recovery columns are the mean of the three subsamples' percent recoveries for the given matrix.

Table 7. Example Matrix Recovery Table

Analyte	Low Spike Recovery n =	Low Spike Recovery % Mean Recovery	Low Spike Recovery RSD	Low Spike Recovery SD	Medium Spike Recovery n =	Medium Spike Recovery % Mean Recovery	Medium Spike Recovery RSD	Medium Spike Recovery SD	High Spike Recovery n =	High Spike Recovery % Mean Recovery	High Spike Recovery RSD	High Spike Recovery SD
PFBA	7	106.26	1.6	1.7	7	106.6	2.27	2.42	7	105.63	0.86	0.91
PFPeA	7	107.08	5.33	5.71	7	104.95	5.75	6.04	7	109.7	1.09	1.2
PFHxA	7	99.83	3.12	3.12	7	101.28	3.91	3.96	7	101.13	1.84	1.86
PFHpA	7	100.9	3.38	3.42	7	101.06	3.09	3.12	7	102.3	3.55	3.63
PFOA	6	98.88	2.69	2.66	6	100.48	4.22	4.24	6	100.63	1.55	1.56
PFNA	7	102.9	3.65	3.76	7	103.94	4.96	5.15	7	103.64	1.76	1.83
PFDA	6	103.24	3.12	3.22	6	105.89	5.3	5.61	6	106.92	1.05	1.12
PFUnA	7	101.03	5.27	5.32	7	104.41	4.63	4.83	7	103.42	1.07	1.11
PFDoA	6	103.25	3.93	4.06	7	105.59	6.68	7.06	7	106.54	5.4	5.76
PFTrDA	7	111.91	3.45	3.87	7	114.71	1.51	1.73	7	111.14	4.2	4.67
PFTeDA	7	103.8	5.79	6.01	7	106.43	2.54	2.71	7	105.14	0.79	0.84
PFBS	7	104.18	3.02	3.14	7	106.39	3.16	3.36	7	106.44	1.96	2.09
PFPeS	7	103.61	1.79	1.85	7	103.39	3.33	3.44	7	104.1	3.67	3.82
PFHxS	7	107.3	1.93	2.07	7	110.53	2.82	3.12	7	110.14	2.47	2.72
PFHpS	7	97.71	1.4	1.37	7	99.74	3.95	3.94	7	99.82	1.98	1.98
PFOS	6	107.53	3.25	3.5	6	110.82	5.26	5.83	7	109.09	2.53	2.75
PFNS	7	101.5	9.23	9.37	7	99.83	4.78	4.77	7	101.09	7.2	7.28
PFDS	7	99.01	3.91	3.88	7	102.3	2.64	2.7	7	100.48	2.82	2.83
PFDoS	7	88.73	9.09	8.06	7	91.24	5.28	4.82	7	90.74	5.11	4.64
4:2 FTS	7	100.67	3.43	3.45	7	105.28	3.18	3.34	7	105.78	2.07	2.19
6:2 FTS	3	107.68	4.01	4.32	5	100.12	39.51	39.55	7	79.36	49.89	39.59
8:2 FTS	7	120.18	5.05	6.07	7	122.28	4.31	5.27	7	122.01	1.17	1.43
PFOSA	7	104.41	1.81	1.89	7	107.72	2.83	3.05	7	106.36	1.1	1.17
N-MeFOSA	7	108.96	3.41	3.72	7	109.36	1.75	1.91	7	108.35	1.63	1.77
N-EtFOSA	7	111.05	2.13	2.36	7	113.71	2.31	2.62	7	113.28	1.02	1.16
N-MeFOSAA	7	100.51	3.36	3.38	7	104.68	4.67	4.89	7	103.88	3.65	3.79
N-EtFOSAA	7	109.07	2.66	2.91	7	108.32	4.5	4.87	7	110.84	2.39	2.65
N-MeFOSE	7	105.4	1.56	1.65	7	109.35	2.29	2.51	7	108.52	0.78	0.84

Analyte	Low Spike Recovery n =	Low Spike Recovery % Mean Recovery	Low Spike Recovery RSD	Low Spike Recovery SD	Medium Spike Recovery n =	Medium Spike Recovery % Mean Recovery	Medium Spike Recovery RSD	Medium Spike Recovery SD	High Spike Recovery n =	High Spike Recovery % Mean Recovery	High Spike Recovery RSD	High Spike Recovery SD
N-EtFOSE	7	108.55	1.39	1.51	7	110.45	2.84	3.14	7	112.16	1.04	1.17
HFPO-DA	7	104.56	6.11	6.39	7	104.67	11.17	11.69	7	105.92	5.19	5.5
ADONA	7	106.41	9.23	9.82	7	106.44	8.34	8.88	7	99.31	8.55	8.49
PFMPA	7	113.46	2.88	3.26	7	110.57	4.44	4.91	7	101.56	2.21	2.24
PFMBA	7	117.07	3.54	4.15	7	113.53	4.55	5.17	7	100.74	5.02	5.05
NFDHA	7	95.39	20.38	19.44	7	91.4	14.58	13.32	7	104.15	12.21	12.72
9Cl-PF3ONS	7	114.11	8.59	9.81	7	115.9	8.57	9.94	7	104.89	9.33	9.79
11Cl-PF3OUdS	7	111.36	7.63	8.5	7	112.96	7.5	8.48	7	106.55	11.59	12.34
PFEESA	7	103.38	2.78	2.87	7	99.01	3	2.97	7	99.39	2.08	2.06
3:3 FTCA	7	108.56	2.81	3.05	7	106.52	4.76	5.08	7	97.82	4.44	4.34
5:3 FTCA	7	100.01	5.4	5.4	7	94.09	3.11	2.93	7	95.14	6.74	6.41
7:3 FTCA	7	100.2	4.08	4.09	7	97.76	2.68	2.62	7	99.98	5.88	5.88

Analyte	n =	ALL SPIKE RECOVERIES % Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFBA	3	106.16	101.56	108.65	0.46	0.49
PFPeA	3	107.25	97.74	115.34	2.22	2.38
PFHxA	3	100.75	95.71	106.25	0.79	0.8
PFHpA	3	101.42	96.88	107.24	0.75	0.76
PFOA	3	100	94.2	106.42	0.97	0.97
PFNA	3	103.5	98.27	110.54	0.52	0.54
PFDA	3	105.35	99.3	112.17	1.8	1.9
PFUnA	3	102.95	91.73	108.82	1.69	1.74
PFDoA	3	105.13	90.57	116.27	1.61	1.69
PFTrDA	3	112.59	105.58	119.32	1.67	1.88
PFTeDA	3	105.12	93.43	112.88	1.25	1.31
PFBS	3	105.67	99.21	109.65	1.22	1.29
PFPeS	3	103.7	98.11	110.76	0.35	0.37
PFHxS	3	109.32	104.73	114.88	1.61	1.76

Analyte	n =	ALL SPIKE RECOVERIES % Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFHpS	3	99.09	94	103.28	1.21	1.2
PFOS	3	109.15	102.49	118.6	1.51	1.65
PFNS	3	100.81	88.02	111.46	0.87	0.87
PFDS	3	100.6	91.87	106.42	1.64	1.65
PFDoS	3	90.24	76.7	100.1	1.47	1.33
4:2 FTS	3	103.91	96.06	110.38	2.71	2.82
6:2 FTS	3	95.72	11.24	147.45	15.32	14.66
8:2 FTS	3	121.49	113.31	131.95	0.94	1.14
PFOSA	3	106.16	102.02	110.54	1.57	1.66
N-MeFOSA	3	108.89	102.26	113.86	0.47	0.51
N-EtFOSA	3	112.68	108.13	117.03	1.27	1.43
N-MeFOSAA	3	103.02	97.15	110.15	2.15	2.21
N-EtFOSAA	3	109.41	101.21	114.53	1.18	1.29
N-MeFOSE	3	107.76	104.07	112.51	1.94	2.09
N-EtFOSE	3	110.39	105.49	114.25	1.64	1.81
HFPO-DA	3	105.05	87.79	119.7	0.72	0.76
ADONA	3	104.05	91.91	123.47	3.95	4.11
PFMPA	3	108.53	98.34	116.95	5.72	6.21
PFMBA	3	110.44	90.68	122.27	7.78	8.59
NFDHA	3	96.98	70.63	125.78	6.73	6.52
9Cl-PF3ONS	3	111.64	96.26	130.43	5.29	5.91
11Cl-PF3OUdS	3	110.29	93.93	127.26	3.02	3.34
PFEESA	3	100.59	94.93	106.75	2.41	2.42
3:3 FTCA	3	104.3	92.31	112.86	5.47	5.7
5:3 FTCA	3	96.42	86.49	108.3	3.27	3.16
7:3 FTCA	3	99.32	90.65	108.69	1.36	1.35

A description of each column in Table 7:

- Headings: Low, Medium, and High Spike Recovery
  - Analyte—Always 40 PFAS analytes.
  - n=—Number of samples for a matrix, for a spike concentrations, that do not have U or B flagged values. Can range from 0 to 7.
  - % Mean Recovery—Mean of the sample mean percent recoveries, for a spike concentration, for the analyte, that does not have U or B flagged values (from the sample matrix recovery tables in section D of the digital appendix, column % Recovery). If all percent recoveries are NA, (i.e., if n = 0 in or mean percent recovery < 0), then NA was used.
  - RSD—Percent RSD of the mean of the sample mean percent recoveries, used to calculate column % Recovery. If the number of samples is less than two, then NA was used.
  - SD—SD of the mean of sample mean percent recoveries. If the number of samples is less than two, then NA was used.
- Heading: ALL SPIKE RECOVERIES
  - n=—Number of spike categories with percent recovery values, without NA in column % Recovery, for the analyte.
  - % Mean Recovery—Mean of the matrix mean percent recoveries, for the analyte. If the number of spike categories is less than two, then NA was used.
  - Lowest % Recovery—Lowest percent recovery of the mean of the three subsamples' percent recoveries for the matrix for values not flagged with U or B and non-negative values from the sample matrix recovery tables in section D of the digital appendix column % Recovery.
  - Highest % Recovery—Highest percent recovery of the mean of the three subsamples' percent recoveries for the matrix for values not flagged with U or B and non-negative values from the sample matrix recovery tables in section D of the digital appendix column % Recovery.
  - RSD—Percent RSD of the matrix mean percent recoveries from columns % Recovery. If the number of samples is less than two, then NA was used.
  - SD—SD of the matrix mean percent recoveries from columns % Recovery. If the number of samples is less than two, then NA was used.

For the groundwater matrix, we calculated the mean of the mean percent recoveries, percent RSD, and SD of the percent recoveries for an analyte across all four spike concentrations (low, medium, high, *and* high 2).

Table 8 is constructed from all groundwater samples. This file is E-1 Groundwater Sample Matrix Recovery.csv in section E of the digital appendix. There is only one table like this, containing "high 2" concentration subsamples, and only for the groundwater matrix. The % Mean Recovery column is the mean of the mean percent recoveries and is potentially biased toward means with lower sample sizes since the sample means are not composed from the same size number of data. The Lowest % Recovery and Highest % Recovery columns are the mean of the three subsamples' percent recoveries for the matrix.

Table 8. Example Groundwater Matrix Recovery Table

	Low Spike Recovery				Medium Spike Recovery			
Analyte	n =	% Mean Recovery	RSD	SD	n =	% Mean Recovery	RSD	SD
PFBA	2	100.5	1.63	1.63	3	112.32	3.4	3.82
PFPeA	0	NA	NA	NA	2	115.54	3.25	3.75
PFHxA	0	NA	NA	NA	0	NA	NA	NA
PFHpA	0	NA	NA	NA	3	107.93	5.98	6.46
PFOA	0	NA	NA	NA	0	NA	NA	NA
PFNA	3	98.03	7.56	7.41	3	107.94	5	5.4
PFDA	3	103.39	8.25	8.53	3	117.09	6.09	7.13
PFUnA	3	99.85	8.42	8.4	3	112.78	4.95	5.58
PFDoA	3	95.24	12.82	12.21	3	113.65	4.98	5.66
PFTrDA	3	100.3	9.59	9.62	3	117.29	5.42	6.36
PFTeDA	3	99.89	7.2	7.19	3	115.48	4.59	5.31
PFBS	1	101.13	NA	NA	1	113.28	NA	NA
PFPeS	1	94.14	NA	NA	1	121.36	NA	NA
PFHxS	0	NA	NA	NA	0	NA	NA	NA
PFHpS	1	91.96	NA	NA	3	118.48	9.93	11.76
PFOS	0	NA	NA	NA	0	NA	NA	NA
PFNS	3	108.59	20.41	22.17	3	117.16	14.53	17.02
PFDS	3	93.64	15.28	14.3	3	106.21	11.88	12.62
PFDoS	3	88.74	14.23	12.62	3	99.14	5.56	5.51
4:2 FTS	3	98.79	10.05	9.93	3	112.46	6.05	6.8
6:2 FTS	1	108.06	NA	NA	2	125.32	5.81	7.29
8:2 FTS	2	108.9	0.91	0.99	3	128.21	3.91	5.02
PFOSA	0	NA	NA	NA	2	120.21	11.81	14.2
N-MeFOSA	3	88.07	14.57	12.83	3	108.6	6.13	6.66
N-EtFOSA	3	86.08	12.45	10.71	3	104.99	8.67	9.1
N-MeFOSAA	3	98.15	8.49	8.33	3	129.64	14.46	18.75
N-EtFOSAA	3	95.3	7.38	7.03	3	123.55	9.92	12.25
N-MeFOSE	3	96.41	9.85	9.49	3	113	3.84	4.34

Analyte	Low Spike Recovery n =	% Mean Recovery	RSD	SD	Medium Spike Recovery n =	% Mean Recovery	RSD	SD
N-EtFOSE	3	97.76	9.16	8.95	3	117.62	3.32	3.9
HFPO-DA	3	99.49	6.82	6.78	3	118.3	9.22	10.91
ADONA	3	93.84	10.9	10.23	3	110.96	8.87	9.84
PFMPA	3	102.51	6	6.15	3	115.14	4	4.6
PFMBA	3	99.75	13.71	13.68	3	112.01	3.47	3.89
NFDHA	3	98.28	14.25	14.01	3	106.93	2.01	2.15
9Cl-PF3ONS	3	96.42	15.11	14.57	3	113.13	8.39	9.5
11Cl-PF3OUdS	3	90.03	18.98	17.09	3	104.51	8.89	9.29
PFEESA	3	98.63	8.13	8.02	3	111.25	4.76	5.3
3:3 FTCA	3	99.19	6.69	6.63	3	115.21	4.41	5.09
5:3 FTCA	3	98.15	11.12	10.91	3	111.09	10.9	12.11
7:3 FTCA	3	99.22	13.75	13.64	3	113.36	12.7	14.4

Analyte	High Spike Recovery n =	% Mean Recovery	RSD	SD	High 2 Spike Recovery (groundwater only) n =	% Mean Recovery	RSD	SD
PFBA	3	108.21	4.96	5.37	3	99.67	0.43	0.43
PFPeA	3	114.82	2.19	2.51	3	104.95	1.03	1.09
PFHxA	0	NA	NA	NA	3	98.68	1.1	1.09
PFHpA	3	107.59	0.36	0.39	3	98.99	1.88	1.86
PFOA	0	NA	NA	NA	3	96.56	0.52	0.5
PFNA	3	108.46	3.56	3.87	3	98.96	0.35	0.35
PFDA	3	114.94	4.78	5.5	3	104.45	0.28	0.29
PFUnA	3	111.86	4.21	4.71	3	99.91	0.12	0.12
PFDoA	3	110.28	8.57	9.45	3	99.68	1.47	1.47
PFTrDA	3	114.66	8.85	10.14	3	93.57	3.08	2.88
PFTeDA	3	112.83	4.75	5.36	3	98.28	0.99	0.97
PFBS	1	111.97	NA	NA	3	104.02	0.43	0.45
PFPeS	2	113.27	2.48	2.81	3	93.01	1.54	1.43
PFHxS	0	NA	NA	NA	3	106.72	1.63	1.74
PFHpS	3	117.17	8.55	10.01	3	92.27	2.54	2.34
PFOS	0	NA	NA	NA	2	109.76	1.5	1.64
PFNS	3	120.44	9.47	11.41	3	99.5	4.12	4.1
PFDS	3	103.1	7.86	8.11	3	93.36	4.63	4.33
PFDoS	3	99.85	7.37	7.36	3	86.89	2.68	2.33
4:2 FTS	3	112.93	5.16	5.83	3	98.66	0.56	0.55
6:2 FTS	2	117.19	2.45	2.87	3	99.64	2.71	2.7
8:2 FTS	3	124.02	0.38	0.47	3	109.19	4.95	5.41
PFOSA	2	131.4	17.31	22.75	3	102.02	0.52	0.53
N-MeFOSA	3	110.62	6.51	7.2	3	98.07	0.03	0.03
N-EtFOSA	3	107.64	5.98	6.44	3	103.16	1.31	1.35
N-MeFOSAA	3	132.94	12.51	16.64	3	95.77	2.2	2.11

Analyte	High Spike Recovery n =	% Mean Recovery	RSD	SD	High 2 Spike Recovery (groundwater only) n =	% Mean Recovery	RSD	SD
N-EtFOSAA	3	122.3	6.86	8.39	3	96.6	1.52	1.47
N-MeFOSE	3	112.03	4.91	5.5	3	105.31	0.26	0.28
N-EtFOSE	3	115.69	5.2	6.02	3	107.79	0.9	0.97
HFPO-DA	3	114.45	7.92	9.07	3	102.94	2.41	2.48
ADONA	3	115.3	15.8	18.22	3	84.2	0.58	0.49
PFMPA	3	109.17	7.34	8.02	3	98.33	1.61	1.58
PFMBA	3	107.39	6.53	7.01	3	99.39	1.04	1.03
NFDHA	3	105.54	10.45	11.03	3	107.22	26.73	28.66
9Cl-PF3ONS	3	117.74	18.24	21.47	3	89.55	1.19	1.07
11Cl-PF3OUdS	3	107.1	15.27	16.36	3	88.09	2.79	2.46
PFEESA	3	111.46	6.25	6.96	3	100.17	2.37	2.38
3:3 FTCA	3	109.09	5.7	6.22	3	95.19	1.59	1.51
5:3 FTCA	3	108.62	9.91	10.77	3	88.59	3.28	2.91
7:3 FTCA	3	109.1	13.12	14.32	3	97.59	3.74	3.65

Analyte	ALL SPIKE RECOVERIES n =	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFBA	4	105.17	99.21	116.73	5.82	6.12
PFPeA	3	111.77	104.01	118.19	5.29	5.92
PFHxA	1	98.68	97.8	99.9	NA	NA
PFHpA	3	104.84	97.66	115.22	4.83	5.06
PFOA	1	96.56	96.05	97.05	NA	NA
PFNA	4	103.34	90.56	112.89	5.44	5.62
PFDA	4	109.97	96.23	124.71	6.41	7.05
PFUnA	4	106.1	91.09	119.07	6.78	7.19
PFDoA	4	104.71	82.26	120.89	8.29	8.68
PFTrDA	4	106.45	89.23	126.37	10.69	11.38
PFTeDA	4	106.62	91.86	121.26	8.25	8.79
PFBS	4	107.6	101.13	113.28	5.53	5.95
PFPeS	4	105.45	91.43	121.36	13.38	14.11
PFHxS	1	106.72	104.81	108.21	NA	NA
PFHpS	4	104.97	89.6	131.5	14.15	14.85
PFOS	1	109.76	108.59	110.92	NA	NA
PFNS	4	111.42	83.08	130.47	8.43	9.39
PFDS	4	99.08	78.04	115.08	6.63	6.56
PFDoS	4	93.66	74.59	105.99	7.25	6.79
4:2 FTS	4	105.71	87.74	119.66	7.63	8.07
6:2 FTS	4	112.55	97.04	130.47	9.89	11.13
8:2 FTS	4	117.58	104.19	134	8.51	10
PFOSA	3	117.88	101.57	147.49	12.58	14.83
N-MeFOSA	4	101.34	73.25	115.11	10.28	10.42

		ALL SPIKE RECOVERIES				
Analyte	n =	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
N-EtFOSA	4	100.47	73.76	113.73	9.72	9.77
N-MeFOSAA	4	114.13	90.25	151.29	17.43	19.89
N-EtFOSAA	4	109.44	87.55	137.41	14.25	15.59
N-MeFOSE	4	106.68	85.57	117.66	7.18	7.66
N-EtFOSE	4	109.72	87.64	122.13	8.23	9.03
HFPO-DA	4	108.79	92.23	125.39	8.28	9
ADONA	4	101.07	83.75	136.2	14.42	14.58
PFMPA	4	106.29	95.42	120.45	6.96	7.4
PFMBA	4	104.63	84.43	116.45	5.87	6.15
NFDHA	4	104.49	82.88	138.81	4.02	4.2
9Cl-PF3ONS	4	104.21	82.45	142.53	12.86	13.4
11Cl-PF3OUdS	4	97.43	72.26	125.93	10.02	9.76
PFEESA	4	105.38	91.34	118.04	6.58	6.93
3:3 FTCA	4	104.67	91.75	119.89	8.73	9.14
5:3 FTCA	4	101.61	85.42	118.28	10.17	10.33
7:3 FTCA	4	104.82	83.5	122.44	7.28	7.64

See Table 7 for a description of the columns for the spike concentrations Low, Medium, High Spike Recoveries, and ALL SPIKE RECOVERIES. A description of the High 2 columns in Table 8:

- Heading: High 2 Spike Recovery (groundwater only)
  - n=—Number of samples for a matrix, for a spike concentrations, that do not have U or B flagged values. Can range from 0 to 3.
  - % Recovery—Mean of the sample mean percent recoveries, for a spike concentration, for the analyte, that does not have U or B flagged values (from the sample matrix recovery tables in section D of the digital appendix, column % Recovery). If all percent recoveries are NA, (i.e., if n = 0 in or mean percent recovery < 0), then NA was used.
  - RSD—%RSD of the mean of the sample mean percent recoveries, used to calculate column % Recovery. If the number of samples is less than two, then NA was used.
  - SD—SD of the mean of sample mean percent recoveries. If the number of samples is less than two, then NA was used.

The sponsor provided a separate template with additional columns for the high 2 spike concentration for the aqueous media that we used.

Table 9 is constructed from all aqueous matrices. This is table E-9 Aqueous Sample Matrix Recovery.csv in section E of the digital appendix. The other table does not have the

high 2 spike concentrations and summarizes results across all solid media (including biosolid, sediment, and soil, but excluding tissue). The % Mean Recovery column is the mean of the mean percent recoveries and is potentially biased toward means with lower sample sizes since the sample means are not composed from the same number of data. The Lowest % Recovery and Highest % Recovery columns are the mean of the three subsamples' percent recoveries for the matrix.

Table 9. Example Media Type Recovery Table

	Low Spike Recovery				Medium Spike Recovery			
Analyte	n =	% Mean Recovery	RSD	SD	n =	% Mean Recovery	RSD	SD
PFBA	4	98.92	3.31	3.27	4	106.59	6.71	7.15
PFPeA	4	103.53	1.67	1.73	4	113.43	6.41	7.27
PFHxA	4	94.11	1.1	1.04	4	100.74	3.57	3.6
PFHpA	4	97.03	8.67	8.41	4	107.63	6.37	6.85
PFOA	4	96.79	8.5	8.22	4	102.43	4.83	4.94
PFNA	4	98.24	1.12	1.1	4	105.14	5.91	6.22
PFDA	4	101.52	5.55	5.63	4	112.12	5.42	6.07
PFUnA	4	98.04	1.52	1.49	4	108.01	6.13	6.62
PFDoA	4	98.43	4.08	4.01	4	107.93	5.88	6.34
PFTrDA	4	102.85	4.08	4.2	4	112.19	4.98	5.59
PFTeDA	4	102.52	7.71	7.91	4	110.57	6.19	6.84
PFBS	4	99.36	4.41	4.38	4	108.89	4.26	4.63
PFPeS	4	97.06	4.35	4.22	4	111.22	8.66	9.63
PFHxS	4	107.76	9.14	9.85	4	109.85	2.36	2.59
PFHpS	4	99.32	6.4	6.36	4	111.28	4.95	5.51
PFOS	4	102.18	4.2	4.3	4	109.09	11.61	12.67
PFNS	4	98.3	7.39	7.26	4	104.99	8.36	8.78
PFDS	4	83.37	9.48	7.9	4	90.79	15.54	14.11
PFDoS	4	70.96	18.92	13.43	4	72.72	31.27	22.74
4:2 FTS	4	101.06	4.9	4.95	4	109.44	5.57	6.1
6:2 FTS	4	108.94	4.63	5.04	4	121.18	8.99	10.9
8:2 FTS	4	112.04	6.67	7.48	4	122.02	4.4	5.37
PFOSA	4	118.01	16.37	19.31	4	113.8	4.93	5.62
N-MeFOSA	4	92.71	8.72	8.08	4	107.16	6.05	6.48
N-EtFOSA	4	90.97	8.84	8.05	4	105.97	4.67	4.95
N-MeFOSAA	4	136.99	19.81	27.14	4	154.1	16.96	26.14
N-EtFOSAA	4	114.65	13.94	15.98	4	133.02	9	11.98
N-MeFOSE	4	96.51	6.38	6.16	4	106.76	8.05	8.6
N-EtFOSE	4	99.82	5.22	5.21	4	112.94	6.44	7.27
HFPO-DA	4	101.23	6.05	6.13	4	110.94	8.1	8.99
ADONA	4	107.15	21.23	22.75	4	115.6	16.75	19.36
PFMPA	4	105.08	6.51	6.84	4	120.06	14	16.8
PFMBA	4	104.47	15.48	16.17	4	113.62	13.13	14.91
NFDHA	4	80.8	27.01	21.82	4	86.71	30.94	26.83
9Cl-PF3ONS	4	104.16	21.6	22.5	4	112.66	20.76	23.39
11Cl-PF3OUdS	4	88.58	15.99	14.16	4	96.76	16.59	16.05
PFEESA	4	109.99	20.41	22.45	4	121.81	25.71	31.32
3:3 FTCA	4	99.76	4.19	4.18	4	111.54	18.47	20.6
5:3 FTCA	4	115.43	26.23	30.28	4	122.79	24.84	30.5
7:3 FTCA	4	97.26	10.22	9.94	4	111.83	16.34	18.27

Analyte	High Spike Recovery n =	% Mean Recovery	RSD	SD	High 2 Spike Recovery (groundwater only) n =	% Mean Recovery	RSD	SD
PFBA	4	104.88	3.77	3.95	1	99.67	NA	NA
PFPeA	4	111.54	4.11	4.58	1	104.95	NA	NA
PFHxA	4	102.98	2.69	2.77	1	98.68	NA	NA
PFHpA	4	104.1	4.91	5.11	1	98.99	NA	NA
PFOA	4	103.11	4.37	4.5	1	96.56	NA	NA
PFNA	4	106.98	3.89	4.17	1	98.96	NA	NA
PFDA	4	110.12	4.57	5.03	1	104.45	NA	NA
PFUnA	4	108.61	4.5	4.88	1	99.91	NA	NA
PFDoA	4	109.6	6.79	7.45	1	99.68	NA	NA
PFTrDA	4	111.12	2.25	2.5	1	93.57	NA	NA
PFTeDA	4	108.62	3.93	4.27	1	98.28	NA	NA
PFBS	4	111.57	4.33	4.83	1	104.02	NA	NA
PFPeS	4	109.87	6.73	7.4	1	93.01	NA	NA
PFHxS	4	111.98	3.79	4.24	1	106.72	NA	NA
PFHpS	4	111.58	5.75	6.42	1	92.27	NA	NA
PFOS	4	111.93	7.81	8.74	1	109.76	NA	NA
PFNS	4	107.83	9.05	9.75	1	99.5	NA	NA
PFDS	4	90.49	13.61	12.32	1	93.36	NA	NA
PFDoS	4	74.41	30.04	22.35	1	86.89	NA	NA
4:2 FTS	4	108.79	3.99	4.34	1	98.66	NA	NA
6:2 FTS	4	110.82	3.98	4.41	1	99.64	NA	NA
8:2 FTS	4	122.08	3.47	4.24	1	109.19	NA	NA
PFOSA	4	120.61	8.5	10.25	1	102.02	NA	NA
N-MeFOSA	4	108.89	3.72	4.05	1	98.07	NA	NA
N-EtFOSA	4	107.3	4.04	4.34	1	103.16	NA	NA
N-MeFOSAA	4	155.06	17.89	27.73	1	95.77	NA	NA
N-EtFOSAA	4	136.2	11.89	16.2	1	96.6	NA	NA
N-MeFOSE	4	107.09	7.02	7.52	1	105.31	NA	NA
N-EtFOSE	4	111.89	5.4	6.05	1	107.79	NA	NA
HFPO-DA	4	106.59	6.02	6.41	1	102.94	NA	NA
ADONA	4	116.8	16.01	18.7	1	84.2	NA	NA
PFMPA	4	117.77	17.5	20.61	1	98.33	NA	NA
PFMBA	4	112.04	12.85	14.4	1	99.39	NA	NA
NFDHA	4	89.22	28.11	25.08	1	107.22	NA	NA
9Cl-PF3ONS	4	114.44	17.67	20.23	1	89.55	NA	NA
11Cl-PF3OUdS	4	98.02	16.06	15.74	1	88.09	NA	NA
PFEESA	4	124.2	27.49	34.14	1	100.17	NA	NA
3:3 FTCA	4	111.72	18.26	20.4	1	95.19	NA	NA
5:3 FTCA	4	120.87	25.86	31.26	1	88.59	NA	NA
7:3 FTCA	4	112.19	15.48	17.37	1	97.59	NA	NA

Analyte	ALL SPIKE RECOVERIES n =	% Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFBA	4	102.51	86.44	118.89	3.7	3.8
PFPeA	4	108.36	89.3	130.04	4.48	4.86
PFHxA	4	99.13	83.88	114.37	3.81	3.78
PFHpA	4	101.94	81.06	125.56	4.73	4.82
PFOA	4	99.72	83.56	119.23	3.54	3.53
PFNA	4	102.33	79.75	123.85	4.28	4.38
PFDA	4	107.05	78.41	124.71	4.59	4.92
PFUnA	4	103.64	86.75	119.96	5.26	5.45

Analyte	n =	ALL SPIKE RECOVERIES % Mean Recovery	Lowest % Recovery	Highest % Recovery	RSD	SD
PFDoA	4	103.91	82.26	129.86	5.46	5.67
PFTrDA	4	104.93	87.19	136.24	8.24	8.65
PFTeDA	4	105	87.2	122.51	5.37	5.64
PFBS	4	105.96	93.97	123.82	5.09	5.4
PFPeS	4	102.79	88.86	125.46	8.88	9.12
PFHxS	4	109.08	95.26	125.4	2.14	2.33
PFHpS	4	103.61	84.43	132.05	9.14	9.47
PFOS	4	108.24	91.35	123.36	3.9	4.22
PFNS	4	102.66	76.66	130.47	4.4	4.52
PFDS	4	89.5	55.96	115.08	4.79	4.29
PFDoS	4	76.25	22.91	105.99	9.49	7.24
4:2 FTS	4	104.49	87.32	122.34	5.21	5.44
6:2 FTS	4	110.15	65.44	149.19	8.02	8.84
8:2 FTS	4	116.33	100.45	139.39	5.76	6.7
PFOSA	4	113.61	88.51	178.31	7.24	8.22
N-MeFOSA	4	101.71	73.25	132.85	7.52	7.65
N-EtFOSA	4	101.85	73.76	135.08	7.32	7.46
N-MeFOSAA	4	135.48	85.62	411.9	20.48	27.74
N-EtFOSAA	4	120.12	54.83	260.82	15.26	18.33
N-MeFOSE	4	103.92	76.32	123.75	4.81	5
N-EtFOSE	4	108.11	85.23	135.86	5.51	5.95
HFPO-DA	4	105.42	83.3	125.39	4.08	4.3
ADONA	4	105.94	82.04	196.71	14.27	15.11
PFMPA	4	110.31	64.16	197.46	9.38	10.35
PFMBA	4	107.38	84.43	166.11	6.2	6.66
NFDHA	4	90.99	17.3	139.28	12.51	11.38
9Cl-PF3ONS	4	105.2	49.8	200.22	10.8	11.36
11Cl-PF3OUdS	4	92.86	30.92	141.74	5.66	5.26
PFEESA	4	114.04	85.34	274.32	9.77	11.14
3:3 FTCA	4	104.55	23.76	210.24	8.02	8.38
5:3 FTCA	4	111.92	68.65	219.25	14.17	15.86
7:3 FTCA	4	104.72	60.18	167.59	8.05	8.42

A description of each column in Table 9:

- Headings: Low, Medium, High, and High 2 Spike Recovery
  - Analyte—Always 40 PFAS analytes.
  - n=—Number of matrices for a media type, for spike concentrations, that do not have U or B flagged values. Can be 4 or 1.
  - % Mean Recovery—Mean of the matrix mean percent recoveries, for a spike concentration, for the analyte (from the media type matrix recovery tables in section E of the digital appendix, column % Recovery). If all percent recoveries are NA, (i.e., if n = 0 or mean percent recovery < 0), NA was used.
  - RSD—Percent RSD of the mean of the matrix mean percent recoveries, used to calculate column % Recovery. If the number of samples is less than two, NA was used.
  - SD—SD of the mean of sample mean percent recoveries. If the number of samples is less than two, NA was used.
- Heading: ALL SPIKE RECOVRIES
  - n=—Number of spike categories with percent recovery values, without NA in column % Recovery, for the analyte.
  - % Mean Recovery—Mean of the mean percent recoveries for the media type, for the analyte. If the number of spike categories is less than two, NA was used.
  - Lowest % Recovery—Lowest percent recovery of the mean of the three subsamples' percent recoveries for the matrix for values without U or B flags and non-negative values from the sample matrix recovery tables in section D of the digital appendix, column % Recovery.
  - Highest % Recovery—Highest percent recovery of the mean of the three subsamples' percent recoveries for the matrix for values without U or B flags and non-negative values from the sample matrix recovery tables in section D of the digital appendix, column % Recovery.
  - RSD—Percent RSD of the mean percent recoveries for the media from columns % Recovery. If the number of samples is less than two, NA was used.
  - SD—SD of the mean percent recoveries for the media from columns % Recovery. If the number of samples is less than two, NA was used.

F. Extracted Internal Standard (EIS) Spike Recovery Tables

We generated a total of 11 EIS spike recovery tables, which are section F of the digital appendix. EIS spike recovery values indicate the extraction efficiency of the sample preparation and detection method for PFAS compounds. We calculated the mean sample concentration, mean of the sample percent recoveries, and percent RSD for 24 isotopically labeled analog PFAS analytes as the EIS compounds, for the 32 environmental samples. At the sponsor's direction, we used the sample percent recoveries values that were already calculated in the EDD. We then calculated the mean of the sample mean percent recoveries, percent RSD, and SD of the sample mean percent recoveries for an analyte, for the matrix. We also identified the lowest and highest sample mean percent recovery for the matrix.

We then calculated the mean of the matrix mean concentrations; mean of the matrix mean percent recoveries; and percent RSD, SD, and 2×SD of the matrix mean percent recoveries for the 24 EIS compounds, for all aqueous matrices, all solid matrices (including biosolid, sediment, and soil), and all solid matrices and tissue. We identified the lowest and highest sample mean percent recovery for the media type.

Table 10 is constructed from all tissue samples. This table is file F-8 Tissue Sample EIS Spike Recovery Table.csv in section F of the digital appendix. There are eight tables like this, one for each of the eight sample matrices. The Mean Percent Recovery columns for each sample is the mean of the percent recovery entries in the EDD. The Mean Percent Recovery column for all concentrations is the mean of the sample percent recoveries.

Table 10. Example Matrix EIS Spike Recovery Table

Compound	Clam Tissue				Fish Tissue # 2			
	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD
Perfluoro-n-[13C4]butanoic acid	20.1	12	98.52	2.44	20.1	12	93.36	14.03
Perfluoro-n-[13C5]pentanoic acid	10	12	93.84	6.09	10	12	107.34	19.46
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	5.01	12	94.88	3.65	5.02	12	92.31	15.7
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	5.01	12	93.01	2.09	5.02	12	82.66	19.48
Perfluoro-n-[13C8]octanoic acid	5.06	12	94.72	3.45	5.07	12	90.46	14.9
Perfluoro-n-[13C9]nonanoic acid	2.51	12	92.12	4.07	2.51	12	90.3	15.68
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	2.51	12	83.28	6.06	2.51	12	90.68	15.39
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	2.51	12	71.19	10	2.51	12	88.58	13.08
Perfluoro-n-[1,2-13C2]dodecanoic acid	2.51	12	53.52	14.64	2.51	12	70.06	18.56
Perfluoro-n-[1,2-13C2]tetradecanoic acid	2.51	12	31.36	21.81	2.51	12	38.36	75.43
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	5.01	12	97.87	3.29	5.02	12	88.83	19.85
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	5.06	12	98.39	2.18	5.07	12	98.64	20.1
Perfluoro-1-[13C8]octanesulfonic acid	5.06	12	96.11	3.99	5.07	12	91.9	15.79
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	10.1	12	192.33	8.87	10.1	12	214.83	17.31
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	10	12	145.25	13.18	10	12	149.49	20.1
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	10	12	164.92	27	10	12	136.17	25.55
Perfluoro-1-[13C8]octanesulfonamide	5.01	12	95.52	6.15	5.02	12	87.44	15.31
N-methyl-d3-perfluoro-1-octanesulfonamide	5.01	12	37.65	18.52	5.02	12	26.8	33.2
N-ethyl-d5-perfluoro-1-octanesulfonamide	5.01	12	30.11	27.83	5.02	12	21.64	36.58
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	10	12	125.83	10.5	10	12	138.53	15.91
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	10	12	141.25	11.5	10	12	151.09	14.23
N-methyl-d7-perfluorooctanesulfonamidoethanol	50.1	12	30.27	44.94	50.2	12	12.32	68.11
N-ethyl-d9-perfluorooctane sulfonamidoethanol	50.1	12	29.23	31.33	50.2	12	12.55	58.71
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	20.1	12	92.52	11.8	20.1	12	101.9	17.44

Compound	Fish Tissue #1				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD
Perfluoro-n-[13C4]butanoic acid	20.1	12	84.01	23.09	20.1	3	91.96	8
Perfluoro-n-[13C5]pentanoic acid	10.1	12	86.44	14.08	10	3	95.88	11.05
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	5.02	12	94.94	6.77	5.02	3	94.04	1.6
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	5.02	12	79.69	6.35	5.02	3	85.12	8.21

Compound	Fish Tissue #1				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD
Perfluoro-n-[13C8]octanoic acid	5.07	12	95.25	4.72	5.07	3	93.48	2.81
Perfluoro-n-[13C9]nonanoic acid	2.51	12	98.02	3.67	2.51	3	93.48	4.32
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	2.51	12	97.19	4.18	2.51	3	90.39	7.7
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	2.51	12	90.94	4.83	2.51	3	83.57	12.9
Perfluoro-n-[1,2-13C2]dodecanoic acid	2.51	12	95.83	8.81	2.51	3	73.14	29.15
Perfluoro-n-[1,2-13C2]tetradecanoic acid	2.51	12	101.68	13.3	2.51	3	57.13	67.81
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	5.02	12	90.92	5.84	5.02	3	92.54	5.12
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	5.07	12	98.46	4.93	5.07	3	98.5	0.13
Perfluoro-1-[13C8]octanesulfonic acid	5.07	12	103.37	2.63	5.07	3	97.12	5.97
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	10.1	12	194.37	17.99	10.1	3	200.51	6.21
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	10.1	12	229.75	5.98	10	3	174.83	27.23
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	10.1	12	220.25	9.59	10	3	173.78	24.59
Perfluoro-1-[13C8]octanesulfonamide	5.02	12	92.63	6.9	5.02	3	91.87	4.46
N-methyl-d3-perfluoro-1-octanesulfonamide	5.02	12	8.08	16.75	5.02	3	24.18	61.86
N-ethyl-d5-perfluoro-1-octanesulfonamide	5.02	12	7.51	18.7	5.02	3	19.75	57.81
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	10.1	12	106.41	7.62	10	3	123.59	13.09
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	10.1	12	78.52	8.08	10	3	123.62	31.84
N-methyl-d7-perfluorooctanesulfonamidoethanol	50.2	12	5.2	23.74	50.2	3	15.93	81.09
N-ethyl-d9-perfluorooctane sulfonamidoethanol	50.2	12	0.37	63.6	50.2	3	14.05	103.11
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	20.1	12	94.49	7.44	20.1	3	96.31	5.13

A description of each column in Table 10:

- Heading: Sample ID
  - Compound—Always 24 EIS compounds.
  - Mean Concentration—Mean spike concentration of the EDD entries for a compound from a sample of a matrix in µg/kg (solid matrices) or ng/L (aqueous matrices).
  - Number EIS Spikes (n=)—Number of EDD entries for the compound from a sample of a matrix. Can be 12 or 16.
  - Mean Percent Recovery—Mean of the percent recovery entries in the EDD for a compound from a sample of a matrix.
  - RSD—Percent RSD of the mean percent recoveries reported in the EDD. If the number of samples is less than two, then NA was used.
- Heading: ALL EIS SPIKE RECOVERIES
  - Mean Concentration—Mean of the sample mean spike concentrations from columns Mean Concentration.
  - Number EIS Spikes (n=)—Number of samples for a matrix, for the compound. Always three.
  - Mean Percent Recovery—Mean of the sample mean percent recoveries from columns Mean Percent Recovery.
  - RSD—Percent RSD of the sample mean percent recoveries reported in the EDD. If the number of samples is less than two, then NA was used.

Table 11 summarizes EIS results across all soil, sediment and biosolids matrices (excluding tissue). This table is file F-10 Soil, Sediment and Biosolids EIS Spike Recovery Table.csv in section F of the digital appendix. There are three tables like this, summarizing results across all aqueous samples, most solid samples (including biosolid, sediment, and soil, but excluding tissue), and all solid samples (including biosolid, sediment, soil, and tissue). The Mean Percent Recovery column is the mean of the mean percent recoveries. Highest % Recovery and Lowest % Recovery columns are the highest and lowest mean of the sample percent recoveries.

Table 11. Example Media Type EIS Spike Recovery Table

Compound	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	Highest % Recovery	Lowest % Recovery	RSD	SD	2× SD
Perfluoro-n-[13C4]butanoic acid	142	4	81.9	97.21	8.99	15.9	13.02	26.05
Perfluoro-n-[13C5]pentanoic acid	70.8	4	87.89	103.4	38.88	13.34	11.73	23.45
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	35.4	4	91.19	96.9	72.87	2.7	2.46	4.92
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	35.4	4	89.89	94.73	76.61	2.39	2.15	4.3
Perfluoro-n-[13C8]octanoic acid	35.8	4	92.35	95.05	87.27	0.81	0.75	1.49
Perfluoro-n-[13C9]nonanoic acid	17.7	4	91.22	95.19	82.37	1.59	1.45	2.89
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	17.7	4	87.48	92.71	70.56	3.31	2.9	5.79
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	17.7	4	85.66	94.49	56.25	6.46	5.53	11.06
Perfluoro-n-[1,2-13C2]dodecanoic acid	17.7	4	72.27	86.74	33.68	13.68	9.88	19.77
Perfluoro-n-[1,2-13C2]tetradecanoic acid	17.7	4	64.04	153.44	16.96	26.17	16.76	33.51
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	35.4	4	93.55	99.66	72.28	4.7	4.39	8.79
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	35.8	4	92.05	94.78	79.44	1.57	1.45	2.89
Perfluoro-1-[13C8]octanesulfonic acid	35.8	4	91.33	96.04	66.75	3.58	3.27	6.55
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	71.2	4	124.2	199	80.57	14.82	18.41	36.82
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	70.8	4	105.29	182.5	64.02	16.36	17.22	34.45
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	70.8	4	93.4	139.33	65.08	8.38	7.83	15.66
Perfluoro-1-[13C8]octanesulfonamide	35.4	4	77.6	92.62	26.68	15.42	11.97	23.94
N-methyl-d3-perfluoro-1-octanesulfonamide	35.4	4	59.82	74.05	13.51	16.42	9.82	19.64
N-ethyl-d5-perfluoro-1-octanesulfonamide	35.4	4	58.44	70.18	11.93	16.53	9.66	19.31
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	70.8	4	87.42	113.46	21.46	7.32	6.4	12.8
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	70.8	4	85.78	105.67	11.68	8.21	7.04	14.09
N-methyl-d7-perfluorooctanesulfonamidoethanol	354	4	61.92	77.01	10.99	18.55	11.48	22.97
N-ethyl-d9-perfluorooctane sulfonamidoethanol	354	4	57.63	72.61	7.99	19.62	11.31	22.61
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	142	4	101.07	112.5	91.83	2.02	2.04	4.08

A description of each column in Table 11:

- Compound—Always 24 EIS compounds.
- Mean Concentration—Mean of the matrix mean spike concentration (from Appendix F Matrix Tables, under ALL EIS SPIKE RECOVERIES, Column Mean Concentration) for solid matrices.
- Number EIS Spikes (n=)—Number of matrices for all media types. Always four.
- Mean Percent Recovery—Mean of the matrix mean percent recovery (from Appendix F Matrix Tables, under ALL EIS SPIKE RECOVERIES, Column Mean Percent Recovery) for solid media.
- Highest % Recovery—Highest percent recovery of the mean of the sample percent recoveries (from EIS spike recovery tables in section F of the digital appendix, under Sample IDs, column Mean Percent Recovery) for solid media.
- Lowest % Recovery—Lowest percent recovery of the mean of the sample percent recoveries (from EIS spike recovery tables in section F of the digital appendix, under Sample IDs, column Mean Percent Recovery) for solid media.
- RSD—Percent RSD of the matrix mean percent recoveries. If the number of samples is less than two, then NA was used.
- SD—SD of the matrix mean percent recoveries. If the number of samples is less than two, then NA was used.
- 2× SD—SD of the matrix mean percent recoveries times two. If the number of samples is less than two, then NA was used.

G. Injected Internal Standard (IIS) Spike Recovery Tables

We generated a total of 11 IIS spike recovery tables, which are section G of the digital appendix. IIS spike recovery tables indicate the extraction efficiency of the SLV method for isotopically labeled compounds. We calculated the mean of the sample percent recoveries, percent RSD, SD, and 2× SD for 7 isotopically labeled analog PFAS analytes, for the 32 environmental samples. Upon the sponsor's direction, we used the sample percent recoveries values in the EDD.

We then calculated the mean of the matrix mean percent recoveries; percent RSD, SD, and 2× SD of the matrix mean percent recoveries for the seven IIS compounds, for aqueous, solid, and all media.

Table 12 is constructed from all wastewater samples. This table is file G-3 Wastewater Sample IIS Spike Recovery Table.csv in section G of the digital appendix. There are eight tables like this, one for each environmental matrix.

Table 12. Example Matrix IIS Spike Recovery Table

Media	PFAS	n	Mean % Recovery	RSD	SD	2× SD
Wastewater	13C3-PFBA	105	81.56	14.46	11.8	23.59
Wastewater	13C2-PFHXA	105	73.13	14.98	10.96	21.91
Wastewater	13C4-PFOA	105	77.06	13.52	10.42	20.84
Wastewater	13C5-PFNA	105	78.95	13.83	10.92	21.84
Wastewater	13C2-PFDA	105	64.52	34.85	22.48	44.97
Wastewater	18O2-PFHXS	105	76.85	14.39	11.06	22.12
Wastewater	13C4-PFOS	105	68.87	16.55	11.4	22.79

Note: Percent recovery values used in the Mean % Recovery column were reported in the EDD and not calculated by IDA using Equation 1.

A description of each column in Table 12:

- Media—Media type. Eight possible matrices
- PFAS—IIS Compound. Always seven.
- n=—Number of subsamples in the EDD for a matrix type with numeric values. Entries represented as N/A are not counted.
- Mean % Recovery—Mean of the subsample percent recovery, for a compound, for a matrix.
- RDS—Percent RSD of the mean of the sample percent recoveries.
- SD—SD of the sample percent recoveries.
- 2× SD—SD of the sample percent recoveries times two.

Table 13 summarizes these results across all aqueous matrices. This table is file G-9 Aqueous Sample IIS Spike Recovery Table.csv in section G of the digital appendix. There are two tables like this, one for all solid media and one for all aqueous media. The Mean Percent Recovery column is the mean of the mean percent recoveries.

Table 13. Example Media Type IIS Spike Recovery Table

Media	PFAS	n =	Mean % Recovery	RSD	SD	2× SD
Aqueous	13C3-PFBA	4	75.77	10.25	7.77	15.53
Aqueous	13C2-PFHXA	4	68.3	18.6	12.7	25.41
Aqueous	13C4-PFOA	4	72.95	9.68	7.07	14.13
Aqueous	13C5-PFNA	4	75.36	7.47	5.63	11.25
Aqueous	13C2-PFDA	4	71.75	9.96	7.15	14.3
Aqueous	18O2-PFHXS	4	73.24	9.58	7.02	14.03
Aqueous	13C4-PFOS	4	71.02	7.52	5.34	10.67

A description of each column in Table 13:

- Media—Media type. Two possible media types.
- PFAS—IIS Compound. Always seven.
- n=—Number of matrices for a media type. Always four.
- Mean % Recovery—Mean of the matrix mean percent recoveries, for a compound, for a media type.
- RDS—Percent RSD of the mean of the matrix mean percent recoveries.
- SD—SD of the matrix mean percent recoveries.
- 2× SD—SD of the matrix mean percent recoveries times two.

Table 14 summarizes results across all matrices. This table is file G-11 All Media Sample IIS Spike Recovery Table.csv in section G of the digital appendix. This is the only table of this type, summarizing results across *all* environmental media. The Mean % Recovery column is the mean of the mean percent recoveries and is potentially biased toward means with lower sample sizes since the sample means are not composed from the same size.

Table 14. Example All Media IIS Spike Recovery Table

Media	PFAS	n =	Mean % Recovery	RSD	SD	2× SD
All	13C3-PFBA	8	71.73	9.62	6.9	13.8
All	13C2-PFHXA	8	65.99	13.81	9.11	18.22
All	13C4-PFOA	8	70.34	8.11	5.7	11.41
All	13C5-PFNA	8	72.62	7.12	5.17	10.34
All	13C2-PFDA	8	70.06	7.96	5.58	11.15
All	18O2-PFHXS	8	70.35	8.47	5.96	11.91
All	13C4-PFOS	8	69.29	6.31	4.37	8.74

A description of each column in Table 14:

- Media—Media type. All.
- PFAS—IIS Compound. Always seven.
- n=—Number of matrices for a media type. Always eight.
- Mean % Recovery—Mean of the matrix mean percent recoveries, for a compound, for all media.
- RSD—Percent RSD of the mean of the matrix mean percent recoveries.
- SD—SD of the matrix mean percent recoveries.
- 2× SD—SD of the matrix mean percent recoveries times two.

H. Ongoing Precision and Recovery (OPR) Spike Recovery Tables

We generated a total of four OPR spike recovery tables, which is section H of the digital appendix. OPR indicates the performance of the sample preparation and detection method at a mid-range spike concentration in the study as a quality control check. We calculated the mean of the OPR subsample percent recoveries and percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes, from the 32 samples, for aqueous media and solid media (including biosolid, sediment, and soil, but excluding tissue). Separately, at the sponsor's direction, we also calculated the mean of the OPR subsample percent recoveries, percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes from the tissue matrix. We used the sample percent recoveries values in the EDD and did not include data that had B flagged values (where the results was associated with a contaminated blank in our calculations).

We then calculated the mean of the OPR matrix mean percent recoveries and percent RSD, SD, 2× SD, and 3× SD of the matrix mean percent recoveries for the 64 analytes, from all media (i.e., aqueous, solid, and tissue).

Table 15 is constructed from tissue samples. This table is file H-3 Tissue Sample OPR Spike Recovery Table.csv in section H of the digital appendix. There are three tables like this, one for aqueous, one for solid (excluding tissue), and one for tissue media. The % Mean Recovery column is the mean of the mean percent recoveries.

Table 15. Example Media Type OPR Spike Recovery Table

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluorobutanoic acid	4	99.88	5.12	5.11	10.23	15.34
Perfluoropentanoic acid	4	104.78	4.33	4.53	9.06	13.59
Perfluorohexanoic acid	4	100.62	5.19	5.22	10.44	15.65
Perfluoroheptanoic acid	4	102.53	7.78	7.97	15.95	23.92
Perfluorooctanoic acid	4	97.7	8.19	8	15.99	23.99
Perfluorononanoic acid	4	102.7	7.74	7.95	15.9	23.85
Perfluorodecanoic acid	4	98.35	7.14	7.02	14.04	21.05
Perfluoroundecanoic acid	4	103.53	6.29	6.51	13.03	19.54
Perfluorododecanoic acid	4	108.97	14.72	16.04	32.08	48.12
Perfluorotridecanoic acid	4	119.75	5.72	6.85	13.7	20.55
Perfluorotetradecanoic acid	4	100.9	5.14	5.19	10.38	15.57
Perfluorobutanesulfonic acid	4	102.88	6.63	6.82	13.64	20.45
Perfluoropentanesulfonic acid	4	100.8	5.8	5.85	11.69	17.54
Perfluorohexanesulfonic acid	4	106.92	7.64	8.17	16.33	24.5
Perfluoroheptanesulfonic acid	4	97.05	5.88	5.7	11.41	17.11
Perfluorooctanesulfonic acid	4	110.75	6.18	6.85	13.7	20.55
Perfluorononanesulfonic acid	4	99.5	7.08	7.05	14.09	21.14
Perfluorodecanesulfonic acid	4	93.9	8.67	8.14	16.28	24.41
Perfluorododecanesulfonic acid	4	68.43	28.97	19.82	39.64	59.46

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
4:2 fluorotelomersulfonic acid	4	96.4	3.36	3.24	6.48	9.72
6:2 fluorotelomersulfonic acid	4	105.35	6.46	6.81	13.62	20.43
8:2 fluorotelomersulfonic acid	4	118.75	7.09	8.42	16.84	25.26
Perfluorooctanesulfonamide	4	108.95	5.75	6.26	12.52	18.79
N-methyl perfluorooctanesulfonamide	4	101.55	7.52	7.63	15.27	22.9
N-ethyl perfluorooctanesulfonamide	4	108.75	8.51	9.25	18.5	27.75
N-methyl perfluorooctanesulfonamidoacetic acid	4	104.72	5.67	5.94	11.87	17.81
N-ethyl perfluorooctanesulfonamidoacetic acid	4	103.58	6.69	6.93	13.85	20.78
N-methyl perfluorooctanesulfonamidoethanol	4	231.25	24.47	56.58	113.15	169.73
N-ethyl perfluorooctanesulfonamidoethanol	4	109.9	22.29	24.5	48.99	73.49
Hexafluoropropylene oxide dimer acid	4	100.03	6.92	6.92	13.83	20.75
4,8-dioxa-3H-perfluorononanoic acid	4	109.03	10.69	11.65	23.31	34.96
Perfluoro-3-methoxypropanoic acid	4	97.6	5.97	5.83	11.65	17.48
Perfluoro-4-methoxybutanoic acid	4	100.45	8.12	8.15	16.31	24.46
Perfluoro-3,6-dioxaheptanoic acid	4	85.7	17.17	14.72	29.43	44.15
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	4	110.25	7.08	7.8	15.61	23.41
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	4	116.25	9.54	11.09	22.17	33.26
Perfluoro(2-ethoxyethane)sulfonic acid	4	97.38	4.91	4.78	9.56	14.35
2H, 2H, 3H, 3H-perfluorohexanoic acid	4	83.28	25.45	21.2	42.39	63.59
2H, 2H, 3H, 3H-perfluorooctanoic acid	4	138.53	21.91	30.35	60.69	91.04
2H, 2H, 3H, 3H-perfluorodecanoic acid	4	119	8.26	9.83	19.66	29.5
Perfluoro-n-[13C4]butanoic acid	4	100	2.38	2.38	4.76	7.14
Perfluoro-n-[13C5]pentanoic acid	4	95.97	3.82	3.66	7.33	10.99
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	4	93.12	2.68	2.49	4.99	7.48
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	4	90.95	6.21	5.65	11.3	16.96
Perfluoro-n-[13C8]octanoic acid	4	93.78	4.37	4.1	8.19	12.29
Perfluoro-n-[13C9]nonanoic acid	4	95.22	3.03	2.88	5.77	8.65
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	4	96.88	3.64	3.52	7.05	10.57
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	4	98.75	5.29	5.23	10.46	15.69
Perfluoro-n-[1,2-13C2]dodecanoic acid	4	88.95	10.61	9.43	18.87	28.3
Perfluoro-n-[1,2-13C2]tetradecanoic acid	4	59.85	41.55	24.87	49.73	74.6
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	4	100.15	2.74	2.75	5.5	8.25
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	4	96.7	3.15	3.05	6.09	9.14
Perfluoro-1-[13C8]octanesulfonic acid	4	98.95	1.86	1.84	3.68	5.51
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	4	223.25	15.28	34.12	68.24	102.36
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	4	132.75	6.04	8.02	16.03	24.05
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	4	191.75	29.3	56.18	112.36	168.54
Perfluoro-1-[13C8]octanesulfonamide	4	103.8	7.76	8.06	16.12	24.18
N-methyl-d3-perfluoro-1-octanesulfonamide	4	18.32	42.76	7.84	15.67	23.51

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
N-ethyl-d5-perfluoro-1-octanesulfonamide	4	26.4	55.67	14.7	29.39	44.09
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	4	169.75	7.7	13.07	26.15	39.22
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	4	199	5.99	11.92	23.83	35.75
N-methyl-d7-perfluorooctanesulfonamidoethanol	4	2.56	103.07	2.64	5.27	7.91
N-ethyl-d9-perfluorooctane sulfonamidoethanol	4	14.07	65.76	9.26	18.51	27.77
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	4	93.22	6.68	6.23	12.46	18.69

Note: Percent recovery values used in the %Mean Recovery column were reported in the EDD and not calculated by IDA using Equation 1.

A description of each column in Table 15:

- Analyte—Always 64 PFAS analytes.
- n=—Number of OPR subsamples that do not have B flagged values for the analyte in the EDD, for the media type (e.g., aqueous, solid (biosolid, soil, sediment), tissue).
- % Mean Recovery—Mean of the OPR subsample percent recoveries of the EDD entries.
- RSD—Percent RSD of the mean of the sample percent recoveries.
- 1× SD—SD of the sample mean percent recoveries.
- 2× SD—SD of the sample mean percent recoveries times two.
- 3× SD—SD of the sample mean percent recoveries times three.

Table 16 is constructed from all matrices. This table is file H-4 All Sample OPR Spike Recovery Table.csv in section H of the digital appendix. There is only one table of this type, summarizing results across *all* media. The % Mean Recovery column is the mean of the mean percent recoveries and is potentially biased toward means with lower sample sizes since the sample means are not composed from the same number of data.

Table 16. Example All Media OPR Spike Recovery Table

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluorobutanoic acid	3	100.35	0.52	0.52	1.05	1.57
Perfluoropentanoic acid	3	104.94	0.39	0.41	0.82	1.23
Perfluorohexanoic acid	3	99.36	1.53	1.52	3.04	4.57
Perfluoroheptanoic acid	3	100.1	2.33	2.34	4.67	7.01
Perfluorooctanoic acid	3	98.36	0.99	0.97	1.95	2.92
Perfluorononanoic acid	3	100.95	1.54	1.56	3.12	4.68

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluorodecanoic acid	3	101.77	2.91	2.96	5.92	8.88
Perfluoroundecanoic acid	3	101.8	1.5	1.53	3.06	4.6
Perfluorododecanoic acid	3	105.19	3.12	3.28	6.56	9.85
Perfluorotridecanoic acid	3	110.7	7.1	7.86	15.73	23.59
Perfluorotetradecanoic acid	3	101.43	1.01	1.03	2.06	3.08
Perfluorobutanesulfonic acid	3	101.81	0.94	0.95	1.91	2.86
Perfluoropentanesulfonic acid	3	100.61	0.22	0.22	0.44	0.66
Perfluorohexanesulfonic acid	3	106.72	1.68	1.79	3.58	5.37
Perfluoroheptanesulfonic acid	3	97.73	2.08	2.03	4.06	6.09
Perfluorooctanesulfonic acid	3	106.84	3.21	3.43	6.86	10.29
Perfluorononanesulfonic acid	3	100.2	3.85	3.86	7.71	11.57
Perfluorodecanesulfonic acid	3	94.88	0.91	0.86	1.72	2.58
Perfluorododecanesulfonic acid	3	82.25	14.61	12.02	24.03	36.05
4:2 fluorotelomersulfonic acid	3	100.54	4.42	4.44	8.89	13.33
6:2 fluorotelomersulfonic acid	3	107.82	4.27	4.6	9.2	13.8
8:2 fluorotelomersulfonic acid	3	115.27	3.2	3.69	7.37	11.06
Perfluorooctanesulfonamide	3	106.43	2.4	2.55	5.1	7.65
N-methyl perfluorooctanesulfonamide	3	101.04	3.11	3.15	6.29	9.44
N-ethyl perfluorooctanesulfonamide	3	103.42	6.47	6.69	13.39	20.08
N-methyl perfluorooctanesulfonamidoacetic acid	3	102.24	2.16	2.21	4.41	6.62
N-ethyl perfluorooctanesulfonamidoacetic acid	3	103.31	1.11	1.15	2.3	3.44
N-methyl perfluorooctanesulfonamidoethanol	3	145.87	50.69	73.94	147.89	221.83
N-ethyl perfluorooctanesulfonamidoethanol	3	106.76	2.57	2.75	5.5	8.25
Hexafluoropropylene oxide dimer acid	3	100.25	0.52	0.52	1.03	1.55
4,8-dioxa-3H-perfluorononanoic acid	3	101.96	6.15	6.27	12.54	18.81
Perfluoro-3-methoxypropanoic acid	3	99.88	1.99	1.99	3.98	5.98
Perfluoro-4-methoxybutanoic acid	3	100.36	2.51	2.52	5.03	7.55
Perfluoro-3,6-dioxaheptanoic acid	3	93.48	7.21	6.74	13.47	20.21
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	3	105.16	4.83	5.08	10.16	15.24
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	3	104.66	9.99	10.45	20.91	31.36
Perfluoro(2-ethoxyethane)sulfonic acid	3	98.73	1.43	1.42	2.83	4.25
2H, 2H, 3H, 3H-perfluorohexanoic acid	3	92.04	8.25	7.6	15.19	22.79
2H, 2H, 3H, 3H-perfluorooctanoic acid	3	109.04	23.68	25.82	51.65	77.47
2H, 2H, 3H, 3H-perfluorodecanoic acid	3	102.43	14.72	15.08	30.15	45.23
Perfluoro-n-[13C4]butanoic acid	3	100.04	2.16	2.16	4.32	6.48
Perfluoro-n-[13C5]pentanoic acid	3	96.3	1.31	1.26	2.52	3.79
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	3	95.81	2.95	2.83	5.66	8.49
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	3	93.44	2.32	2.17	4.34	6.51
Perfluoro-n-[13C8]octanoic acid	3	95.86	2.27	2.18	4.36	6.54
Perfluoro-n-[13C9]nonanoic acid	3	96.34	1.75	1.69	3.38	5.06
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	3	96.45	1.44	1.39	2.78	4.17

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	3	99.56	3.48	3.47	6.94	10.4
Perfluoro-n-[1,2-13C2]dodecanoic acid	3	88.47	1.62	1.44	2.87	4.31
Perfluoro-n-[1,2-13C2]tetradecanoic acid	3	78.5	20.88	16.39	32.78	49.18
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	3	100.64	1.84	1.85	3.71	5.56
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	3	96.66	2.63	2.54	5.09	7.63
Perfluoro-1-[13C8]octanesulfonic acid	3	99.51	1.96	1.95	3.9	5.85
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	3	156.51	37.62	58.87	117.74	176.61
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	3	120.49	10.4	12.53	25.06	37.59
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	3	134.62	36.79	49.53	99.06	148.58
Perfluoro-1-[13C8]octanesulfonamide	3	89.69	13.62	12.22	24.44	36.66
N-methyl-d3-perfluoro-1-octanesulfonamide	3	41.4	52.63	21.79	43.58	65.37
N-ethyl-d5-perfluoro-1-octanesulfonamide	3	42.63	44.2	18.84	37.68	56.52
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	3	119.71	36.29	43.45	86.89	130.34
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	3	128.34	47.77	61.31	122.62	183.93
N-methyl-d7-perfluorooctanesulfonamidoethanol	3	42.84	83.27	35.67	71.35	107.02
N-ethyl-d9-perfluorooctane sulfonamidoethanol	3	44.33	61.49	27.26	54.51	81.77
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	3	100.5	6.27	6.3	12.61	18.91

A description of each column in Table 16:

- Analyte—Always 64 PFAS analytes.
- n=—Number of matrices for the analyte in the EDD, for aqueous, solid (biosolid, sediment, and soil), and tissue media.
- % Mean Recovery—Mean of the OPR matrix mean percent recoveries, for aqueous, solid (biosolid, sediment, and soil), and tissue media.
- RSD—Percent RSD of the mean of the sample percent recoveries.
- 1× SD—SD of the matrix mean percent recoveries.
- 2× SD—SD of the matrix mean percent recoveries times two.
- 3× SD—SD of the matrix mean percent recoveries times three.

I. Limit of Quantitation Verification (LOQVER) Spike Recovery Tables

We generated a total of four LOQVER spike recovery tables, which are section I of the digital appendix. LOQVER indicates the performance of the sample preparation and

detection method at a lowest spike concentration in the study as a quality control check. We calculated the mean of the LOQVER subsample percent recoveries and percent RSD, SD, 2× SD, and 3× SD for 64 analytes, from the 32 samples, for aqueous media and solid media (including biosolid, sediment, and soil, but excluding tissue). At the sponsor's direction, we also calculated the mean of the LOQVER subsample percent recoveries and percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes, for the tissue matrix. At the sponsor's direction, we used the sample percent recoveries values in the EDD.

We then calculated the mean of the LOQVER matrix mean percent recoveries and percent RSD, SD, 2× SD and 3× SD of the matrix mean percent recoveries for the 64 analytes, for all media (i.e., aqueous, solid, and tissue).

Table 17 is constructed from solid samples. This table is file I-2 Solid Sample LOQVER Spike Recovery Table.csv in section I of the digital appendix. There are three tables like this, summarizing results for aqueous, solid (excluding tissue), and tissue media.

Table 17. Example Media Type LOQVER Spike Recovery Table

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluorobutanoic acid	17	100.96	7.2	7.27	14.53	21.8
Perfluoropentanoic acid	17	100.44	9.77	9.81	19.63	29.44
Perfluorohexanoic acid	17	97.24	6.62	6.43	12.87	19.3
Perfluoroheptanoic acid	17	97.36	6.79	6.61	13.22	19.83
Perfluorooctanoic acid	17	98.34	8.04	7.9	15.8	23.71
Perfluorononanoic acid	17	98.82	5.64	5.58	11.16	16.73
Perfluorodecanoic acid	17	100.06	7.04	7.04	14.09	21.13
Perfluoroundecanoic acid	17	97.56	7.13	6.95	13.91	20.86
Perfluorododecanoic acid	17	98.55	7.82	7.71	15.42	23.13
Perfluorotridecanoic acid	17	102.25	7.34	7.51	15.02	22.53
Perfluorotetradecanoic acid	17	100.28	6.75	6.77	13.55	20.32
Perfluorobutanesulfonic acid	17	100.66	7.12	7.17	14.34	21.52
Perfluoropentanesulfonic acid	17	97.71	8.34	8.15	16.3	24.45
Perfluorohexanesulfonic acid	17	103.94	7.67	7.97	15.94	23.91
Perfluoroheptanesulfonic acid	17	93.24	6.52	6.08	12.16	18.24
Perfluorooctanesulfonic acid	17	100.27	8.92	8.95	17.89	26.84
Perfluorononanesulfonic acid	17	88.45	13.28	11.75	23.5	35.25
Perfluorodecanesulfonic acid	17	91.68	8.68	7.96	15.91	23.87
Perfluorododecanesulfonic acid	17	85	8.77	7.45	14.9	22.35
4:2 fluorotelomersulfonic acid	17	96.69	6.93	6.7	13.39	20.09
6:2 fluorotelomersulfonic acid	16	128.41	53.88	69.18	138.37	207.55
8:2 fluorotelomersulfonic acid	17	113.05	7.85	8.87	17.75	26.62
Perfluorooctanesulfonamide	17	104.66	7.01	7.33	14.67	22
N-methyl perfluorooctanesulfonamide	17	95.91	9.24	8.87	17.73	26.6
N-ethyl perfluorooctanesulfonamide	17	99.75	9.38	9.36	18.71	28.07

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
N-methyl perfluorooctanesulfonamidoacetic acid	17	92.45	14.12	13.05	26.1	39.15
N-ethyl perfluorooctanesulfonamidoacetic acid	17	99.38	11.43	11.36	22.71	34.07
N-methyl perfluorooctanesulfonamidoethanol	17	97.56	5.63	5.49	10.99	16.48
N-ethyl perfluorooctanesulfonamidoethanol	17	100.8	6.16	6.21	12.43	18.64
Hexafluoropropylene oxide dimer acid	17	95.55	11.92	11.39	22.77	34.16
4,8-dioxa-3H-perfluorononanoic acid	17	92.54	12.4	11.47	22.94	34.42
Perfluoro-3-methoxypropanoic acid	17	96.12	7.19	6.91	13.82	20.73
Perfluoro-4-methoxybutanoic acid	17	95.32	10.87	10.36	20.72	31.08
Perfluoro-3,6-dioxaheptanoic acid	17	90.95	24.78	22.54	45.07	67.61
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	17	97.06	11.7	11.35	22.7	34.05
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	17	94.19	14.03	13.22	26.43	39.65
Perfluoro(2-ethoxyethane)sulfonic acid	17	93.18	9.56	8.91	17.81	26.72
2H, 2H, 3H, 3H-perfluorohexanoic acid	17	88.01	10.5	9.24	18.47	27.71
2H, 2H, 3H, 3H-perfluorooctanoic acid	17	86.91	6.91	6.01	12.02	18.03
2H, 2H, 3H, 3H-perfluorodecanoic acid	17	86.06	8.62	7.42	14.83	22.25
Perfluoro-n-[13C4]butanoic acid	17	102.26	3.44	3.52	7.03	10.55
Perfluoro-n-[13C5]pentanoic acid	17	95.68	8.03	7.68	15.36	23.04
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	17	99.08	4.1	4.06	8.13	12.19
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	17	95.31	4.56	4.34	8.68	13.03
Perfluoro-n-[13C8]octanoic acid	17	98.42	3.94	3.88	7.75	11.63
Perfluoro-n-[13C9]nonanoic acid	17	98.99	4.97	4.92	9.84	14.75
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	17	97.68	3.98	3.89	7.78	11.66
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	17	101.82	6.13	6.24	12.48	18.72
Perfluoro-n-[1,2-13C2]dodecanoic acid	17	91.36	7.24	6.61	13.22	19.84
Perfluoro-n-[1,2-13C2]tetradecanoic acid	17	88.42	9.34	8.26	16.51	24.77
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	17	103.06	3.84	3.96	7.92	11.88
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	17	98.15	3.17	3.11	6.23	9.34
Perfluoro-1-[13C8]octanesulfonic acid	17	101.51	4.27	4.33	8.67	13
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	17	143.29	4.7	6.73	13.47	20.2
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	17	122.47	6.08	7.45	14.9	22.35
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	17	109.51	6.12	6.7	13.4	20.1
Perfluoro-1-[13C8]octanesulfonamide	17	84.56	11.83	10	20.01	30.01
N-methyl-d3-perfluoro-1-octanesulfonamide	17	42.41	26.9	11.41	22.81	34.22
N-ethyl-d5-perfluoro-1-octanesulfonamide	17	35.94	33.93	12.19	24.39	36.58
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	17	98.52	9.02	8.88	17.77	26.65
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	17	97.76	10.18	9.95	19.91	29.86
N-methyl-d7-perfluorooctanesulfonamidoethanol	17	54.89	20.11	11.04	22.08	33.11

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
N-ethyl-d9-perfluorooctane sulfonamidoethanol	17	51.39	20.79	10.68	21.36	32.05
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	17	106.15	10.09	10.71	21.43	32.14

Note: Percent recovery values used in the % Mean Recovery column were reported in the EDD and not calculated by IDA using Equation 1.

A description of each column in Table 17:

- Analyte—Always 64 PFAS analytes.
- n=—Number of LOQVER subsamples that do not have B flag values, for the analyte in the EDD, for the media type.
- % Mean Recovery—Mean of the LOQVER subsample percent recoveries of the EDD entries.
- RSD—Percent RSD of the mean of the sample percent recoveries.
- 1× SD—SD of the sample mean percent recoveries.
- 2× SD—SD of the sample mean percent recoveries times two.
- 3× SD—SD of the sample mean percent recoveries times three.

Table 18 is constructed from all matrices. This is file I-4 All Sample LOQVER Spike Recovery Table.csv in section I of the digital appendix. There is only one table like this, summarizing results across all media. The % Mean Recovery column is the mean of the mean percent recoveries and is potentially biased toward means with lower sample sizes since the sample means are not composed from the same number of data.

Table 18. Example All Media LOQVER Spike Recovery Table

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluorobutanoic acid	3	103.5	2.14	2.22	4.44	6.66
Perfluoropentanoic acid	3	106.09	4.62	4.9	9.79	14.69
Perfluorohexanoic acid	3	102.33	4.31	4.41	8.82	13.23
Perfluoroheptanoic acid	3	101.13	3.35	3.38	6.77	10.15
Perfluorooctanoic acid	3	102.74	4.15	4.26	8.52	12.78
Perfluorononanoic acid	3	103.14	3.74	3.86	7.72	11.58
Perfluorodecanoic acid	3	103.22	2.73	2.82	5.64	8.46
Perfluoroundecanoic acid	3	106.31	10.07	10.71	21.42	32.13
Perfluorododecanoic acid	3	100.83	1.95	1.97	3.94	5.91
Perfluorotridecanoic acid	3	112.01	11.1	12.43	24.86	37.29
Perfluorotetradecanoic acid	3	103.29	2.66	2.75	5.51	8.26
Perfluorobutanesulfonic acid	3	102.65	1.72	1.76	3.53	5.29
Perfluoropentanesulfonic acid	3	100.44	2.4	2.41	4.81	7.22
Perfluorohexanesulfonic acid	3	109.15	4.19	4.58	9.15	13.73

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluoroheptanesulfonic acid	3	97.21	3.62	3.52	7.04	10.56
Perfluorooctanesulfonic acid	3	111.8	12.13	13.57	27.13	40.7
Perfluorononanesulfonic acid	3	97.85	8.33	8.15	16.29	24.44
Perfluorodecanesulfonic acid	3	94.96	2.99	2.84	5.68	8.51
Perfluorododecanesulfonic acid	3	78.22	18.79	14.7	29.4	44.09
4:2 fluorotelomersulfonic acid	3	100.73	4.83	4.87	9.74	14.6
6:2 fluorotelomersulfonic acid	3	121.36	5.11	6.2	12.39	18.59
8:2 fluorotelomersulfonic acid	3	118.99	6.67	7.93	15.86	23.8
Perfluorooctanesulfonamide	3	109.57	5.8	6.35	12.71	19.06
N-methyl perfluorooctanesulfonamide	3	91.37	11.9	10.87	21.74	32.61
N-ethyl perfluorooctanesulfonamide	3	96.2	16.33	15.71	31.41	47.12
N-methyl perfluorooctanesulfonamidoacetic acid	3	93.89	7.63	7.16	14.33	21.49
N-ethyl perfluorooctanesulfonamidoacetic acid	3	103.28	3.86	3.99	7.98	11.97
N-methyl perfluorooctanesulfonamidoethanol	3	144.75	55.35	80.11	160.23	240.34
N-ethyl perfluorooctanesulfonamidoethanol	3	108.51	13.58	14.74	29.47	44.21
Hexafluoropropylene oxide dimer acid	3	97.64	4.21	4.11	8.23	12.34
4,8-dioxa-3H-perfluorononanoic acid	3	100.05	8.59	8.59	17.19	25.78
Perfluoro-3-methoxypropanoic acid	3	97.92	4.57	4.48	8.96	13.44
Perfluoro-4-methoxybutanoic acid	3	99.91	3.99	3.98	7.97	11.95
Perfluoro-3,6-dioxaheptanoic acid	3	94.73	5.31	5.03	10.06	15.09
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	3	104.06	8.15	8.49	16.97	25.46
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	3	102.37	12.4	12.7	25.39	38.09
Perfluoro(2-ethoxyethane)sulfonic acid	3	98.2	6.01	5.9	11.8	17.71
2H, 2H, 3H, 3H-perfluorohexanoic acid	3	87.96	7.3	6.42	12.84	19.26
2H, 2H, 3H, 3H-perfluorooctanoic acid	3	107.6	23.69	25.49	50.98	76.47
2H, 2H, 3H, 3H-perfluorodecanoic acid	3	101.5	15.24	15.47	30.94	46.41
Perfluoro-n-[13C4]butanoic acid	3	99.27	2.65	2.63	5.26	7.89
Perfluoro-n-[13C5]pentanoic acid	3	97.57	2.92	2.85	5.71	8.56
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	3	96.37	2.86	2.75	5.51	8.26
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	3	92.83	2.53	2.35	4.7	7.06
Perfluoro-n-[13C8]octanoic acid	3	96.96	1.55	1.5	3	4.5
Perfluoro-n-[13C9]nonanoic acid	3	96.84	2.35	2.28	4.56	6.84
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	3	97.29	2.63	2.56	5.11	7.67
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	3	99.49	2.58	2.57	5.14	7.71
Perfluoro-n-[1,2-13C2]dodecanoic acid	3	91.25	4.56	4.16	8.33	12.49
Perfluoro-n-[1,2-13C2]tetradecanoic acid	3	73.84	30.5	22.52	45.04	67.56
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	3	99.07	3.63	3.59	7.19	10.78
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	3	96.21	2.01	1.93	3.87	5.8
Perfluoro-1-[13C8]octanesulfonic acid	3	99.54	2.05	2.04	4.09	6.13
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	3	160.17	34.26	54.87	109.74	164.61

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	3	119.86	7.3	8.75	17.5	26.25
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	3	131.97	32.04	42.28	84.57	126.85
Perfluoro-1-[13C8]octanesulfonamide	3	89.86	13.61	12.23	24.46	36.69
N-methyl-d3-perfluoro-1-octanesulfonamide	3	39.32	50.06	19.68	39.36	59.04
N-ethyl-d5-perfluoro-1-octanesulfonamide	3	37.97	51.14	19.42	38.84	58.26
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	3	120.29	36.82	44.29	88.58	132.87
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	3	130.18	48.89	63.64	127.28	190.92
N-methyl-d7-perfluorooctanesulfonamidoethanol	3	41.21	84.74	34.92	69.84	104.76
N-ethyl-d9-perfluorooctane sulfonamidoethanol	3	41.78	67.48	28.19	56.39	84.58
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	3	102.22	5.33	5.45	10.9	16.35

A description of each column in Table 18:

- Analyte—Always 64 PFAS analytes.
- n=—Number of matrices for the analyte in the EDD, for all media type.
- % Mean Recovery—Mean of the LOQVER matrix mean percent recoveries for all media.
- RSD—Percent RSD of the mean of the sample percent recoveries.
- 1× SD—SD of the matrix mean percent recoveries.
- 2× SD—SD of the matrix mean percent recoveries times two.
- 3× SD—SD of the matrix mean percent recoveries times three.

J. Method Blank (MB) Spike Recovery Tables

We generated a total of four MB spike recovery tables which are section J of the digital appendix. MBs are quality assurance samples. Spike recoveries of MBs indicate the meticulousness of the laboratory's preparation of samples. We calculated the mean of the MB subsample percent recoveries and percent RSD, SD, 2× SD, and 3× SD for 24 analytes, from the 32 samples, for aqueous and solid media (including biosolid, sediment, and soil, but excluding tissue). At the sponsor's direction, we also performed similar calculations for the tissue matrix only. We used the sample percent recovery values in the EDD and did not include data that had B flags (where the results was associated with a contaminated blank in our calculations).

We then calculated the mean of the MB matrix mean percent recoveries and percent RSD, SD, 2× SD, and 3× SD of the matrix mean percent recoveries for the 24 analytes, for all media (i.e., aqueous, solid, and tissue).

Table 19 is constructed from aqueous samples. This table is file J-1 Aqueous Sample MB Spike Recovery Table.csv in section J of the digital appendix. There are three tables like this, for aqueous, solid (excluding tissue), and tissue matrixes.

Table 19. Example Media Type MB Spike Recovery Table

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluoro-n-[13C4]butanoic acid	22	95.95	5.39	5.17	10.35	15.52
Perfluoro-n-[13C5]pentanoic acid	22	96.7	7.69	7.44	14.88	22.31
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	22	93.34	6.57	6.13	12.26	18.39
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	22	92.47	5.66	5.23	10.46	15.69
Perfluoro-n-[13C8]octanoic acid	22	94.09	5.86	5.52	11.04	16.55
Perfluoro-n-[13C9]nonanoic acid	22	93.77	5.29	4.96	9.91	14.87
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	22	92.05	7.25	6.68	13.35	20.03
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	22	94.32	6.72	6.34	12.67	19.01
Perfluoro-n-[1,2-13C2]dodecanoic acid	22	84.48	9.84	8.31	16.62	24.93
Perfluoro-n-[1,2-13C2]tetradecanoic acid	22	82.75	10.51	8.7	17.39	26.09
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	22	97.9	4.54	4.45	8.89	13.34
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	22	94.1	5.18	4.88	9.75	14.63
Perfluoro-1-[13C8]octanesulfonic acid	22	96	7.1	6.81	13.62	20.43
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	22	119.35	10.36	12.36	24.72	37.08
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	22	113.13	17.57	19.88	39.75	59.63
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	22	108.87	15.27	16.62	33.24	49.87
Perfluoro-1-[13C8]octanesulfonamide	22	80.41	16.13	12.97	25.94	38.91
N-methyl-d3-perfluoro-1-octanesulfonamide	22	59.4	19.28	11.45	22.9	34.36
N-ethyl-d5-perfluoro-1-octanesulfonamide	22	60.33	17.9	10.8	21.6	32.39
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	22	90.97	13.18	11.99	23.97	35.96
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	22	88.4	13.45	11.89	23.79	35.68
N-methyl-d7-perfluorooctanesulfonamidoethanol	22	69.03	21.74	15.01	30.02	45.03
N-ethyl-d9-perfluorooctane sulfonamidoethanol	22	64.19	19.49	12.51	25.02	37.54
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	22	101.23	9.72	9.84	19.69	29.53

Note: Percent recovery values used in the % Mean Recovery column were reported in the EDD and not calculated by IDA using Equation 1.

A description of each column in Table 19:

- Analyte—Always 24 PFAS analytes.
- n=—Number of MB subsamples that do not have B flag values for the analyte in the EDD, for the media type (e.g., aqueous, solid (i.e., biosolid, sediment, and soil), tissue).
- % Mean Recovery—Mean of the MB subsample percent recoveries of the EDD entries.
- RSD—Percent RSD of the mean of the sample percent recoveries.
- 1× SD—SD of the sample mean percent recoveries.
- 2× SD—SD of the sample mean percent recoveries times two.
- 3× SD—SD of the sample mean percent recoveries times three.

Table 20 is constructed from all matrices. This is file J-4 All Sample MB Spike Recovery Table.csv from section J of the digital appendix. This is the only table of this type, summarizing results across *all* matrices. The % Mean Recovery column is the mean of the mean percent recoveries and is potentially biased toward means with lower sample sizes since the sample means are not composed from the same number of data.

Table 20. Example All Media MB Spike Recovery Table

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
Perfluoro-n-[13C4]butanoic acid	3	99.05	2.78	2.76	5.52	8.28
Perfluoro-n-[13C5]pentanoic acid	3	96.16	1.09	1.05	2.1	3.14
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	3	95.79	2.27	2.17	4.34	6.51
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	3	92.74	1.35	1.25	2.51	3.76
Perfluoro-n-[13C8]octanoic acid	3	95.78	1.93	1.84	3.69	5.53
Perfluoro-n-[13C9]nonanoic acid	3	95.57	1.64	1.56	3.13	4.69
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	3	95.07	2.81	2.67	5.33	8
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	3	99.2	4.31	4.27	8.54	12.82
Perfluoro-n-[1,2-13C2]dodecanoic acid	3	88.98	5.5	4.9	9.8	14.69
Perfluoro-n-[1,2-13C2]tetradecanoic acid	3	79	14.97	11.83	23.66	35.48
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	3	98.45	2.83	2.78	5.57	8.35
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	3	95.93	1.77	1.7	3.4	5.1
Perfluoro-1-[13C8]octanesulfonic acid	3	98.68	2.39	2.35	4.71	7.06
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	3	153.93	26.65	41.02	82.04	123.05
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	3	119.92	5.58	6.69	13.37	20.06
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	3	128.1	26.81	34.34	68.69	103.03
Perfluoro-1-[13C8]octanesulfonamide	3	91.83	17.32	15.91	31.82	47.73

Analyte	n =	% Mean Recovery	RSD	1× SD	2× SD	3× SD
N-methyl-d3-perfluoro-1-octanesulfonamide	3	46.76	24.47	11.44	22.88	34.32
N-ethyl-d5-perfluoro-1-octanesulfonamide	3	45.02	29.45	13.26	26.52	39.78
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	3	114.52	31.46	36.03	72.06	108.09
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	3	125.58	45.25	56.83	113.66	170.48
N-methyl-d7-perfluorooctanesulfonamidoethanol	3	43.52	77.43	33.69	67.39	101.08
N-ethyl-d9-perfluorooctane sulfonamidoethanol	3	47.6	40.75	19.4	38.8	58.19
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	3	98.94	4.15	4.1	8.21	12.31

A description of each column in Table 20:

- Analyte—Always 24 PFAS analytes.
- n=—Number of matrices for the analyte in the EDD, for all media type.
- % Mean Recovery—Mean of the MB matrix mean percent recoveries, for all media.
- RSD—Percent RSD of the mean of the sample percent recoveries.
- 1× SD—SD of the matrix mean percent recoveries.
- 2× SD—SD of the matrix mean percent recoveries times two.
- 3× SD—SD of the matrix mean percent recoveries times three.

4. Discussion

We plotted the mean native concentration of the 40 PFAS analytes measured from each of the 32 environmental samples in the SLV study, using tables described in Section 3.C and in section C of the digital appendix (Figure 1). Figure 1 illustrates the prevalence of PFAS contamination across environmental media. The native PFAS analyte concentration was reported by the laboratory in units of ng/L for aqueous matrices and µg/kg for solid matrices. Concentrations of chemical compounds in aqueous solution are typically measured in units of mass of chemical (e.g., grams) per volume of water (e.g., liters) but can also be written as a mass fraction in units of mass of chemical per mass of water since the density of water is 1 g/mL. We converted the PFAS analyte concentrations for the aqueous samples to mass fraction in units of µg/kg or parts per billion (ppb) to plot the native PFAS analyte concentration for both the aqueous and solid matrices with the same scale on the plot. Each square was based on 0, 1, 2, or 3 subsample points, after throwing out all subsamples with U or B flags (refer back to Section 3.C). The gray NA color means all three subsamples had U or B flags (e.g., NA is used when the analyte was not detected or associated with contamination).

Figure 1 shows that several of the samples (columns), including one of the leachate samples (column LC1), contain many of the PFAS analytes. Across the analytes (rows), several (e.g., PFOA and PFOS) were detected in almost every sample. A thick grey line separates the aqueous sample columns from the solid sample columns.

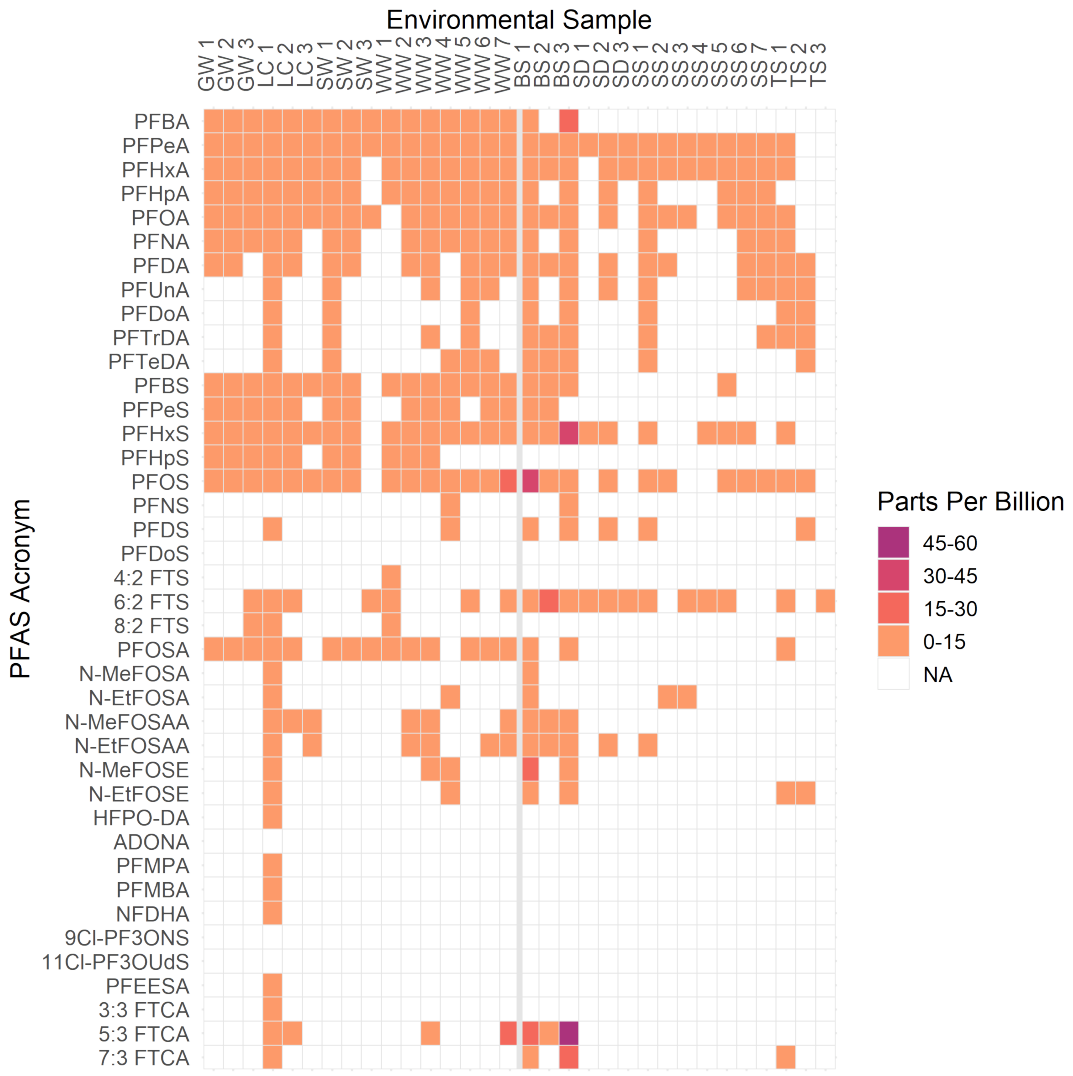


Figure 1. Mean Native Concentrations of 40 PFAS Analytes from 32 Environmental Samples in the SLV Study from the Tables in Section C of the Digital Appendix for Groundwater (GW), Landfill Leachate (LC), Surface Water (SW), Wastewater (WW), Biosolid (BS), Sediment (SD), Soil (SS), and Tissue (TS) Matrices. Numbers reported are mean values over all subsamples for that analyte/matrix combination that are greater than the detection limit.

We then plotted the mean percent recovery values for the 40 PFAS analytes measured from each of the environmental samples in the SLV, using tables described in the Section 3.D and in section D of the digital appendix (Figure 2). Each square was based on zero to four subsample points after throwing out U and B flags (refer to Section 3.C). The average percent recovery was greater than 75% for 92% of the analyte/matrix combinations. The analytes were spiked at different concentrations so the individual percent recoveries for a specific concentration cannot be determined from the figure. Figure 2 does show in white where we could not calculate the percent recovery when the mean native concentration for the analyte was greater than the subsample spike concentration or three subsample values

had B flags. The native concentration was greater than the spike concentration (see Figure 1) for several analytes in the leachate samples (columns LC1, LC2, and LC3), so the mean percent recovery could not be calculated in Figure 2 (shown in white). Comparing Figure 1 and Figure 2, we could not calculate the mean percent recovery for some analytes (e.g., PFOA and PFOS); these two analytes were prevalent in many of the native samples. Analytes PFDoS and 6:2 FTS had the lowest mean percent recoveries in some of the environmental samples.

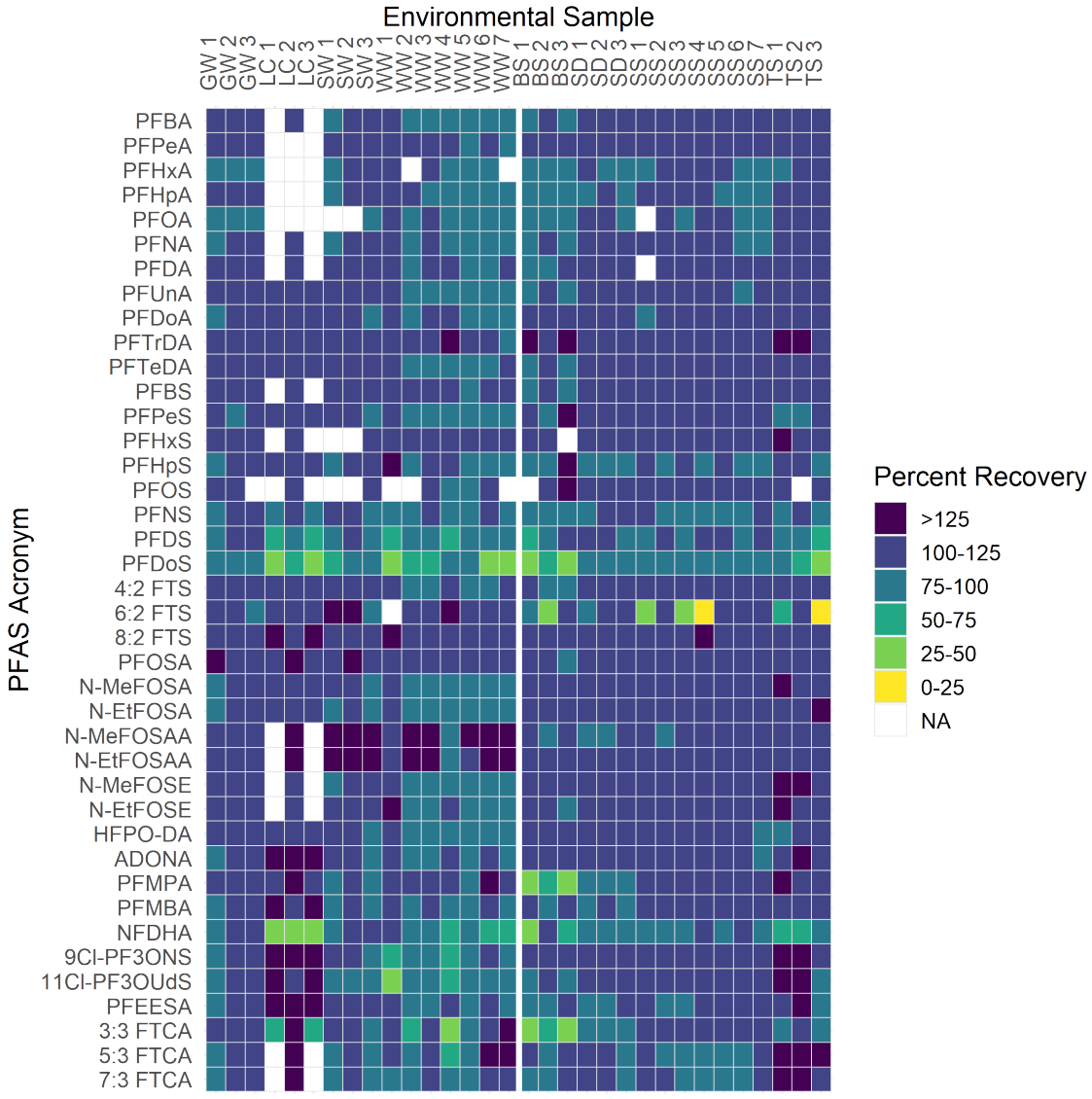


Figure 2. Matrix Spike Percent Recovery Values for the 40 PFAS Analytes from the 32 Environmental Samples in the SLV Study Corresponding to the Tables in Section D of the Digital Appendix for Groundwater (GW), Landfill Leachate (LC), Surface Water (SW), Wastewater (WW), Biosolid (BS), Sediment (SD), Soil (SS), and Tissue (TS) Matrices. Numbers reported are mean values over all subsamples for that analyte/matrix combination that are greater than the detection limit.

5. Conclusion

We compiled the laboratory reported results and calculated the summary statistics in support of the government's SLV study of PFAS analytes in environmental media. We automatically generated data tables in a systematic and reproducible fashion using a coded Python computer script to eliminate human error. We performed a rigorous quality check on our summary statistics to be confident they were calculated correctly. These data can now be used to support the government's SLV of the novel PFAS method.

The calculations included the mean native concentration, the mean spike concentration, and the percent recoveries of the 40 PFAS analytes in 32 environmental samples from aqueous and solid matrices. In addition, we calculated the mean spike concentration and percent recovery of the PFAS isotopically labeled EIS and IIS in the same environmental media. We also calculated the ongoing precision spike recovery and limit of quantitation verification spike recovery for the PFAS analytes across all the samples. Several of the samples had very high native concentrations of many of the PFAS analytes, which exemplifies the prevalence of PFAS throughout the environment. The average percent recovery was greater than 75% for 92% of the analyte/matrix combinations.

Appendix A.
Figures and Tables

Figures

Tables

Appendix B.
Abbreviations

AFFF	aqueous film-forming foam
CAS	chemical abstract service
EDD	electronic data delivery
EIS	extracted internal standard
EPA	Environmental Protection Agency
ESTCP	Environmental Security Technology Certification Program
IIS	injected internal standard
LC	liquid chromatography
LOQVER	limit of quantitation verification
MS	mass spectroscopy
NAVSEA	Naval Sea Systems Command
OLEM	Office of Land and Emergency Management
OPR	ongoing precision and recovery
PFAS	per- and polyfluoroalkyl substances
PFBS	perfluorobutane sulfonic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctane sulfonate
RSD	relative standard deviation
SD	standard deviation
SERDP	Strategic Environmental Research and Development Program
SLV	single laboratory validation

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
August 2021	FINAL	

4. TITLE AND SUBTITLE

Data Compilation in Support of Single Laboratory Validation of a Novel Per- and Polyfluoroalkyl Substances (PFAS) Detection Method for Environmental Matrices

5a. CONTRACT NUMBER

HQ0034-19-D-0001

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

Buytendyk, Allyson M.
Runkel, Sara C.
Cazares, Shelley M.

5d. PROJECT NUMBER

AM-2-1528

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Institute for Defense Analyses
4850 Mark Center Drive
Alexandra, VA 22311-1882

8. PERFORMING ORGANIZATION REPORT NUMBER

IDA Document D-22794

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

SERDP and ESTCP
4800 Mark Center Drive
Suite 16F16
Alexandria, VA 22350-3605

10. SPONSOR/MONITOR'S ACRONYM(S)

SERDP/ESTCP

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited (3 September 2021).

13. SUPPLEMENTARY NOTES

14. ABSTRACT

The Institute for Defense Analyses (IDA) performed statistical analyses to assist in the validation of the novel mass spectrometry detection methods of per- and polyfluoroalkyl substances (PFAS) as part of the DoD's Single Laboratory Validation study. IDA processed the data reported by the laboratory to calculate the average native concentration, the average spike concentration, and the percent recovery of the PFAS analytes in aqueous and solid type matrices from the environment. Additionally, IDA calculated the average spike concentration and percent recovery of the PFAS isotopically labeled internal standards in the same eight matrices. IDA also calculated the ongoing precision and recovery, limit of quantitation verification, and method blank spike recovery for the PFAS analytes. Over 100 data tables were automatically generated by an IDA-coded Python computer script to illustrate the performance of the PFAS analytical method with environmental media.

15. SUBJECT TERMS

environmental remediation; mass spectrometry; native concentration; per- and poly-fluoroalkyl substances (PFAS); percent recovery; single laboratory validation (SLV); spike concentration

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Nelson, Herbert
Unclassified	Unclassified	Unclassified	SAR	73	19b. TELEPHONE NUMBER *(Include area code)* 571-372-6400

Reset

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

Appendix C

Individual Sample Native Concentrations

APPENDIX C. NATIVE PFAS CONCENTRATIONS IN TEST ENVIRONMENTAL MEDIA

This appendix reports the background or "native" concentrations in each of the environmental matrices; groundwater, surface water, wastewater, and landfill leachate, soil, sediment, biosolid, and tissue.

For each matrix samples, the native concentrations were measured in triplicate, and reported.

Details of the data compilation and analyses process are presented in Appendix K (IDA, 2021).

- Analyte—40 PFAS analytes listed in Table 1 of the Single Lab Validation Study Report
- Concentration—Native concentration for each of the three subsamples of the analyte from the EDD in µg/kg for solid matrices and tissues, ng/L for aqueous matrices.
- Qualifier—Lab flag for the native concentration of each of the three analyte subsamples from the EDD.
- Number of Detections—Number of subsamples for the analyte that do not have a U or B flag for native concentration. Could be 0, 1, 2 or 3.
- Mean—Mean native concentration of the subsamples that do not have a U or B flag. If all three subsamples have U or B, then the value is NA.
- RSD— Percent RSD of the mean native concentration. If the number of samples is less than two then NA is reported.

Table C-1(a) GW CO Native Concentration

Analyte	L33076-1		L33076-2		L33076-3		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	18.2		18.2		18.2		3	18.2	0.00
PFPeA	22.2		22		22.4		3	22.2	0.90
PFHxA	19.9		19.5		19.4		3	19.6	1.35
PFHpA	8.16		7.51		6.74		3	7.47	9.52
PFOA	23.80		21.5		23.8		3	23.03	5.77
PFNA	2.11		1.94		2.2		3	2.08	6.34
PFDA	0.561	J	0.582	J	0.524	J	3	0.56	5.28
PFUnA	0.246	U	0.242	U	0.259	U	0	NA	NA
PFDoA	0.359	U	0.353	U	0.379	U	0	NA	NA
PFTrDA	0.227	U	0.223	U	0.239	U	0	NA	NA
PFTeDA	0.246	U	0.242	U	0.259	U	0	NA	NA
PFBS	19.6		20.5		21.6		3	20.57	4.87
PFPeS	8.05		8.17		7.97		3	8.06	1.25
PFHxS	46.8		47.1		50.3		3	48.07	4.04
PFHpS	1.31	J	1.25	J	1.35	J	3	1.3	3.86
PFOS	123		118		131		3	124	5.29
PFNS	0.284	U	0.279	U	0.299	U	0	NA	NA
PFDS	0.312	U	0.307	U	0.329	U	0	NA	NA
PFDoS	0.17	U	0.167	U	0.179	U	0	NA	NA
4:2 FTS	2.15	U	2.120	U	2.27	U	0	NA	NA
6:2 FTS	3.75	U	3.69	U	3.96	U	0	NA	NA
8:2 FTS	1.48	U	1.46	U	1.56	U	0	NA	NA
PFOSA	5.44		5.81		5.56		3	5.6	3.37
N-MeFOSA	0.189	U	0.186	U	0.199	U	0	NA	NA
N-EtFOSA	0.548	U	0.539	U	0.578	U	0	NA	NA
N-MeFOSAA	0.558	U	0.548	U	0.588	U	0	NA	NA
N-EtFOSAA	0.302	U	0.297	U	0.319	U	0	NA	NA

Table C-1(a) GW CO Native Concentration

Analyte	L33076-1		L33076-2		L33076-3		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
N-MeFOSE	1.12	U	1.11	U	1.19	U	0	NA	NA
N-EtFOSE	0.964	U	0.948	U	1.02	U	0	NA	NA
HFPO-DA	0.388	U	0.381	U	0.409	U	0	NA	NA
ADONA	0.737	U	0.725	U	0.777	U	0	NA	NA
PFMPA	0.17	U	0.167	U	0.179	U	0	NA	NA
PFMBA	0.113	U	0.112	U	0.12	U	0	NA	NA
NFDHA	1.3	U	1.28	U	1.38	U	0	NA	NA
9Cl-PF3ONS	0.822	U	0.809	U	0.867	U	0	NA	NA
11Cl-PF3OUdS	0.775	U	0.762	U	0.817	U	0	NA	NA
PFEESA	0.132	U	0.13	U	0.14	U	0	NA	NA
3:3 FTCA	0.68	U	0.669	U	0.717	U	0	NA	NA
5:3 FTCA	4.79	U	4.71	U	5.05	U	0	NA	NA
7:3 FTCA	5.61	U	5.52	U	5.92	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-1(b) GW CO Native HIGH 2 Native Concentration

Analyte	L33877-1	
	Concentration (ng/L)	Qualifier
PFBA	4.9	J
PFPeA	5.8	
PFHxA	5.0	
PFHpA	1.98	
PFOA	5.74	
PFNA	0.45	J
PFDA	0.329	U
PFUnA	0.259	U
PFDoA	0.379	U
PFTrDA	0.240	U
PFTeDA	0.259	U
PFBS	5.1	
PFPeS	1.99	
PFHxS	11.1	
PFHpS	0.26	J
PFOS	20	
PFNS	0.299	U
PFDS	0.329	U
PFDoS	0.18	U
4:2 FTS	2.28	U
6:2 FTS	3.96	U
8:2 FTS	1.57	U
PFOSA	0.97	J
N-MeFOSA	0.200	U
N-EtFOSA	0.579	U
N-MeFOSAA	0.589	U
N-EtFOSAA	0.319	U
N-MeFOSE	1.19	U
N-EtFOSE	1.020	U
HFPO-DA	0.409	U
ADONA	0.778	U
PFMPA	0.18	U
PFMBA	0.120	U
NFDHA	1.4	U
9Cl-PF3ONS	0.868	U
11Cl-PF3OUdS	0.818	U
PFEESA	0.140	U
3:3 FTCA	0.72	U
5:3 FTCA	5.06	U
7:3 FTCA	5.93	U

py_Log Date 6/28/2021 16:01

Table C-2 (a): GW 2 WL378 Native Concentration

Analyte	L33208-7		L33208-8		L33208-9		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	10.5		10.4		10.5		3	10.47	0.55
PFPeA	13.7		13.5		13.7		3	13.63	0.85
PFHxA	50.8		48.8		49.7		3	49.77	2.01
PFHpA	7.27		7.18		7.06		3	7.17	1.47
PFOA	44		40.6		39.2		3	41.27	5.98
PFNA	0.86	J	0.554	J	0.49	J	3	0.63	31.16
PFDA	0.416	J	0.455	J	0.314	U	2	0.44	6.33
PFUnA	0.258	U	0.249	U	0.247	U	0	NA	NA
PFDoA	0.377	U	0.364	U	0.361	U	0	NA	NA
PFTrDA	0.238	U	0.23	U	0.228	U	0	NA	NA
PFTeDA	0.258	U	0.249	U	0.247	U	0	NA	NA
PFBS	19.6		20.5		20.2		3	20.1	2.28
PFPeS	18.7		18.1		18.4		3	18.4	1.63
PFHxS	244		233		232		3	236.33	2.82
PFHpS	6.22		4.92		5.18		3	5.44	12.65
PFOS	421		308		308		3	345.67	18.87
PFNS	0.298	U	0.288	U	0.285	U	0	NA	NA
PFDS	0.327	U	0.316	U	0.314	U	0	NA	NA
PFDoS	0.179	U	0.173	U	0.171	U	0	NA	NA
4:2 FTS	2.26	U	2.19	U	2.17	U	0	NA	NA
6:2 FTS	23.4	B	3.81	U	3.77	U	0	NA	NA
8:2 FTS	1.56	U	1.5	U	1.49	U	0	NA	NA
PFOSA	4.88		4.59		4.68		3	4.72	3.15
N-MeFOSA	0.198	U	0.192	U	0.19	U	0	NA	NA
N-EtFOSA	0.575	U	0.556	U	0.551	U	0	NA	NA
N-MeFOSAA	0.585	U	0.565	U	0.561	U	0	NA	NA
N-EtFOSAA	0.317	U	0.307	U	0.304	U	0	NA	NA
N-MeFOSE	1.18	U	1.14	U	1.13	U	0	NA	NA

Table C-2 (a): GW 2 WL378 Native Concentration

Analyte	L33208-7		L33208-8		L33208-9		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
N-EtFOSE	1.01	U	0.978	U	0.97	U	0	NA	NA
HFPO-DA	0.407	U	0.393	U	0.39	U	0	NA	NA
ADONA	0.773	U	0.748	U	0.742	U	0	NA	NA
PFMPA	0.179	U	0.173	U	0.171	U	0	NA	NA
PFMBA	0.119	U	0.115	U	0.114	U	0	NA	NA
NFDHA	1.37	U	1.32	U	1.31	U	0	NA	NA
9Cl-PF3ONS	0.863	U	0.834	U	0.827	U	0	NA	NA
11Cl-PF3OUdS	0.813	U	0.786	U	0.78	U	0	NA	NA
PFEESA	0.139	U	0.134	U	0.133	U	0	NA	NA
3:3 FTCA	0.714	U	0.69	U	0.685	U	0	NA	NA
5:3 FTCA	5.03	U	4.86	U	4.82	U	0	NA	NA
7:3 FTCA	5.89	U	5.69	U	5.65	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-2(b) GW WL378 Native HIGH 2 Concentration

Analyte	L33895-1	
	Concentration (ng/L)	Qualifier
PFBA	2.9	J
PFPeA	3.6	
PFHxA	12.2	
PFHpA	1.91	
PFOA	8.97	
PFNA	0.22	U
PFDA	0.331	U
PFUnA	0.261	U
PFDoA	0.381	U
PFTrDA	0.241	U
PFTeDA	0.261	U
PFBS	5.1	
PFPeS	4.44	
PFHxS	54.3	
PFHpS	1.03	J
PFOS	57	
PFNS	0.301	U
PFDS	0.331	U
PFDoS	0.18	U
4:2 FTS	2.29	U
6:2 FTS	3.98	U
8:2 FTS	1.57	U
PFOSA	0.63	J
N-MeFOSA	0.200	U
N-EtFOSA	0.581	U
N-MeFOSAA	0.591	U
N-EtFOSAA	0.321	U
N-MeFOSE	1.19	U
N-EtFOSE	1.020	U
HFPO-DA	0.411	U
ADONA	0.782	U
PFMPA	0.18	U
PFMBA	0.120	U
NFDHA	1.4	U
9Cl-PF3ONS	0.872	U
11Cl-PF3OUdS	0.822	U
PFEESA	0.140	U
3:3 FTCA	0.72	U
5:3 FTCA	5.08	U
7:3 FTCA	5.95	U

py_Log Date 6/28/2021 16:01

Table C-3(a) GW Wurtsmith AFB Native Concentration

Analyte	L33171-4		L33171-5		L33171-6		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	3.86	J	3.88	J	3.77	J	3	3.84	1.53
PFPeA	15.3		15.4		14.5		3	15.07	3.27
PFHxA	37.8		38.4		38.5		3	38.23	0.99
PFHpA	7.59		7.84		7.68		3	7.7	1.64
PFOA	64.5		65.1		63.3		3	64.3	1.43
PFNA	1.24	J	1.25	J	1.26	J	3	1.25	0.8
PFDA	0.342	U	0.332	U	0.313	U	0	NA	NA
PFUnA	0.269	U	0.261	U	0.247	U	0	NA	NA
PFDoA	0.394	U	0.382	U	0.361	U	0	NA	NA
PFTrDA	0.249	U	0.241	U	0.228	U	0	NA	NA
PFTeDA	0.269	U	0.261	U	0.247	U	0	NA	NA
PFBS	1.95		1.98		1.78		3	1.9	5.67
PFPeS	3.49		3.31		3.37		3	3.39	2.7
PFHxS	148		151		147		3	148.67	1.4
PFHpS	4.6		4.85		4.84		3	4.76	2.97
PFOS	354		420		370		3	381.33	9.03
PFNS	0.311	U	0.302	U	0.285	U	0	NA	NA
PFDS	0.342	U	0.332	U	0.313	U	0	NA	NA
PFDoS	0.187	U	0.181	U	0.171	U	0	NA	NA
4:2 FTS	2.36	U	2.29	U	2.16	U	0	NA	NA
6:2 FTS	47.6		46.9		46.8		3	47.1	0.93
8:2 FTS	14.5		19.4		15.5		3	16.47	15.72
PFOSA	21.5		26.2		22.6		3	23.43	10.49
N-MeFOSA	0.207	U	0.201	U	0.19	U	0	NA	NA
N-EtFOSA	0.601	U	0.583	U	0.55	U	0	NA	NA
N-MeFOSAA	0.612	U	0.593	U	0.56	U	0	NA	NA
N-EtFOSAA	0.332	U	0.322	U	0.304	U	0	NA	NA
N-MeFOSE	1.23	U	1.2	U	1.13	U	0	NA	NA

Table C-3(a) GW Wurtsmith AFB Native Concentration

Analyte	L33171-4		L33171-5		L33171-6		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
N-EtFOSE	1.06	U	1.03	U	0.968	U	0	NA	NA
HFPO-DA	0.425	U	0.412	U	0.389	U	0	NA	NA
ADONA	0.808	U	0.784	U	0.74	U	0	NA	NA
PFMPA	0.187	U	0.181	U	0.171	U	0	NA	NA
PFMBA	0.124	U	0.121	U	0.114	U	0	NA	NA
NFDHA	1.43	U	1.39	U	1.31	U	0	NA	NA
9Cl-PF3ONS	0.902	U	0.875	U	0.826	U	0	NA	NA
11Cl-PF3OUdS	0.85	U	0.825	U	0.778	U	0	NA	NA
PFEESA	0.145	U	0.141	U	0.133	U	0	NA	NA
3:3 FTCA	0.746	U	0.724	U	0.683	U	0	NA	NA
5:3 FTCA	5.26	U	5.1	U	4.81	U	0	NA	NA
7:3 FTCA	6.16	U	5.97	U	5.64	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-3(b) GW Wurtsmith AFB HIGH 2 Native Concentration

Analyte	L33878-1	
	Concentration (ng/L)	Qualifier
PFBA	1.1	J
PFPeA	3.6	
PFHxA	9.3	
PFHpA	1.86	
PFOA	15.20	
PFNA	0.34	J
PFDA	0.330	U
PFUnA	0.260	U
PFDoA	0.380	U
PFTrDA	0.240	U
PFTeDA	0.260	U
PFBS	0.4	J
PFPeS	0.69	J
PFHxS	32.4	
PFHpS	1.10	J
PFOS	100	
PFNS	0.300	U
PFDS	0.330	U
PFDoS	0.18	U
4:2 FTS	2.28	U
6:2 FTS	11.70	
8:2 FTS	4.60	J
PFOSA	6.36	
N-MeFOSA	0.200	U
N-EtFOSA	0.580	U
N-MeFOSAA	0.590	U
N-EtFOSAA	0.320	U
N-MeFOSE	1.19	U
N-EtFOSE	1.020	U
HFPO-DA	0.410	U
ADONA	0.780	U
PFMPA	0.18	U
PFMBA	0.120	U
NFDHA	1.4	U
9Cl-PF3ONS	0.870	U
11Cl-PF3OUdS	0.820	U
PFEESA	0.140	U
3:3 FTCA	0.72	U
5:3 FTCA	5.07	U
7:3 FTCA	5.94	U

py_Log Date 6/28/2021 16:01

Table C-4 WW#3 Native Concentration

Analyte	L33173-4		L33173-5		L33173-6		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	3.87	J	3.87	J	3.84	J	3	3.86	0.45
PFPeA	1.9	J	2.27	J	1.98	J	3	2.05	9.5
PFHxA	1.83		1.76		1.9		3	1.83	3.83
PFHpA	1.61		1.41	J	1.62		3	1.55	7.66
PFOA	0.286	U	0.308	U	0.299	U	0	NA	NA
PFNA	0.21	U	0.226	U	0.22	U	0	NA	NA
PFDA	0.315	U	0.339	U	0.329	U	0	NA	NA
PFUnA	0.248	U	0.267	U	0.26	U	0	NA	NA
PFDoA	0.363	U	0.391	U	0.379	U	0	NA	NA
PFTrDA	0.229	U	0.247	U	0.24	U	0	NA	NA
PFTeDA	0.248	U	0.267	U	0.26	U	0	NA	NA
PFBS	0.67	J	0.577	J	0.688	J	3	0.65	9.24
PFPeS	0.191	U	0.206	U	0.2	U	0	NA	NA
PFHxS	0.21	U	0.226	U	0.251	J	1	0.25	NA
PFHpS	0.259	J	0.238	J	0.222	J	3	0.24	7.74
PFOS	443		350		323		3	372	16.92
PFNS	0.286	U	0.308	U	0.299	U	0	NA	NA
PFDS	0.315	U	0.339	U	0.329	U	0	NA	NA
PFDoS	0.172	U	0.185	U	0.18	U	0	NA	NA
4:2 FTS	6.64		6.67		6.71		3	6.67	0.53
6:2 FTS	9770	D	10500	D	8800	D	3	9690	8.8
8:2 FTS	19.8		14.3		14.7		3	16.27	18.85
PFOSA	0.434	J	0.888	J	0.611	J	3	0.64	35.51
N-MeFOSA	0.191	U	0.206	U	0.2	U	0	NA	NA
N-EtFOSA	0.554	U	0.596	U	0.579	U	0	NA	NA
N-MeFOSAA	0.563	U	0.606	U	0.589	U	0	NA	NA
N-EtFOSAA	0.305	U	0.329	U	0.319	U	0	NA	NA
N-MeFOSE	1.14	U	1.22	U	1.19	U	0	NA	NA

Table C-4 WW#3 Native Concentration

Analyte	L33173-4		L33173-5		L33173-6		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
N-EtFOSE	0.973	U	1.05	U	1.02	U	0	NA	NA
HFPO-DA	0.391	U	0.421	U	0.409	U	0	NA	NA
ADONA	0.744	U	0.802	U	0.779	U	0	NA	NA
PFMPA	0.172	U	0.185	U	0.18	U	0	NA	NA
PFMBA	0.115	U	0.123	U	0.12	U	0	NA	NA
NFDHA	1.32	U	1.42	U	1.38	U	0	NA	NA
9Cl-PF3ONS	0.83	U	0.894	U	0.868	U	0	NA	NA
11Cl-PF3OUdS	0.783	U	0.843	U	0.819	U	0	NA	NA
PFEESA	0.134	U	0.144	U	0.14	U	0	NA	NA
3:3 FTCA	0.687	U	0.74	U	0.719	U	0	NA	NA
5:3 FTCA	4.84	U	5.21	U	5.06	U	0	NA	NA
7:3 FTCA	5.67	U	6.11	U	5.93	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-5 WW#5 Native Concentration

Analyte	L33037-1		L33037-2		L33037-3		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	7.87		7.86		7.86		3	7.86	0.07
PFPeA	10.9		11		10.7		3	10.87	1.41
PFHxA	16.3		16.4		15.4		3	16.03	3.44
PFHpA	4.37		4.46		4.75		3	4.53	4.39
PFOA	8.73		8.34		8.52		3	8.53	2.29
PFNA	2.82		2.93		2.89		3	2.88	1.93
PFDA	0.844	J	0.665	J	0.579	J	3	0.7	19.42
PFUnA	0.254	U	0.257	U	0.267	U	0	NA	NA
PFDoA	0.372	U	0.376	U	0.39	U	0	NA	NA
PFTrDA	0.235	U	0.238	U	0.246	U	0	NA	NA
PFTeDA	0.254	U	0.257	U	0.267	U	0	NA	NA
PFBS	12.7		11.8		12		3	12.17	3.88
PFPeS	0.768	J	0.721	J	0.788	J	3	0.76	4.53
PFHxS	8.16		7.64		7.93		3	7.91	3.29
PFHpS	0.303	J	0.337	J	0.335	J	3	0.33	5.87
PFOS	11.9		13.3		12.6		3	12.6	5.56
PFNS	0.293	U	0.297	U	0.308	U	0	NA	NA
PFDS	0.323	U	0.327	U	0.338	U	0	NA	NA
PFDoS	0.176	U	0.178	U	0.185	U	0	NA	NA
4:2 FTS	2.23	U	2.26	U	2.34	U	0	NA	NA
6:2 FTS	3.88	U	3.93	U	4.07	U	0	NA	NA
8:2 FTS	1.53	U	1.55	U	1.61	U	0	NA	NA
PFOSA	0.25	J	0.28	J	0.439	J	3	0.32	31.45
N-MeFOSA	0.196	U	0.198	U	0.205	U	0	NA	NA
N-EtFOSA	0.567	U	0.574	U	0.595	U	0	NA	NA
N-MeFOSAA	0.577	U	0.844	J	0.605	U	1	0.84	NA
N-EtFOSAA	0.357	JQ	0.317	U	0.328	U	1	0.36	NA
N-MeFOSE	1.16	U	1.18	U	1.22	U	0	NA	NA
N-EtFOSE	0.997	U	1.01	U	1.05	U	0	NA	NA

Table C-5 WW#5 Native Concentration

Analyte	L33037-1		L33037-2		L33037-3		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.401	U	0.406	U	0.42	U	0	NA	NA
ADONA	0.763	U	0.772	U	0.8	U	0	NA	NA
PFMPA	0.176	U	0.178	U	0.185	U	0	NA	NA
PFMBA	0.117	U	0.119	U	0.123	U	0	NA	NA
NFDHA	1.35	U	1.37	U	1.41	U	0	NA	NA
9Cl-PF3ONS	0.851	U	0.861	U	0.892	U	0	NA	NA
11Cl-PF3OUdS	0.802	U	0.812	U	0.841	U	0	NA	NA
PFEESA	0.137	U	0.139	U	0.144	U	0	NA	NA
3:3 FTCA	0.704	U	0.713	U	0.738	U	0	NA	NA
5:3 FTCA	4.96	U	5.02	U	5.2	U	0	NA	NA
7:3 FTCA	5.81	U	5.88	U	6.09	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-6 WW#6 Native Concentration

Analyte	L33038-4		L33038-5		L33038-6		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	4.34	J	4.28	J	4.31	J	3	4.31	0.7
PFPeA	7.05		7.01		6.92		3	6.99	0.95
PFHxA	6.76		6.34		6.56		3	6.55	3.21
PFHpA	3.45		3.81		3.69		3	3.65	5.02
PFOA	9.82		9.95		11.2		3	10.32	7.38
PFNA	1.37	J	1.27	J	1.57		3	1.4	10.88
PFDA	0.819	J	0.659	J	1.04	J	3	0.84	22.79
PFUnA	0.314	JQ	0.249	U	0.365	J	2	0.34	10.62
PFDoA	0.372	U	0.364	U	0.373	U	0	NA	NA
PFTrDA	0.276	JQ	0.26	JQ	0.235	U	2	0.27	4.22
PFTeDA	0.255	U	0.249	U	0.255	U	0	NA	NA
PFBS	5.04		4.8		5.13		3	4.99	3.42
PFPeS	0.41	J	0.619	J	0.423	J	3	0.48	24.19
PFHxS	3.55		3.62		3.66		3	3.61	1.54
PFHpS	0.23	J	0.25	J	0.181	J	3	0.22	16.11
PFOS	11.3		11.7		11.4		3	11.47	1.82
PFNS	0.294	U	0.288	U	0.294	U	0	NA	NA
PFDS	0.323	U	0.316	U	0.324	U	0	NA	NA
PFDoS	0.176	U	0.173	U	0.177	U	0	NA	NA
4:2 FTS	2.23	U	2.19	U	2.24	U	0	NA	NA
6:2 FTS	3.89	U	3.81	U	3.89	U	0	NA	NA
8:2 FTS	1.54	U	1.51	U	1.54	U	0	NA	NA
PFOSA	0.977	J	0.541	J	3.1		3	1.54	88.94
N-MeFOSA	0.196	U	0.192	U	0.196	U	0	NA	NA
N-EtFOSA	0.569	U	0.556	U	0.569	U	0	NA	NA
N-MeFOSAA	0.691	J	0.566	U	1.9		2	1.3	65.99
N-EtFOSAA	0.515	J	0.493	J	1.16	J	3	0.72	52.43
N-MeFOSE	1.8	J	1.93	J	1.17	U	2	1.86	4.93
N-EtFOSE	1	U	0.978	U	1	U	0	NA	NA

Table C-6 WW#6 Native Concentration

Analyte	L33038-4		L33038-5		L33038-6		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.402	U	0.393	U	0.402	U	0	NA	NA
ADONA	0.765	U	0.748	U	0.765	U	0	NA	NA
PFMPA	0.176	U	0.173	U	0.177	U	0	NA	NA
PFMBA	0.118	U	0.115	U	0.118	U	0	NA	NA
NFDHA	1.35	U	1.32	U	1.35	U	0	NA	NA
9Cl-PF3ONS	0.853	U	0.834	U	0.853	U	0	NA	NA
11Cl-PF3OUdS	0.804	U	0.786	U	0.804	U	0	NA	NA
PFEESA	0.137	U	0.134	U	0.137	U	0	NA	NA
3:3 FTCA	0.706	U	0.69	U	0.706	U	0	NA	NA
5:3 FTCA	41.1		52.6		45.1		3	46.27	12.62
7:3 FTCA	5.82	U	5.69	U	5.82	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-7 WW#7 Native Concentration

Analyte	L33274-1		L33274-2		L33274-3		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	1.25	J	1.18	J	1.22	J	3	1.22	2.89
PFPeA	0.79	J	0.747	J	0.961	J	3	0.83	13.59
PFHxA	1.22	JQ	1.33	J	1.29	J	3	1.28	4.35
PFHpA	0.365	J	0.376	J	0.365	J	3	0.37	1.72
PFOA	1.17	J	1.27	J	1.28	J	3	1.24	4.91
PFNA	0.382	J	0.442	JQ	0.413	JQ	3	0.41	7.28
PFDA	0.329	U	0.311	U	0.333	U	0	NA	NA
PFUnA	0.259	U	0.245	U	0.262	U	0	NA	NA
PFDoA	0.379	U	0.358	U	0.383	U	0	NA	NA
PFTrDA	0.239	U	0.226	U	0.242	U	0	NA	NA
PFTeDA	0.539	JQ	0.444	JQ	0.45	JQ	3	0.48	11.14
PFBS	0.511	JQ	0.948	JQ	0.554	JQ	3	0.67	35.89
PFPeS	1.11	JQ	1.31	JQ	1.03	JQ	3	1.15	12.54
PFHxS	0.368	JQ	0.368	JQ	0.373	J	3	0.37	0.78
PFHpS	0.14	U	0.132	U	0.141	U	0	NA	NA
PFOS	0.485	J	0.713	J	0.538	J	3	0.58	20.62
PFNS	0.727	JQ	0.282	U	0.302	U	1	0.73	NA
PFDS	0.76	JQ	0.311	U	0.333	U	1	0.76	NA
PFDoS	0.18	U	0.169	U	0.181	U	0	NA	NA
4:2 FTS	2.27	U	2.15	U	2.3	U	0	NA	NA
6:2 FTS	3.96	U	3.74	U	4	U	0	NA	NA
8:2 FTS	4.7	UD	4.43	UD	4.75	UD	0	NA	NA
PFOSA	0.229	U	0.217	U	0.232	U	0	NA	NA
N-MeFOSA	0.199	U	0.188	U	0.202	U	0	NA	NA
N-EtFOSA	0.578	U	0.564	J	0.585	U	1	0.56	NA
N-MeFOSAA	0.588	U	0.555	U	0.595	U	0	NA	NA
N-EtFOSAA	0.319	U	0.301	U	0.323	U	0	NA	NA
N-MeFOSE	1.29	J	1.16	J	1.2	U	2	1.23	7.5
N-EtFOSE	1.57	J	1.21	J	1.14	J	3	1.31	17.66

Table C-7 WW#7 Native Concentration

Analyte	L33274-1		L33274-2		L33274-3		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.409	U	0.386	U	0.413	U	0	NA	NA
ADONA	0.778	U	0.734	U	0.786	U	0	NA	NA
PFMPA	0.18	U	0.169	U	0.181	U	0	NA	NA
PFMBA	0.12	U	0.113	U	0.121	U	0	NA	NA
NFDHA	1.38	U	1.3	U	1.39	U	0	NA	NA
9Cl-PF3ONS	0.868	U	0.819	U	0.877	U	0	NA	NA
11Cl-PF3OUdS	0.818	U	0.772	U	0.827	U	0	NA	NA
PFEESA	0.14	U	0.132	U	0.141	U	0	NA	NA
3:3 FTCA	0.718	U	0.678	U	0.726	U	0	NA	NA
5:3 FTCA	5.06	U	4.77	U	5.11	U	0	NA	NA
7:3 FTCA	5.92	U	5.59	U	5.99	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-8 WW#8 Native Concentration

Analyte	L33040-1		L33040-2		L33040-19		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	1.69	J	1.69	J	1.56	J	3	1.65	4.56
PFPeA	1.86	J	2.03	J	2.02	J	3	1.97	4.84
PFHxA	2.17		2.3		2.1		3	2.19	4.63
PFHpA	1.74		2.16		1.88		3	1.93	11.1
PFOA	4.6		4.75		4.35		3	4.57	4.42
PFNA	1.75		1.69		1.55	J	3	1.66	6.17
PFDA	3.52		3.46		3.33		3	3.44	2.83
PFUnA	0.614	J	0.578	J	0.555	J	3	0.58	5.11
PFDoA	1.76		1.77		1.52	J	3	1.68	8.41
PFTrDA	0.244	J	0.236	J	0.246	U	2	0.24	2.36
PFTeDA	0.25	U	0.251	U	0.359	JQ	1	0.36	NA
PFBS	1.57		1.68		1.59	J	3	1.61	3.63
PFPeS	0.192	U	0.193	U	0.205	U	0	NA	NA
PFHxS	0.702	J	0.608	J	0.695	J	3	0.67	7.84
PFHpS	0.134	U	0.135	U	0.143	U	0	NA	NA
PFOS	2.71		2.93		2.84		3	2.83	3.91
PFNS	0.288	U	0.289	U	0.307	U	0	NA	NA
PFDS	0.317	U	0.318	U	0.338	U	0	NA	NA
PFDoS	0.173	U	0.174	U	0.184	U	0	NA	NA
4:2 FTS	2.19	U	2.2	U	2.34	U	0	NA	NA
6:2 FTS	4.88	J	3.83	U	4.07	U	1	4.88	NA
8:2 FTS	1.51	U	1.51	U	1.61	U	0	NA	NA
PFOSA	1.99		1.63		1.79		3	1.8	10
N-MeFOSA	0.192	U	0.193	U	0.205	U	0	NA	NA
N-EtFOSA	0.557	U	0.559	U	0.594	U	0	NA	NA
N-MeFOSAA	0.566	U	0.569	U	0.605	U	0	NA	NA
N-EtFOSAA	0.307	U	0.309	U	0.328	U	0	NA	NA
N-MeFOSE	1.14	U	1.15	U	1.22	U	0	NA	NA
N-EtFOSE	0.979	U	0.983	U	1.05	U	0	NA	NA

Table C-8 WW#8 Native Concentration

Analyte	L33040-1		L33040-2		L33040-19		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.394	U	0.395	U	0.42	U	0	NA	NA
ADONA	0.749	U	0.752	U	0.799	U	0	NA	NA
PFMPA	0.173	U	0.174	U	0.184	U	0	NA	NA
PFMBA	0.115	U	0.116	U	0.123	U	0	NA	NA
NFDHA	1.32	U	1.33	U	1.41	U	0	NA	NA
9Cl-PF3ONS	0.835	U	0.839	U	0.892	U	0	NA	NA
11Cl-PF3OUdS	0.787	U	0.791	U	0.84	U	0	NA	NA
PFEESA	0.134	U	0.135	U	0.143	U	0	NA	NA
3:3 FTCA	0.691	U	0.694	U	0.738	U	0	NA	NA
5:3 FTCA	4.87	U	4.89	U	5.2	U	0	NA	NA
7:3 FTCA	5.7	U	5.73	U	6.09	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

C-9 WW#9 Native Concentration

Analyte	L33041-4		L33041-5		L33041-6		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	1.8	J	1.03	J	1.4	J	3	1.41	27.31
PFPeA	1.09	J	2.22	J	1.39	J	3	1.57	37.36
PFHxA	2.99		3.43		3.21		3	3.21	6.85
PFHpA	0.549	J	0.602	J	0.411	J	3	0.52	18.94
PFOA	1.97		2.07		1.87		3	1.97	5.08
PFNA	0.403	J	0.473	J	0.518	J	3	0.46	12.47
PFDA	0.323	U	0.471	JQ	0.338	JQ	2	0.4	23.25
PFUnA	0.29	J	0.254	U	0.253	U	1	0.29	NA
PFDoA	0.372	U	0.371	U	0.369	U	0	NA	NA
PFTrDA	0.235	U	0.235	U	0.233	U	0	NA	NA
PFTeDA	0.342	J	0.254	U	0.354	JQ	2	0.35	2.44
PFBS	1.61	Q	1.79	Q	1.43	JQ	3	1.61	11.18
PFPeS	2.14	Q	1.89	Q	1.82	Q	3	1.95	8.63
PFHxS	0.555	J	0.459	J	0.578	J	3	0.53	11.89
PFHpS	0.137	U	0.137	U	0.136	U	0	NA	NA
PFOS	1.32	J	2.22		2.14		3	1.89	26.31
PFNS	0.294	U	0.293	U	0.292	U	0	NA	NA
PFDS	0.323	U	0.323	U	0.321	U	0	NA	NA
PFDoS	0.176	U	0.176	U	0.175	U	0	NA	NA
4:2 FTS	2.23	U	2.23	U	2.22	U	0	NA	NA
6:2 FTS	3.89	U	3.88	U	3.86	U	0	NA	NA
8:2 FTS	1.54	U	1.53	U	1.53	U	0	NA	NA
PFOSA	2.68		3.11		2.18		3	2.66	17.52
N-MeFOSA	0.196	U	0.195	U	0.194	U	0	NA	NA
N-EtFOSA	0.568	U	0.567	U	0.564	U	0	NA	NA
N-MeFOSAA	0.578	U	0.577	U	0.574	U	0	NA	NA
N-EtFOSAA	0.562	J	0.33	J	0.478	J	3	0.46	25.72
N-MeFOSE	1.17	U	1.16	U	1.16	U	0	NA	NA
N-EtFOSE	0.999	U	0.997	U	0.992	U	0	NA	NA

C-9 WW#9 Native Concentration

Analyte	L33041-4		L33041-5		L33041-6		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.402	U	0.401	U	0.399	U	0	NA	NA
ADONA	0.764	U	0.762	U	0.758	U	0	NA	NA
PFMPA	0.176	U	0.176	U	0.175	U	0	NA	NA
PFMBA	0.118	U	0.117	U	0.117	U	0	NA	NA
NFDHA	1.35	U	1.35	U	1.34	U	0	NA	NA
9Cl-PF3ONS	0.852	U	0.85	U	0.846	U	0	NA	NA
11Cl-PF3OUdS	0.803	U	0.801	U	0.797	U	0	NA	NA
PFEESA	0.137	U	0.137	U	0.136	U	0	NA	NA
3:3 FTCA	0.705	U	0.704	U	0.7	U	0	NA	NA
5:3 FTCA	4.97	U	4.95	U	4.93	U	0	NA	NA
7:3 FTCA	5.82	U	5.81	U	5.78	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-10 WW#10 Native Concentration

Analyte	L33042-7		L33042-8		L33042-9		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	6.4		6.54		6.54		3	6.49	1.24
PFPeA	5		5.16		5.42		3	5.19	4.08
PFHxA	13.3		13.5		14.7		3	13.83	5.47
PFHpA	1.42	J	1.6		1.89		3	1.64	14.49
PFOA	4.22		4.13		4.47		3	4.27	4.12
PFNA	0.801	J	0.654	J	0.787	J	3	0.75	10.86
PFDA	1.53	J	1.23	J	1.22	J	3	1.33	13.28
PFUnA	0.249	U	0.252	U	0.253	U	0	NA	NA
PFDoA	0.364	U	0.368	U	0.37	U	0	NA	NA
PFTrDA	0.23	U	0.232	U	0.234	U	0	NA	NA
PFTeDA	0.249	U	0.252	U	0.253	U	0	NA	NA
PFBS	6.98		7.16		7.25		3	7.13	1.93
PFPeS	0.192	U	0.228	J	0.195	U	1	0.23	NA
PFHxS	2.05		1.78		2.21	Q	3	2.01	10.79
PFHpS	0.134	U	0.135	U	0.136	U	0	NA	NA
PFOS	18.3		14.6		15.5		3	16.13	11.96
PFNS	0.287	U	0.29	U	0.292	U	0	NA	NA
PFDS	0.316	U	0.319	U	0.322	U	0	NA	NA
PFDoS	0.172	U	0.174	U	0.175	U	0	NA	NA
4:2 FTS	2.18	U	2.21	U	2.22	U	0	NA	NA
6:2 FTS	12.3		11.6		12.4		3	12.1	3.6
8:2 FTS	1.5	U	1.52	U	1.53	U	0	NA	NA
PFOSA	1.11	J	0.812	J	1.14	J	3	1.02	17.77
N-MeFOSA	0.192	U	0.193	U	0.195	U	0	NA	NA
N-EtFOSA	0.556	U	0.561	U	0.565	U	0	NA	NA
N-MeFOSAA	1.7		1.52	J	2.01	Q	3	1.74	14.22
N-EtFOSAA	0.625	J	0.893	J	0.884	JQ	3	0.8	19.01
N-MeFOSE	1.14	U	1.15	U	1.16	U	0	NA	NA
N-EtFOSE	0.977	U	0.987	U	0.994	U	0	NA	NA

Table C-10 WW#10 Native Concentration

Analyte	L33042-7		L33042-8		L33042-9		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.393	U	0.397	U	0.4	U	0	NA	NA
ADONA	0.747	U	0.755	U	0.76	U	0	NA	NA
PFMPA	0.172	U	0.174	U	0.175	U	0	NA	NA
PFMBA	0.115	U	0.116	U	0.117	U	0	NA	NA
NFDHA	1.32	U	1.33	U	1.34	U	0	NA	NA
9Cl-PF3ONS	0.833	U	0.842	U	0.848	U	0	NA	NA
11Cl-PF3OUdS	0.785	U	0.793	U	0.799	U	0	NA	NA
PFEESA	0.134	U	0.135	U	0.136	U	0	NA	NA
3:3 FTCA	0.69	U	0.696	U	0.702	U	0	NA	NA
5:3 FTCA	26.6	J	26.3	J	30	J	3	27.63	7.44
7:3 FTCA	5.69	U	5.75	U	5.79	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-11. SW COP-SW01 to 11 Native Concentration

Analyte	L33079-7		L33079-8		L33079-9		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	4.06	J	3.87	J	3.99	J	3	3.97	2.42
PFPeA	3.44		3.31		3.45		3	3.4	2.3
PFHxA	5.45		5.27		5.26		3	5.33	2.01
PFHpA	3.62		3.57		3.61		3	3.6	0.73
PFOA	19.2		19.1		19.6		3	19.3	1.37
PFNA	2.13		1.98		2.07		3	2.06	3.66
PFDA	0.436	J	0.366	J	0.393	J	3	0.4	8.86
PFUnA	0.33	J	0.262	J	0.274	J	3	0.29	12.57
PFDoA	0.471	J	0.401	J	0.452	J	3	0.44	8.2
PFTrDA	0.933	J	0.947	J	1.01	J	3	0.96	4.26
PFTeDA	0.526	J	0.454	J	0.42	J	3	0.47	11.6
PFBS	7.38		6.89		7.4		3	7.22	4
PFPeS	6.06		6.19		5.79		3	6.01	3.39
PFHxS	50.5		47.9		48.1		3	48.83	2.96
PFHpS	3.14		3.08		3.13		3	3.12	1.03
PFOS	99.2		94.9		96.2		3	96.77	2.28
PFNS	0.282	U	0.278	U	0.283	U	0	NA	NA
PFDS	0.31	U	0.306	U	0.311	U	0	NA	NA
PFDoS	0.169	U	0.167	U	0.17	U	0	NA	NA
4:2 FTS	2.14	U	2.11	U	2.15	U	0	NA	NA
6:2 FTS	3.73	U	3.68	U	3.74	U	0	NA	NA
8:2 FTS	1.48	U	1.45	U	1.48	U	0	NA	NA
PFOSA	0.826	J	0.917	J	1.23	J	3	0.99	21.38
N-MeFOSA	0.188	U	0.185	U	0.189	U	0	NA	NA
N-EtFOSA	0.546	U	0.537	U	0.547	U	0	NA	NA
N-MeFOSAA	0.555	U	0.546	U	0.556	U	0	NA	NA
N-EtFOSAA	0.301	U	0.296	U	0.302	U	0	NA	NA
N-MeFOSE	1.12	U	1.1	U	1.12	U	0	NA	NA
N-EtFOSE	0.959	U	0.945	U	0.962	U	0	NA	NA

Table C-11. SW COP-SW01 to 11 Native Concentration

Analyte	L33079-7		L33079-8		L33079-9		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.386	U	0.38	U	0.387	U	0	NA	NA
ADONA	0.734	U	0.722	U	0.735	U	0	NA	NA
PFMPA	0.169	U	0.167	U	0.17	U	0	NA	NA
PFMBA	0.113	U	0.111	U	0.113	U	0	NA	NA
NFDHA	1.3	U	1.28	U	1.3	U	0	NA	NA
9Cl-PF3ONS	0.818	U	0.806	U	0.82	U	0	NA	NA
11Cl-PF3OUdS	0.771	U	0.759	U	0.773	U	0	NA	NA
PFEESA	0.132	U	0.13	U	0.132	U	0	NA	NA
3:3 FTCA	0.677	U	0.667	U	0.679	U	0	NA	NA
5:3 FTCA	4.77	U	4.7	U	4.78	U	0	NA	NA
7:3 FTCA	5.59	U	5.5	U	5.6	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-12 SW RP-SW01 to 11 Native Concentration

Analyte							Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	3.86	J	3.83	J	3.83	J	3	3.84	0.45
PFPeA	8.02		8.02		7.98		3	8.01	0.29
PFHxA	9.19		9.53		9.72		3	9.48	2.83
PFHpA	7.08		6.88		6.89		3	6.95	1.62
PFOA	12.6		12.7		12.8		3	12.7	0.79
PFNA	3.95		3.84		3.96		3	3.92	1.7
PFDA	0.334	U	0.398	J	0.358	J	2	0.38	7.48
PFUnA	0.263	U	0.252	U	0.256	U	0	NA	NA
PFDoA	0.384	U	0.368	U	0.375	U	0	NA	NA
PFTrDA	0.243	U	0.233	U	0.237	U	0	NA	NA
PFTeDA	0.263	U	0.252	U	0.256	U	0	NA	NA
PFBS	4.94		4.62		4.72		3	4.76	3.44
PFPeS	5.34		5		5.35		3	5.23	3.81
PFHxS	59.9		61.4		60.9		3	60.73	1.26
PFHpS	2.34		2.34		2.28		3	2.32	1.49
PFOS	204		214		207		3	208.33	2.46
PFNS	0.304	U	0.291	U	0.296	U	0	NA	NA
PFDS	0.334	U	0.32	U	0.326	U	0	NA	NA
PFDoS	0.182	U	0.174	U	0.178	U	0	NA	NA
4:2 FTS	2.31	U	2.21	U	2.25	U	0	NA	NA
6:2 FTS	9.04	B	3.85	U	3.92	U	0	NA	NA
8:2 FTS	1.59	U	1.52	U	1.55	U	0	NA	NA
PFOSA	0.419	J	0.488	J	0.689	J	3	0.53	26.37
N-MeFOSA	0.202	U	0.194	U	0.197	U	0	NA	NA
N-EtFOSA	0.587	U	0.562	U	0.572	U	0	NA	NA
N-MeFOSAA	0.597	U	0.572	U	0.582	U	0	NA	NA
N-EtFOSAA	0.324	U	0.31	U	0.316	U	0	NA	NA
N-MeFOSE	1.2	U	1.15	U	1.17	U	0	NA	NA
N-EtFOSE	1.03	U	0.989	U	1.01	U	0	NA	NA

Table C-12 SW RP-SW01 to 11 Native Concentration

Analyte							Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.415	U	0.397	U	0.404	U	0	NA	NA
ADONA	0.789	U	0.756	U	0.769	U	0	NA	NA
PFMPA	0.182	U	0.174	U	0.178	U	0	NA	NA
PFMBA	0.121	U	0.116	U	0.118	U	0	NA	NA
NFDHA	1.4	U	1.34	U	1.36	U	0	NA	NA
9Cl-PF3ONS	0.88	U	0.843	U	0.858	U	0	NA	NA
11Cl-PF3OUdS	0.83	U	0.795	U	0.809	U	0	NA	NA
PFEESA	0.142	U	0.136	U	0.138	U	0	NA	NA
3:3 FTCA	0.728	U	0.698	U	0.71	U	0	NA	NA
5:3 FTCA	5.13	U	4.91	U	5	U	0	NA	NA
7:3 FTCA	6.01	U	5.76	U	5.86	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-13 SW Marine Surface Water Native Concentration

Analyte	L33078-10		L33078-11		L33078-1		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	1.23	J	1.29	J	2	J	3	1.51	28.43
PFPeA	0.459	J	0.387	J	0.399	J	3	0.42	9.3
PFHxA	0.291	U	0.292	U	0.287	U	0	NA	NA
PFHpA	0.2	U	0.201	U	0.197	U	0	NA	NA
PFOA	0.273	U	0.274	U	0.271	J	1	0.27	NA
PFNA	0.2	U	0.201	U	0.197	U	0	NA	NA
PFDA	0.3	U	0.301	U	0.296	U	0	NA	NA
PFUnA	0.236	U	0.237	U	0.233	U	0	NA	NA
PFDoA	0.345	U	0.347	U	0.34	U	0	NA	NA
PFTrDA	0.218	U	0.219	U	0.215	U	0	NA	NA
PFTeDA	0.236	U	0.237	U	0.233	U	0	NA	NA
PFBS	0.227	U	0.228	U	0.224	U	0	NA	NA
PFPeS	0.182	U	0.183	U	0.179	U	0	NA	NA
PFHxS	0.2	U	0.201	U	0.197	U	0	NA	NA
PFHpS	0.127	U	0.128	U	0.125	U	0	NA	NA
PFOS	0.3	U	0.301	U	0.296	U	0	NA	NA
PFNS	0.273	U	0.274	U	0.269	U	0	NA	NA
PFDS	0.3	U	0.301	U	0.296	U	0	NA	NA
PFDoS	0.164	U	0.164	U	0.161	U	0	NA	NA
4:2 FTS	2.07	U	2.08	U	2.04	U	0	NA	NA
6:2 FTS	3.61	U	4.65	J	3.56	U	1	4.65	NA
8:2 FTS	1.43	U	1.43	U	1.41	U	0	NA	NA
PFOSA	0.41	J	0.478	J	0.479	J	3	0.46	8.68
N-MeFOSA	0.182	U	0.183	U	0.179	U	0	NA	NA
N-EtFOSA	0.527	U	0.529	U	0.52	U	0	NA	NA
N-MeFOSAA	0.536	U	0.539	U	0.529	U	0	NA	NA
N-EtFOSAA	0.291	U	0.292	U	0.287	U	0	NA	NA
N-MeFOSE	1.08	U	1.09	U	1.07	U	0	NA	NA
N-EtFOSE	0.927	U	0.931	U	0.914	U	0	NA	NA

Table C-13 SW Marine Surface Water Native Concentration

Analyte	L33078-10		L33078-11		L33078-1		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	0.373	U	0.374	U	0.367	U	0	NA	NA
ADONA	0.709	U	0.712	U	0.699	U	0	NA	NA
PFMPA	0.164	U	0.164	U	0.161	U	0	NA	NA
PFMBA	0.109	U	0.11	U	0.108	U	0	NA	NA
NFDHA	1.25	U	1.26	U	1.24	U	0	NA	NA
9Cl-PF3ONS	0.79	U	0.794	U	0.779	U	0	NA	NA
11Cl-PF3OUdS	0.745	U	0.748	U	0.735	U	0	NA	NA
PFEESA	0.127	U	0.128	U	0.125	U	0	NA	NA
3:3 FTCA	0.654	U	0.657	U	0.645	U	0	NA	NA
5:3 FTCA	4.61	U	4.63	U	4.54	U	0	NA	NA
7:3 FTCA	5.4	U	5.42	U	5.32	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-14 LCH C & D Landfill Leachate Native Concentration

Analyte							Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	80.6		82.3		84.4		3	82.43	2.31
PFPeA	439		435		438		3	437.33	0.48
PFHxA	239		252		268		3	253	5.74
PFHpA	76.2		73.9		74.6		3	74.9	1.57
PFOA	125		128		122		3	125	2.4
PFNA	6.51	J	7.41		6.77	J	3	6.9	6.72
PFDA	1.8	J	2.56	J	2.2	J	3	2.19	17.39
PFUnA	1.07	U	1.08	U	1.15	U	0	NA	NA
PFDoA	1.57	U	1.58	U	1.69	U	0	NA	NA
PFTrDA	0.989	U	0.997	U	1.07	U	0	NA	NA
PFTeDA	1.07	U	1.08	U	1.15	U	0	NA	NA
PFBS	45.2		44.6		47.1		3	45.63	2.86
PFPeS	3.3	J	4.48	J	3.61	J	3	3.8	16.11
PFHxS	33.3		33.9		32.2		3	33.13	2.6
PFHpS	1.31	J	1.4	J	1.41	J	3	1.37	4.01
PFOS	43.4		45.1		40.1		3	42.87	5.93
PFNS	1.24	U	1.25	U	1.33	U	0	NA	NA
PFDS	1.36	U	1.37	U	1.47	U	0	NA	NA
PFDoS	0.742	U	0.748	U	0.799	U	0	NA	NA
4:2 FTS	9.39	U	9.47	U	10.1	U	0	NA	NA
6:2 FTS	29.9		27.7		30.8		3	29.47	5.41
8:2 FTS	6.47	U	6.52	U	6.97	U	0	NA	NA
PFOSA	4.53	J	3.63	J	4.23	J	3	4.13	11.1
N-MeFOSA	0.824	U	0.831	U	0.888	U	0	NA	NA
N-EtFOSA	2.39	U	2.41	U	2.58	U	0	NA	NA
N-MeFOSAA	2.51	J	2.57	JQ	2.62	U	2	2.54	1.67
N-EtFOSAA	1.32	U	1.33	U	1.42	U	0	NA	NA
N-MeFOSE	4.9	U	4.94	U	5.28	U	0	NA	NA
N-EtFOSE	4.2	U	4.24	U	4.53	U	0	NA	NA

Table C-14 LCH C & D Landfill Leachate Native Concentration

Analyte							Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	1.69	U	1.7	U	1.82	U	0	NA	NA
ADONA	3.21	U	3.24	U	3.46	U	0	NA	NA
PFMPA	0.742	U	0.748	U	0.799	U	0	NA	NA
PFMBA	0.494	U	0.498	U	0.533	U	0	NA	NA
NFDHA	5.69	U	5.73	U	6.13	U	0	NA	NA
9Cl-PF3ONS	3.58	U	3.61	U	3.86	U	0	NA	NA
11Cl-PF3OUdS	3.38	U	3.41	U	3.64	U	0	NA	NA
PFEESA	0.577	U	0.581	U	0.622	U	0	NA	NA
3:3 FTCA	2.97	U	2.99	U	3.2	U	0	NA	NA
5:3 FTCA	95.6	J	96.4	J	113	J	3	101.67	9.66
7:3 FTCA	24.5	U	24.7	U	26.4	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-15 LCH MSW Landfill Leachate Native Concentration

Analyte	L33081-1		L33081-2		L33081-7		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
PFBA	1620		1600		1610		3	1610	0.62
PFPeA	1250		1280		1210		3	1246.67	2.82
PFHxA	3120		3140		3320		3	3193.33	3.45
PFHpA	617		629		635		3	627	1.46
PFOA	1350		1340		1340		3	1343.33	0.43
PFNA	82.7		83.5		80.3		3	82.17	2.03
PFDA	113		120		123		3	118.67	4.32
PFUnA	12		13.2		12.6		3	12.6	4.76
PFDoA	31.7		30.3		30.9		3	30.97	2.27
PFTrDA	7.79		6.66		6.75		3	7.07	8.89
PFTeDA	17.6		15.9		13.8		3	15.77	12.07
PFBS	1490		1450		1540		3	1493.33	3.02
PFPeS	20.9		23		23.4		3	22.43	5.99
PFHxS	777		774		789		3	780	1.02
PFHpS	3.87	J	4.45	J	4.54	J	3	4.29	8.48
PFOS	218		218		215		3	217	0.8
PFNS	1.24	U	1.23	U	1.25	U	0	NA	NA
PFDS	4.74	J	4.81	J	5.34	J	3	4.96	6.61
PFDoS	0.746	U	0.739	U	0.751	U	0	NA	NA
4:2 FTS	9.45	U	9.36	U	9.51	U	0	NA	NA
6:2 FTS	185		181		182		3	182.67	1.14
8:2 FTS	56.5		63.3		60.4		3	60.07	5.68
PFOSA	33.5		34		34.9		3	34.13	2.08
N-MeFOSA	11.4		12.8		12.8		3	12.33	6.55
N-EtFOSA	22.8		22.4		25.3		3	23.5	6.69
N-MeFOSAA	307		308		317		3	310.67	1.77
N-EtFOSAA	252		260		238		3	250	4.45
N-MeFOSE	2610		2680		2750		3	2680	2.61
N-EtFOSE	2130		2160		2170		3	2153.33	0.97

Table C-15 LCH MSW Landfill Leachate Native Concentration

Analyte	L33081-1		L33081-2		L33081-7		Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	10.1	JQ	11.5	JQ	11.8	J	3	11.13	8.15
ADONA	3.23	U	3.2	U	3.25	U	0	NA	NA
PFMPA	0.796	J	0.739	U	0.751	U	1	0.8	NA
PFMBA	1.44	J	2.54	J	1.53	J	3	1.84	33.25
NFDHA	6.43	JQ	5.67	U	5.76	U	1	6.43	NA
9Cl-PF3ONS	3.61	U	3.57	U	3.63	U	0	NA	NA
11Cl-PF3OUdS	3.4	U	3.37	U	3.42	U	0	NA	NA
PFEESA	9.41		7.61		9.81		3	8.94	13.1
3:3 FTCA	33.1		42.5		41.1		3	38.9	13.04
5:3 FTCA	7610		8820		8260		3	8230	7.36
7:3 FTCA	1620		2050		1890		3	1853.33	11.73

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-16 LCH MSW Incineration Ash Landfill Leachate Native Concentration

Analyte	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Number of Detections	Mean (ng/L)	RSD [1] (%)
PFBA	51.9		52.1		51.7		3	51.9	0.39
PFPeA	52.9		54.5		54.8		3	54.07	1.89
PFHxA	50.5		56.5		54.4		3	53.8	5.66
PFHpA	4.39	J	4.87	J	4.69	J	3	4.65	5.21
PFOA	5.47	J	5.56	J	5.63	J	3	5.55	1.44
PFNA	0.899	U	0.846	U	0.854	U	0	NA	NA
PFDA	1.35	U	1.27	U	1.28	U	0	NA	NA
PFUnA	1.06	U	1	U	1.01	U	0	NA	NA
PFDoA	1.55	U	1.46	U	1.47	U	0	NA	NA
PFTrDA	0.98	U	0.923	U	0.932	U	0	NA	NA
PFTeDA	1.06	U	1	U	1.01	U	0	NA	NA
PFBS	53.9		52.5		52.4		3	52.93	1.58
PFPeS	0.817	U	0.769	U	0.776	U	0	NA	NA
PFHxS	3.31	J	3.09	J	3.62	J	3	3.34	7.97
PFHpS	0.572	U	0.539	U	0.543	U	0	NA	NA
PFOS	1.35	U	1.43	J	1.28	U	1	1.43	NA
PFNS	1.23	U	1.15	U	1.16	U	0	NA	NA
PFDS	1.35	U	1.27	U	1.28	U	0	NA	NA
PFDoS	0.735	U	0.693	U	0.699	U	0	NA	NA
4:2 FTS	9.31	U	8.77	U	8.85	U	0	NA	NA
6:2 FTS	16.2	U	15.3	U	15.4	U	0	NA	NA
8:2 FTS	6.41	U	6.04	U	6.09	U	0	NA	NA
PFOSA	0.94	U	0.885	U	0.893	U	0	NA	NA
N-MeFOSA	0.817	U	0.769	U	0.776	U	0	NA	NA
N-EtFOSA	2.37	U	2.23	U	2.25	U	0	NA	NA
N-MeFOSAA	2.41	U	2.27	U	2.38	J	1	2.38	NA
N-EtFOSAA	1.48	JQ	1.23	U	1.48	JQ	2	1.48	0
N-MeFOSE	4.86	U	4.58	U	4.62	U	0	NA	NA
N-EtFOSE	4.17	U	3.92	U	3.96	U	0	NA	NA

Table C-16 LCH MSW Incineration Ash Landfill Leachate Native Concentration

Analyte							Number of Detections	Mean (ng/L)	RSD [1] (%)
	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier	Concentration (ng/L)	Qualifier			
HFPO-DA	1.67	U	1.58	U	1.59	U	0	NA	NA
ADONA	3.19	U	3	U	3.03	U	0	NA	NA
PFMPA	0.735	U	0.693	U	0.699	U	0	NA	NA
PFMBA	0.49	U	0.462	U	0.466	U	0	NA	NA
NFDHA	5.64	U	5.31	U	5.36	U	0	NA	NA
9Cl-PF3ONS	3.55	U	3.35	U	3.38	U	0	NA	NA
11Cl-PF3OUdS	3.35	U	3.15	U	3.18	U	0	NA	NA
PFEESA	0.572	U	0.539	U	0.543	U	0	NA	NA
3:3 FTCA	2.94	U	2.77	U	2.79	U	0	NA	NA
5:3 FTCA	20.7	U	19.5	U	19.7	U	0	NA	NA
7:3 FTCA	24.3	U	22.9	U	23.1	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-17 Soil 2014-107 Native Concentration

Analyte	L33301-19		L33301-20		L33301-21		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.402	U	0.405	U	0.403	U	0	NA	NA
PFPeA	0.529		0.544		0.52		3	0.53	2.28
PFHxA	0.461		0.482		0.419		3	0.45	7.07
PFHpA	0.343		0.36		0.345		3	0.35	2.66
PFOA	1.87		1.98		1.83		3	1.89	4.1
PFNA	0.586		0.631		0.612		3	0.61	3.71
PFDA	2.27		2.43		2.45		3	2.38	4.14
PFUnA	0.249		0.271		0.213		3	0.24	11.98
PFDoA	0.688		0.62		0.636		3	0.65	5.49
PFTrDA	0.084	J	0.078	J	0.075	J	3	0.08	5.8
PFTeDA	0.175		0.172		0.199		3	0.18	8.13
PFBS	0.014	U	0.0141	U	0.0141	U	0	NA	NA
PFPeS	0.015	U	0.0151	U	0.0151	U	0	NA	NA
PFHxS	0.179		0.17		0.184		3	0.18	3.99
PFHpS	0.0572	U	0.0575	U	0.0573	U	0	NA	NA
PFOS	1.09		1.11		1.09		3	1.1	1.05
PFNS	0.0461	U	0.0464	U	0.0462	U	0	NA	NA
PFDS	0.172	Q	0.185	Q	0.185	Q	3	0.18	4.15
PFDoS	0.0381	U	0.0383	U	0.0382	U	0	NA	NA
4:2 FTS	0.283	U	0.284	U	0.283	U	0	NA	NA
6:2 FTS	4.21		2.77		0.117	U	2	3.49	29.18
8:2 FTS	0.226	U	0.227	U	0.226	U	0	NA	NA
PFOSA	0.0682	U	0.0686	U	0.0683	U	0	NA	NA
N-MeFOSA	0.0491	U	0.0494	U	0.0492	U	0	NA	NA
N-EtFOSA	0.0381	U	0.0383	U	0.0382	U	0	NA	NA
N-MeFOSAA	0.0301	U	0.0303	U	0.0301	U	0	NA	NA
N-EtFOSAA	0.071	J	0.064	J	0.05	J	3	0.06	17.34
N-MeFOSE	0.204	U	0.205	U	0.204	U	0	NA	NA
N-EtFOSE	0.248	U	0.249	U	0.248	U	0	NA	NA

Table C-17 Soil 2014-107 Native Concentration

Analyte	L33301-19		L33301-20		L33301-21		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.136	U	0.137	U	0.137	U	0	NA	NA
ADONA	0.0572	U	0.0575	U	0.0573	U	0	NA	NA
PFMPA	0.0331	U	0.0333	U	0.0332	U	0	NA	NA
PFMBA	0.0291	U	0.0293	U	0.0291	U	0	NA	NA
NFDHA	0.0842	U	0.0847	U	0.0844	U	0	NA	NA
9Cl-PF3ONS	0.0381	U	0.0383	U	0.0382	U	0	NA	NA
11Cl-PF3OUdS	0.0712	U	0.0716	U	0.0713	U	0	NA	NA
PFEESA	0.0181	U	0.0182	U	0.0181	U	0	NA	NA
3:3 FTCA	0.0602	U	0.0605	U	0.0603	U	0	NA	NA
5:3 FTCA	0.364	U	0.366	U	0.365	U	0	NA	NA
7:3 FTCA	0.309	U	0.311	U	0.309	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-18 Soil 2016-106 Native Concentration

Analyte	L33302-22		L33302-23		L33302-24		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.415	U	0.415	U	0.414	U	0	NA	NA
PFPeA	0.031	J	0.037	J	0.036	J	3	0.03	9.27
PFHxA	0.042	J	0.032	J	0.042	J	3	0.04	14.93
PFHpA	0.03	U	0.03	U	0.0299	U	0	NA	NA
PFOA	0.045	J	0.049	J	0.057	J	3	0.05	12.14
PFNA	0.0889	U	0.0889	U	0.0888	U	0	NA	NA
PFDA	0.036	J	0.0321	U	0.032	U	1	0.04	NA
PFUnA	0.0341	U	0.0341	U	0.0341	U	0	NA	NA
PFDoA	0.061	U	0.061	U	0.0609	U	0	NA	NA
PFTrDA	0.0393	U	0.0393	U	0.0392	U	0	NA	NA
PFTeDA	0.0331	U	0.0331	U	0.033	U	0	NA	NA
PFBS	0.0145	U	0.0145	U	0.0144	U	0	NA	NA
PFPeS	0.0155	U	0.0155	U	0.0155	U	0	NA	NA
PFHxS	0.0186	U	0.0186	U	0.0186	U	0	NA	NA
PFHpS	0.0589	U	0.0589	U	0.0588	U	0	NA	NA
PFOS	0.122	J	0.13	J	0.117	J	3	0.12	5.33
PFNS	0.0476	U	0.0476	U	0.0475	U	0	NA	NA
PFDS	0.0414	U	0.0414	U	0.0413	U	0	NA	NA
PFDoS	0.0393	U	0.0393	U	0.0392	U	0	NA	NA
4:2 FTS	0.292	U	0.292	U	0.291	U	0	NA	NA
6:2 FTS	0.12	U	0.12	U	0.12	U	0	NA	NA
8:2 FTS	0.233	U	0.233	U	0.232	U	0	NA	NA
PFOSA	0.0703	U	0.0703	U	0.0702	U	0	NA	NA
N-MeFOSA	0.0507	U	0.0507	U	0.0506	U	0	NA	NA
N-EtFOSA	0.115	JQ	0.085	JQ	0.082	J	3	0.09	19.41
N-MeFOSAA	0.031	U	0.031	U	0.031	U	0	NA	NA
N-EtFOSAA	0.0455	U	0.0455	U	0.0454	U	0	NA	NA
N-MeFOSE	0.21	U	0.21	U	0.21	U	0	NA	NA
N-EtFOSE	0.255	U	0.255	U	0.255	U	0	NA	NA

Table C-18 Soil 2016-106 Native Concentration

Analyte	L33302-22		L33302-23		L33302-24		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.141	U	0.141	U	0.14	U	0	NA	NA
ADONA	0.0589	U	0.0589	U	0.0588	U	0	NA	NA
PFMPA	0.0341	U	0.0341	U	0.0341	U	0	NA	NA
PFMBA	0.03	U	0.03	U	0.0299	U	0	NA	NA
NFDHA	0.0869	U	0.0869	U	0.0867	U	0	NA	NA
9Cl-PF3ONS	0.0393	U	0.0393	U	0.0392	U	0	NA	NA
11Cl-PF3OUdS	0.0734	U	0.0734	U	0.0733	U	0	NA	NA
PFEESA	0.0186	U	0.0186	U	0.0186	U	0	NA	NA
3:3 FTCA	0.0621	U	0.0621	U	0.0619	U	0	NA	NA
5:3 FTCA	0.375	U	0.375	U	0.375	U	0	NA	NA
7:3 FTCA	0.319	U	0.319	U	0.318	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-19 Soil 2017-111 Native Concentration

Analyte	L33303-13		L33303-14		L33303-15		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.409	U	0.41	U	0.412	U	0	NA	NA
PFPeA	0.0214	U	0.027	J	0.0216	U	1	0.03	NA
PFHxA	0.026	J	0.0204	U	0.021	JQ	2	0.02	15.04
PFHpA	0.0296	U	0.0296	U	0.0298	U	0	NA	NA
PFOA	0.0377	U	0.057	J	0.056	J	2	0.06	1.25
PFNA	0.0877	U	0.0879	U	0.0884	U	0	NA	NA
PFDA	0.0316	U	0.0317	U	0.0319	U	0	NA	NA
PFUnA	0.0337	U	0.0337	U	0.0339	U	0	NA	NA
PFDoA	0.0602	U	0.0603	U	0.0607	U	0	NA	NA
PFTrDA	0.0388	U	0.0388	U	0.0391	U	0	NA	NA
PFTeDA	0.0326	U	0.0327	U	0.0329	U	0	NA	NA
PFBS	0.0143	U	0.0143	U	0.0144	U	0	NA	NA
PFPeS	0.0153	U	0.0153	U	0.0154	U	0	NA	NA
PFHxS	0.0184	U	0.0184	U	0.0185	U	0	NA	NA
PFHpS	0.0581	U	0.0582	U	0.0586	U	0	NA	NA
PFOS	0.0683	U	0.0685	U	0.0689	U	0	NA	NA
PFNS	0.0469	U	0.047	U	0.0473	U	0	NA	NA
PFDS	0.0408	U	0.0409	U	0.0411	U	0	NA	NA
PFDoS	0.0388	U	0.0388	U	0.0391	U	0	NA	NA
4:2 FTS	0.288	U	0.288	U	0.29	U	0	NA	NA
6:2 FTS	0.118	U	0.119	U	2.69		1	2.69	NA
8:2 FTS	0.229	U	0.23	U	0.231	U	0	NA	NA
PFOSA	0.0693	U	0.0695	U	0.0699	U	0	NA	NA
N-MeFOSA	0.05	U	0.0501	U	0.0504	U	0	NA	NA
N-EtFOSA	0.092	JQ	0.067	JQ	0.101	JQ	3	0.09	20.33
N-MeFOSAA	0.0306	U	0.0307	U	0.0308	U	0	NA	NA
N-EtFOSAA	0.0449	U	0.045	U	0.0452	U	0	NA	NA
N-MeFOSE	0.207	U	0.207	U	0.209	U	0	NA	NA
N-EtFOSE	0.252	U	0.252	U	0.254	U	0	NA	NA

Table C-19 Soil 2017-111 Native Concentration

Analyte	L33303-13		L33303-14		L33303-15		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.139	U	0.139	U	0.14	U	0	NA	NA
ADONA	0.0581	U	0.0582	U	0.0586	U	0	NA	NA
PFMPA	0.0337	U	0.0337	U	0.0339	U	0	NA	NA
PFMBA	0.0296	U	0.0296	U	0.0298	U	0	NA	NA
NFDHA	0.0857	U	0.0858	U	0.0864	U	0	NA	NA
9Cl-PF3ONS	0.0388	U	0.0388	U	0.0391	U	0	NA	NA
11Cl-PF3OUdS	0.0724	U	0.0726	U	0.073	U	0	NA	NA
PFEESA	0.0184	U	0.0184	U	0.0185	U	0	NA	NA
3:3 FTCA	0.0612	U	0.0613	U	0.0617	U	0	NA	NA
5:3 FTCA	0.37	U	0.371	U	0.373	U	0	NA	NA
7:3 FTCA	0.314	U	0.315	U	0.317	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-20 Soil 2018-105 Native Concentration

Analyte	L33304-13		L33304-14		L33304-15		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.406	U	0.405	U	0.405	U	0	NA	NA
PFPeA	0.0213	U	0.026	J	0.025	J	2	0.03	2.77
PFHxA	0.0203	U	0.0202	U	0.025	JQ	1	0.03	NA
PFHpA	0.0294	U	0.0293	U	0.0293	U	0	NA	NA
PFOA	0.0375	U	0.0373	U	0.0373	U	0	NA	NA
PFNA	0.0872	U	0.0868	U	0.0868	U	0	NA	NA
PFDA	0.0314	U	0.0313	U	0.0313	U	0	NA	NA
PFUnA	0.0334	U	0.0333	U	0.0333	U	0	NA	NA
PFDoA	0.0598	U	0.0596	U	0.0596	U	0	NA	NA
PFTrDA	0.0385	U	0.0384	U	0.0384	U	0	NA	NA
PFTeDA	0.0324	U	0.0323	U	0.0323	U	0	NA	NA
PFBS	0.0142	U	0.0141	U	0.0141	U	0	NA	NA
PFPeS	0.0152	U	0.0151	U	0.0151	U	0	NA	NA
PFHxS	0.0182	U	0.0182	U	0.021	J	1	0.02	NA
PFHpS	0.0578	U	0.0575	U	0.0575	U	0	NA	NA
PFOS	0.0679	U	0.0676	U	0.0676	U	0	NA	NA
PFNS	0.0466	U	0.0464	U	0.0464	U	0	NA	NA
PFDS	0.0405	U	0.0404	U	0.0404	U	0	NA	NA
PFDoS	0.0385	U	0.0384	U	0.0384	U	0	NA	NA
4:2 FTS	0.286	U	0.285	U	0.285	U	0	NA	NA
6:2 FTS	5.66		0.117	U	0.117	U	1	5.66	NA
8:2 FTS	0.228	U	0.227	U	0.227	U	0	NA	NA
PFOSA	0.0689	U	0.0686	U	0.0686	U	0	NA	NA
N-MeFOSA	0.0497	U	0.0495	U	0.0495	U	0	NA	NA
N-EtFOSA	0.0385	U	0.0384	U	0.0384	U	0	NA	NA
N-MeFOSAA	0.0304	U	0.0303	U	0.0303	U	0	NA	NA
N-EtFOSAA	0.0446	U	0.0444	U	0.0444	U	0	NA	NA
N-MeFOSE	0.206	U	0.205	U	0.205	U	0	NA	NA
N-EtFOSE	0.25	U	0.249	U	0.249	U	0	NA	NA

Table C-20 Soil 2018-105 Native Concentration

Analyte	L33304-13		L33304-14		L33304-15		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.138	U	0.137	U	0.137	U	0	NA	NA
ADONA	0.0578	U	0.0575	U	0.0575	U	0	NA	NA
PFMPA	0.0334	U	0.0333	U	0.0333	U	0	NA	NA
PFMBA	0.0294	U	0.0293	U	0.0293	U	0	NA	NA
NFDHA	0.0851	U	0.0848	U	0.0848	U	0	NA	NA
9Cl-PF3ONS	0.0385	U	0.0384	U	0.0384	U	0	NA	NA
11Cl-PF3OUdS	0.072	U	0.0717	U	0.0717	U	0	NA	NA
PFEESA	0.0182	U	0.0182	U	0.0182	U	0	NA	NA
3:3 FTCA	0.0608	U	0.0606	U	0.0606	U	0	NA	NA
5:3 FTCA	0.368	U	0.366	U	0.366	U	0	NA	NA
7:3 FTCA	0.312	U	0.311	U	0.311	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-21 Soil 2018-116 Native Concentration

Analyte	L33305-1		L33305-2		L33305-3		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.419	U	0.419	U	0.418	U	0	NA	NA
PFPeA	0.0219	U	0.028	J	0.0219	U	1	0.03	NA
PFHxA	0.038	J	0.025	J	0.029	J	3	0.03	21.71
PFHpA	0.04	J	0.0303	U	0.0302	U	1	0.04	NA
PFOA	0.086	J	0.083	J	0.084	J	3	0.08	1.81
PFNA	0.0898	U	0.0898	U	0.0896	U	0	NA	NA
PFDA	0.0324	U	0.0324	U	0.0323	U	0	NA	NA
PFUnA	0.0345	U	0.0345	U	0.0344	U	0	NA	NA
PFDoA	0.0616	U	0.0616	U	0.0615	U	0	NA	NA
PFTrDA	0.0397	U	0.0397	U	0.0396	U	0	NA	NA
PFTeDA	0.0334	U	0.0334	U	0.0334	U	0	NA	NA
PFBS	0.021	J	0.019	J	0.024	J	3	0.02	11.8
PFPeS	0.0157	U	0.0157	U	0.0156	U	0	NA	NA
PFHxS	0.028	J	0.028	J	0.03	J	3	0.03	4.03
PFHpS	0.0595	U	0.0595	U	0.0594	U	0	NA	NA
PFOS	0.261		0.257		0.27		3	0.26	2.53
PFNS	0.048	U	0.048	U	0.048	U	0	NA	NA
PFDS	0.0418	U	0.0418	U	0.0417	U	0	NA	NA
PFDoS	0.0397	U	0.0397	U	0.0396	U	0	NA	NA
4:2 FTS	0.295	U	0.295	U	0.294	U	0	NA	NA
6:2 FTS	0.172	J	0.121	U	0.121	U	1	0.17	NA
8:2 FTS	0.235	U	0.235	U	0.235	U	0	NA	NA
PFOSA	0.071	U	0.071	U	0.0709	U	0	NA	NA
N-MeFOSA	0.0512	U	0.0512	U	0.0511	U	0	NA	NA
N-EtFOSA	0.0397	U	0.0397	U	0.0396	U	0	NA	NA
N-MeFOSAA	0.0313	U	0.0313	U	0.0313	U	0	NA	NA
N-EtFOSAA	0.046	U	0.046	U	0.0459	U	0	NA	NA
N-MeFOSE	0.212	U	0.212	U	0.212	U	0	NA	NA
N-EtFOSE	0.258	U	0.258	U	0.257	U	0	NA	NA

Table C-21 Soil 2018-116 Native Concentration

Analyte	L33305-1		L33305-2		L33305-3		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.142	U	0.142	U	0.142	U	0	NA	NA
ADONA	0.0595	U	0.0595	U	0.0594	U	0	NA	NA
PFMPA	0.0345	U	0.0345	U	0.0344	U	0	NA	NA
PFMBA	0.0303	U	0.0303	U	0.0302	U	0	NA	NA
NFDHA	0.0877	U	0.0877	U	0.0876	U	0	NA	NA
9Cl-PF3ONS	0.0397	U	0.0397	U	0.0396	U	0	NA	NA
11Cl-PF3OUdS	0.0742	U	0.0742	U	0.074	U	0	NA	NA
PFEESA	0.0188	U	0.0188	U	0.0188	U	0	NA	NA
3:3 FTCA	0.0627	U	0.0627	U	0.0625	U	0	NA	NA
5:3 FTCA	0.379	U	0.379	U	0.378	U	0	NA	NA
7:3 FTCA	0.322	U	0.322	U	0.321	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-22 Soil 2019-107 Native Concentration

Analyte	L33306-14		L33306-4		L33306-5		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.41	U	0.41	U	0.413	U	0	NA	NA
PFPeA	0.041	J	0.034	J	0.032	J	3	0.04	13.25
PFHxA	0.042	JQ	0.028	J	0.029	J	3	0.03	23.67
PFHpA	0.03	J	0.0297	U	0.035	J	2	0.03	10.88
PFOA	0.139	J	0.124	J	0.103	J	3	0.12	14.82
PFNA	0.088	U	0.092	J	0.092	J	2	0.09	0
PFDA	0.043	J	0.035	J	0.033	J	3	0.04	14.3
PFUnA	0.036	J	0.049	J	0.041	J	3	0.04	15.61
PFDoA	0.0604	U	0.0604	U	0.0607	U	0	NA	NA
PFTrDA	0.0389	U	0.0389	U	0.0391	U	0	NA	NA
PFTeDA	0.0327	U	0.0328	U	0.0329	U	0	NA	NA
PFBS	0.0143	U	0.0143	U	0.0144	U	0	NA	NA
PFPeS	0.0154	U	0.0154	U	0.0154	U	0	NA	NA
PFHxS	0.0184	U	0.0184	U	0.02	J	1	0.02	NA
PFHpS	0.0583	U	0.0583	U	0.0587	U	0	NA	NA
PFOS	0.254		0.232		0.235		3	0.24	4.96
PFNS	0.0471	U	0.0471	U	0.0474	U	0	NA	NA
PFDS	0.0409	U	0.0409	U	0.0412	U	0	NA	NA
PFDoS	0.0389	U	0.0389	U	0.0391	U	0	NA	NA
4:2 FTS	0.289	U	0.289	U	0.29	U	0	NA	NA
6:2 FTS	0.119	U	0.119	U	0.119	U	0	NA	NA
8:2 FTS	0.23	U	0.23	U	0.232	U	0	NA	NA
PFOSA	0.0696	U	0.0696	U	0.07	U	0	NA	NA
N-MeFOSA	0.0501	U	0.0501	U	0.0505	U	0	NA	NA
N-EtFOSA	0.0389	U	0.0389	U	0.0391	U	0	NA	NA
N-MeFOSAA	0.0307	U	0.0307	U	0.0309	U	0	NA	NA
N-EtFOSAA	0.045	U	0.045	U	0.0453	U	0	NA	NA
N-MeFOSE	0.208	U	0.208	U	0.209	U	0	NA	NA
N-EtFOSE	0.253	U	0.253	U	0.254	U	0	NA	NA

Table C-22 Soil 2019-107 Native Concentration

Analyte	L33306-14		L33306-4		L33306-5		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.139	U	0.139	U	0.14	U	0	NA	NA
ADONA	0.0583	U	0.0583	U	0.0587	U	0	NA	NA
PFMPA	0.0338	U	0.0338	U	0.034	U	0	NA	NA
PFMBA	0.0297	U	0.0297	U	0.0299	U	0	NA	NA
NFDHA	0.086	U	0.086	U	0.0865	U	0	NA	NA
9Cl-PF3ONS	0.0389	U	0.0389	U	0.0391	U	0	NA	NA
11Cl-PF3OUdS	0.0727	U	0.0727	U	0.0731	U	0	NA	NA
PFEESA	0.0184	U	0.0184	U	0.0185	U	0	NA	NA
3:3 FTCA	0.0614	U	0.0614	U	0.0618	U	0	NA	NA
5:3 FTCA	0.371	U	0.371	U	0.374	U	0	NA	NA
7:3 FTCA	0.315	U	0.315	U	0.317	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-23 Soil 2019-110 Native Concentration

Analyte	L33307-17		L33307-18		L33307-12		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.405	U	0.403	U	0.404	U	0	NA	NA
PFPeA	0.03	J	0.041	J	0.03	J	3	0.03	18.86
PFHxA	0.033	J	0.042	J	0.051	J	3	0.04	21.43
PFHpA	0.0293	U	0.04	J	0.043	J	2	0.04	5.11
PFOA	0.056	J	0.071	J	0.068	J	3	0.07	12.21
PFNA	0.204		0.21		0.208		3	0.21	1.47
PFDA	0.044	J	0.044	J	0.044	J	3	0.04	0
PFUnA	0.195		0.219		0.194		3	0.2	6.98
PFDoA	0.0595	U	0.0593	U	0.0594	U	0	NA	NA
PFTrDA	0.061	J	0.067	J	0.059	J	3	0.06	6.68
PFTeDA	0.0323	U	0.0322	U	0.0322	U	0	NA	NA
PFBS	0.0141	U	0.0141	U	0.0141	U	0	NA	NA
PFPeS	0.0151	U	0.0151	U	0.0151	U	0	NA	NA
PFHxS	0.0182	U	0.0181	U	0.0181	U	0	NA	NA
PFHpS	0.0575	U	0.0573	U	0.0574	U	0	NA	NA
PFOS	0.138	J	0.147	J	0.119	J	3	0.13	10.61
PFNS	0.0464	U	0.0462	U	0.0463	U	0	NA	NA
PFDS	0.0403	U	0.0402	U	0.0403	U	0	NA	NA
PFDoS	0.0383	U	0.0382	U	0.0383	U	0	NA	NA
4:2 FTS	0.284	U	0.283	U	0.284	U	0	NA	NA
6:2 FTS	0.117	U	0.117	U	0.117	U	0	NA	NA
8:2 FTS	0.227	U	0.226	U	0.227	U	0	NA	NA
PFOSA	0.0686	U	0.0683	U	0.0685	U	0	NA	NA
N-MeFOSA	0.0494	U	0.0492	U	0.0493	U	0	NA	NA
N-EtFOSA	0.0383	U	0.0382	U	0.0383	U	0	NA	NA
N-MeFOSAA	0.0303	U	0.0301	U	0.0302	U	0	NA	NA
N-EtFOSAA	0.0444	U	0.0442	U	0.0443	U	0	NA	NA
N-MeFOSE	0.205	U	0.204	U	0.204	U	0	NA	NA
N-EtFOSE	0.249	U	0.248	U	0.249	U	0	NA	NA

Table C-23 Soil 2019-110 Native Concentration

Analyte	L33307-17		L33307-18		L33307-12		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.137	U	0.137	U	0.137	U	0	NA	NA
ADONA	0.0575	U	0.0573	U	0.0574	U	0	NA	NA
PFMPA	0.0333	U	0.0332	U	0.0332	U	0	NA	NA
PFMBA	0.0293	U	0.0291	U	0.0292	U	0	NA	NA
NFDHA	0.0847	U	0.0844	U	0.0846	U	0	NA	NA
9Cl-PF3ONS	0.0383	U	0.0382	U	0.0383	U	0	NA	NA
11Cl-PF3OUdS	0.0716	U	0.0713	U	0.0715	U	0	NA	NA
PFEESA	0.0182	U	0.0181	U	0.0181	U	0	NA	NA
3:3 FTCA	0.0605	U	0.0603	U	0.0604	U	0	NA	NA
5:3 FTCA	0.366	U	0.365	U	0.365	U	0	NA	NA
7:3 FTCA	0.311	U	0.309	U	0.31	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-24 Marine Sediment Native Concentration

Analyte	L33324-1		L33324-2		L33324-3		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.4	U	0.403	U	0.403	U	0	NA	NA
PFPeA	0.021	U	0.0211	U	0.023	J	1	0.02	NA
PFHxA	0.02	U	0.0201	U	0.0201	U	0	NA	NA
PFHpA	0.0289	U	0.0291	U	0.0291	U	0	NA	NA
PFOA	0.0369	U	0.0372	U	0.0372	U	0	NA	NA
PFNA	0.0858	U	0.0864	U	0.0864	U	0	NA	NA
PFDA	0.0309	U	0.0311	U	0.0311	U	0	NA	NA
PFUnA	0.0329	U	0.0331	U	0.0331	U	0	NA	NA
PFDoA	0.0589	U	0.0592	U	0.0592	U	0	NA	NA
PFTrDA	0.0379	U	0.0382	U	0.0382	U	0	NA	NA
PFTeDA	0.0319	U	0.0321	U	0.0321	U	0	NA	NA
PFBS	0.014	U	0.0141	U	0.0141	U	0	NA	NA
PFPeS	0.015	U	0.0151	U	0.0151	U	0	NA	NA
PFHxS	0.018	JQ	0.021	JQ	0.0181	U	2	0.02	10.88
PFHpS	0.0569	U	0.0572	U	0.0572	U	0	NA	NA
PFOS	0.0669	U	0.0673	U	0.0673	U	0	NA	NA
PFNS	0.0459	U	0.0462	U	0.0462	U	0	NA	NA
PFDS	0.0399	U	0.0402	U	0.0402	U	0	NA	NA
PFDoS	0.0379	U	0.0382	U	0.0382	U	0	NA	NA
4:2 FTS	0.281	U	0.283	U	0.283	U	0	NA	NA
6:2 FTS	0.58		0.116	U	0.116	U	1	0.58	NA
8:2 FTS	0.225	U	0.226	U	0.226	U	0	NA	NA
PFOSA	0.0679	U	0.0683	U	0.0683	U	0	NA	NA
N-MeFOSA	0.0489	U	0.0492	U	0.0492	U	0	NA	NA
N-EtFOSA	0.0379	U	0.0382	U	0.0382	U	0	NA	NA
N-MeFOSAA	0.0299	U	0.0301	U	0.0301	U	0	NA	NA
N-EtFOSAA	0.0439	U	0.0442	U	0.0442	U	0	NA	NA
N-MeFOSE	0.203	U	0.204	U	0.204	U	0	NA	NA
N-EtFOSE	0.246	U	0.248	U	0.248	U	0	NA	NA

Table C-24 Marine Sediment Native Concentration

Analyte	L33324-1		L33324-2		L33324-3		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.136	U	0.137	U	0.137	U	0	NA	NA
ADONA	0.0569	U	0.0572	U	0.0572	U	0	NA	NA
PFMPA	0.0329	U	0.0331	U	0.0331	U	0	NA	NA
PFMBA	0.0289	U	0.0291	U	0.0291	U	0	NA	NA
NFDHA	0.0838	U	0.0843	U	0.0843	U	0	NA	NA
9Cl-PF3ONS	0.0379	U	0.0382	U	0.0382	U	0	NA	NA
11Cl-PF3OUdS	0.0708	U	0.0713	U	0.0713	U	0	NA	NA
PFEESA	0.018	U	0.0181	U	0.0181	U	0	NA	NA
3:3 FTCA	0.0599	U	0.0602	U	0.0602	U	0	NA	NA
5:3 FTCA	0.362	U	0.364	U	0.364	U	0	NA	NA
7:3 FTCA	0.307	U	0.309	U	0.309	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-25 Sediment #2 Native Concentration

Analyte	L33326-10		L33326-11		L33326-12		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.411	U	0.409	U	0.409	U	0	NA	NA
PFPeA	0.027	J	0.03	J	0.039	J	3	0.03	19.52
PFHxA	0.0205	U	0.031	J	0.023	J	2	0.03	20.95
PFHpA	0.0297	U	0.0296	U	0.0296	U	0	NA	NA
PFOA	0.0379	U	0.0377	U	0.0378	U	0	NA	NA
PFNA	0.0881	U	0.0877	U	0.0878	U	0	NA	NA
PFDA	0.0317	U	0.0316	U	0.0317	U	0	NA	NA
PFUnA	0.0338	U	0.0336	U	0.0337	U	0	NA	NA
PFDoA	0.0604	U	0.0602	U	0.0602	U	0	NA	NA
PFTrDA	0.0389	U	0.0387	U	0.0388	U	0	NA	NA
PFTeDA	0.0328	U	0.0326	U	0.0327	U	0	NA	NA
PFBS	0.0143	U	0.0143	U	0.0143	U	0	NA	NA
PFPeS	0.0154	U	0.0153	U	0.0153	U	0	NA	NA
PFHxS	0.0184	U	0.0184	U	0.0184	U	0	NA	NA
PFHpS	0.0584	U	0.0581	U	0.0582	U	0	NA	NA
PFOS	0.0686	U	0.0683	U	0.0684	U	0	NA	NA
PFNS	0.0471	U	0.0469	U	0.047	U	0	NA	NA
PFDS	0.041	U	0.0408	U	0.0408	U	0	NA	NA
PFDoS	0.0389	U	0.0387	U	0.0388	U	0	NA	NA
4:2 FTS	0.289	U	0.288	U	0.288	U	0	NA	NA
6:2 FTS	0.119	U	0.322	J	0.137	J	2	0.23	57
8:2 FTS	0.23	U	0.229	U	0.23	U	0	NA	NA
PFOSA	0.0696	U	0.0693	U	0.0694	U	0	NA	NA
N-MeFOSA	0.0502	U	0.05	U	0.05	U	0	NA	NA
N-EtFOSA	0.0389	U	0.0387	U	0.0388	U	0	NA	NA
N-MeFOSAA	0.0307	U	0.0306	U	0.0306	U	0	NA	NA
N-EtFOSAA	0.0451	U	0.0449	U	0.0449	U	0	NA	NA
N-MeFOSE	0.208	U	0.207	U	0.207	U	0	NA	NA
N-EtFOSE	0.253	U	0.252	U	0.252	U	0	NA	NA

Table C-25 Sediment #2 Native Concentration

Analyte	L33326-10		L33326-11		L33326-12		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.139	U	0.139	U	0.139	U	0	NA	NA
ADONA	0.0584	U	0.0581	U	0.0582	U	0	NA	NA
PFMPA	0.0338	U	0.0336	U	0.0337	U	0	NA	NA
PFMBA	0.0297	U	0.0296	U	0.0296	U	0	NA	NA
NFDHA	0.086	U	0.0856	U	0.0858	U	0	NA	NA
9Cl-PF3ONS	0.0389	U	0.0387	U	0.0388	U	0	NA	NA
11Cl-PF3OUdS	0.0727	U	0.0724	U	0.0725	U	0	NA	NA
PFEESA	0.0184	U	0.0184	U	0.0184	U	0	NA	NA
3:3 FTCA	0.0614	U	0.0612	U	0.0613	U	0	NA	NA
5:3 FTCA	0.372	U	0.37	U	0.371	U	0	NA	NA
7:3 FTCA	0.315	U	0.314	U	0.314	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-26 Sediment #3 Native Concentration

Analyte	L33325-4		L33325-5		L33325-6		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	0.415	U	0.413	U	0.415	U	0	NA	NA
PFPeA	0.037	J	0.026	J	0.039	J	3	0.03	20.59
PFHxA	0.036	J	0.027	J	0.028	J	3	0.03	16.26
PFHpA	0.038	J	0.035	J	0.03	U	2	0.04	5.81
PFOA	0.115	J	0.111	J	0.094	J	3	0.11	10.45
PFNA	0.0889	U	0.0885	U	0.0889	U	0	NA	NA
PFDA	0.055	J	0.062	J	0.068	J	3	0.06	10.55
PFUnA	0.06	J	0.039	J	0.044	J	3	0.05	23.01
PFDoA	0.061	U	0.0607	U	0.061	U	0	NA	NA
PFTrDA	0.0393	U	0.0391	U	0.0393	U	0	NA	NA
PFTeDA	0.0331	U	0.0329	U	0.0331	U	0	NA	NA
PFBS	0.0145	U	0.0144	U	0.0145	U	0	NA	NA
PFPeS	0.0155	U	0.0154	U	0.0155	U	0	NA	NA
PFHxS	0.026	J	0.022	J	0.023	J	3	0.02	8.8
PFHpS	0.0589	U	0.0587	U	0.0589	U	0	NA	NA
PFOS	0.691		0.677		0.688		3	0.69	1.08
PFNS	0.0476	U	0.0473	U	0.0476	U	0	NA	NA
PFDS	0.042	J	0.0412	U	0.0414	U	1	0.04	NA
PFDoS	0.0393	U	0.0391	U	0.0393	U	0	NA	NA
4:2 FTS	0.292	U	0.29	U	0.292	U	0	NA	NA
6:2 FTS	0.182	J	0.119	U	0.12	U	1	0.18	NA
8:2 FTS	0.233	U	0.232	U	0.233	U	0	NA	NA
PFOSA	0.0703	U	0.07	U	0.0703	U	0	NA	NA
N-MeFOSA	0.0507	U	0.0504	U	0.0507	U	0	NA	NA
N-EtFOSA	0.0393	U	0.0391	U	0.0393	U	0	NA	NA
N-MeFOSAA	0.031	U	0.0309	U	0.031	U	0	NA	NA
N-EtFOSAA	0.112	J	0.141	J	0.14	J	3	0.13	12.57
N-MeFOSE	0.21	U	0.209	U	0.21	U	0	NA	NA
N-EtFOSE	0.255	U	0.254	U	0.255	U	0	NA	NA

Table C-26 Sediment #3 Native Concentration

Analyte	L33325-4		L33325-5		L33325-6		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	0.141	U	0.14	U	0.141	U	0	NA	NA
ADONA	0.0589	U	0.0587	U	0.0589	U	0	NA	NA
PFMPA	0.0341	U	0.034	U	0.0341	U	0	NA	NA
PFMBA	0.03	U	0.0298	U	0.03	U	0	NA	NA
NFDHA	0.0868	U	0.0864	U	0.0868	U	0	NA	NA
9Cl-PF3ONS	0.0393	U	0.0391	U	0.0393	U	0	NA	NA
11Cl-PF3OUdS	0.0734	U	0.0731	U	0.0734	U	0	NA	NA
PFEESA	0.0186	U	0.0185	U	0.0186	U	0	NA	NA
3:3 FTCA	0.062	U	0.0617	U	0.062	U	0	NA	NA
5:3 FTCA	0.375	U	0.374	U	0.375	U	0	NA	NA
7:3 FTCA	0.318	U	0.317	U	0.318	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-27 Biosolid #1 Native Concentration

Analyte	L33327-1		L33327-2		L33327-3		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	3.93	U	6.05	J	4.56	J	2	5.3	19.86
PFPeA	1.08	J	0.934	J	0.865	J	3	0.96	11.44
PFHxA	5.57		6.01		5.76		3	5.78	3.82
PFHpA	0.284	U	0.359	J	0.284	U	1	0.36	NA
PFOA	2.51		2.58		2.47		3	2.52	2.21
PFNA	1.19	J	1.3	J	0.951	J	3	1.15	15.56
PFDA	4.49	Q	5.28	Q	4.74	Q	3	4.84	8.35
PFUnA	1.46	J	1.39	J	1.54	J	3	1.46	5.13
PFDoA	2.92		1.44	J	3.21		3	2.52	37.62
PFTrDA	0.883	J	0.676	J	0.864	JQ	3	0.81	14.17
PFTeDA	1.51	J	1.35	JQ	1.51	JQ	3	1.46	6.34
PFBS	2.01	Q	2.91	Q	2.18	Q	3	2.37	20.2
PFPeS	0.147	U	1.64	Q	0.147	U	1	1.64	NA
PFHxS	1.03	JQ	0.914	JQ	0.885	JQ	3	0.94	8.14
PFHpS	0.559	U	0.553	U	0.559	U	0	NA	NA
PFOS	34.3		33.5		32.5		3	33.43	2.7
PFNS	0.451	U	0.446	U	0.451	U	0	NA	NA
PFDS	1.65		1.87		1.51	J	3	1.68	10.82
PFDoS	0.373	U	0.369	U	0.373	U	0	NA	NA
4:2 FTS	2.77	U	2.74	U	2.77	U	0	NA	NA
6:2 FTS	2.11	J	28.8		2.45	J	3	11.12	137.7
8:2 FTS	2.21	U	2.18	U	2.21	U	0	NA	NA
PFOSA	1.53	J	1.51	J	1.55	J	3	1.53	1.31
N-MeFOSA	0.858	JQ	0.847	JQ	0.554	JQ	3	0.75	22.9
N-EtFOSA	0.513	JQ	0.45	J	0.511	J	3	0.49	7.29
N-MeFOSAA	12		11		11.7		3	11.57	4.44
N-EtFOSAA	4.26		4.7		4.37		3	4.44	5.15
N-MeFOSE	15.2	J	15.7		15.2	J	3	15.37	1.88
N-EtFOSE	3.37	J	3.22	J	3.13	J	3	3.24	3.74

Table C-27 Biosolid #1 Native Concentration

Analyte	L33327-1		L33327-2		L33327-3		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	1.33	U	1.32	U	1.33	U	0	NA	NA
ADONA	0.559	U	0.553	U	0.559	U	0	NA	NA
PFMPA	0.324	U	0.32	U	0.324	U	0	NA	NA
PFMBA	0.284	U	0.281	U	0.284	U	0	NA	NA
NFDHA	0.824	U	0.815	U	0.824	U	0	NA	NA
9Cl-PF3ONS	0.373	U	0.369	U	0.373	U	0	NA	NA
11Cl-PF3OUdS	0.696	U	0.689	U	0.696	U	0	NA	NA
PFEESA	0.177	U	0.175	U	0.177	U	0	NA	NA
3:3 FTCA	0.588	U	0.582	U	0.588	U	0	NA	NA
5:3 FTCA	18.6	J	17.5	J	18.1	J	3	18.07	3.05
7:3 FTCA	9.24	J	9.53	J	8.59	J	3	9.12	5.28

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-28 Biosolid #2 Native Concentration

Analyte	L33328-4		L33328-5		L33328-6		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	4	U	3.95	U	4.11	U	0	NA	NA
PFPeA	0.32	J	0.462	J	0.458	J	3	0.41	19.56
PFHxA	1.1	J	0.937	J	1.22	J	3	1.09	13.08
PFHpA	0.289	U	0.286	U	0.297	U	0	NA	NA
PFOA	0.531	J	0.434	J	0.607	J	3	0.52	16.55
PFNA	0.858	U	0.847	U	0.882	U	0	NA	NA
PFDA	0.701	JQ	0.764	JQ	0.712	JQ	3	0.73	4.64
PFUnA	0.329	U	0.325	U	0.338	U	0	NA	NA
PFDoA	0.589	U	0.581	U	0.605	U	0	NA	NA
PFTrDA	0.499	JQ	0.58	JQ	0.584	JQ	3	0.55	8.65
PFTeDA	0.335	JQ	0.37	JQ	0.328	U	2	0.35	7.02
PFBS	0.196	J	0.427	JQ	0.518	JQ	3	0.38	43.64
PFPeS	0.15	U	0.15	JQ	0.154	U	1	0.15	NA
PFHxS	0.196	JQ	0.177	U	0.185	U	1	0.2	NA
PFHpS	0.569	U	0.561	U	0.585	U	0	NA	NA
PFOS	1.38	J	1.09	J	1.27	J	3	1.25	11.74
PFNS	0.459	U	0.453	U	0.472	U	0	NA	NA
PFDS	0.399	U	0.394	U	0.41	U	0	NA	NA
PFDoS	0.379	U	0.374	U	0.39	U	0	NA	NA
4:2 FTS	2.81	U	2.78	U	2.89	U	0	NA	NA
6:2 FTS	1.16	U	23		1.19	U	1	23	NA
8:2 FTS	2.25	U	2.22	U	2.31	U	0	NA	NA
PFOSA	0.679	U	0.67	U	0.697	U	0	NA	NA
N-MeFOSA	0.489	U	0.483	U	0.503	U	0	NA	NA
N-EtFOSA	0.379	U	0.374	U	0.39	U	0	NA	NA
N-MeFOSAA	1.21	J	1.18	J	1.63	JQ	3	1.34	18.78
N-EtFOSAA	0.585	J	0.436	J	0.578	JQ	3	0.53	15.77
N-MeFOSE	2.03	U	2	U	2.08	U	0	NA	NA
N-EtFOSE	2.47	U	2.43	U	2.53	U	0	NA	NA

Table C-28 Biosolid #2 Native Concentration

Analyte	L33328-4		L33328-5		L33328-6		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	1.36	U	1.34	U	1.39	U	0	NA	NA
ADONA	0.569	U	0.561	U	0.585	U	0	NA	NA
PFMPA	0.329	U	0.325	U	0.338	U	0	NA	NA
PFMBA	0.289	U	0.286	U	0.297	U	0	NA	NA
NFDHA	0.838	U	0.827	U	0.862	U	0	NA	NA
9Cl-PF3ONS	0.379	U	0.374	U	0.39	U	0	NA	NA
11Cl-PF3OUdS	0.709	U	0.699	U	0.728	U	0	NA	NA
PFEESA	0.18	U	0.177	U	0.185	U	0	NA	NA
3:3 FTCA	0.599	U	0.591	U	0.615	U	0	NA	NA
5:3 FTCA	10.3	J	9.17	J	10.4	J	3	9.96	6.86
7:3 FTCA	3.07	U	3.03	U	3.16	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-29. Biosolid #3 Native Concentration

Analyte	L33329-10		L33329-11		L33329-12		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
PFBA	17.7		14.4		15.6		3	15.9	10.51
PFPeA	3.52		3.33	J	3.12	J	3	3.32	6.02
PFHxA	4.85		5.05		5.43		3	5.11	5.77
PFHpA	0.315	U	0.321	U	0.308	J	1	0.31	NA
PFOA	1.52	J	1.5	J	1.68		3	1.57	6.3
PFNA	3.47		3.69		3.73		3	3.63	3.86
PFDA	1.78		2.09		2.21		3	2.03	10.95
PFUnA	2.14		2.17		2.09		3	2.13	1.89
PFDoA	2.54		2.45		3.49		3	2.83	20.39
PFTrDA	1.48	J	1.4	J	1.61	J	3	1.5	7.08
PFTeDA	1.96	Q	1.75	J	1.31	J	3	1.67	19.82
PFBS	4.03	Q	2.02	Q	2.05	Q	3	2.7	42.66
PFPeS	0.163	U	0.166	U	0.153	U	0	NA	NA
PFHxS	39.6	Q	38.1	Q	43.8	Q	3	40.5	7.3
PFHpS	0.619	U	0.632	U	0.583	U	0	NA	NA
PFOS	13.3		13.3		13.9		3	13.5	2.57
PFNS	3.08	Q	0.51	U	0.471	U	1	3.08	NA
PFDS	4.52	Q	5.06	Q	5.12	Q	3	4.9	6.74
PFDoS	0.413	U	0.421	U	0.389	U	0	NA	NA
4:2 FTS	3.06	U	3.13	U	2.88	U	0	NA	NA
6:2 FTS	1.7	JQ	2.79	J	1.25	JQ	3	1.91	41.39
8:2 FTS	2.44	U	2.49	U	2.3	U	0	NA	NA
PFOSA	1.13	J	1.25	J	1.27	J	3	1.22	6.22
N-MeFOSA	0.532	U	0.543	U	0.501	U	0	NA	NA
N-EtFOSA	0.413	U	0.421	U	0.389	U	0	NA	NA
N-MeFOSAA	4.6		4.74		5.21		3	4.85	6.59
N-EtFOSAA	8.29		9.46		10.5		3	9.42	11.74
N-MeFOSE	11.5	J	11.7	J	12.1	J	3	11.77	2.6
N-EtFOSE	4.09	J	4.61	J	4.78	J	3	4.49	8

Table C-29. Biosolid #3 Native Concentration

Analyte	L33329-10		L33329-11		L33329-12		Number of Detections	Mean (µg/kg)	RSD [1] (%)
	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier	Concentration (ug/kg)	Qualifier			
HFPO-DA	1.48	U	1.51	U	1.39	U	0	NA	NA
ADONA	0.619	U	0.632	U	0.583	U	0	NA	NA
PFMPA	0.358	U	0.366	U	0.338	U	0	NA	NA
PFMBA	0.315	U	0.321	U	0.297	U	0	NA	NA
NFDHA	0.912	U	0.931	U	0.859	U	0	NA	NA
9Cl-PF3ONS	0.413	U	0.421	U	0.389	U	0	NA	NA
11Cl-PF3OUdS	0.771	U	0.787	U	0.726	U	0	NA	NA
PFEESA	0.195	U	0.199	U	0.184	U	0	NA	NA
3:3 FTCA	0.651	U	0.665	U	0.614	U	0	NA	NA
5:3 FTCA	51.2		51.8		57.5		3	53.5	6.5
7:3 FTCA	14.8	J	17.1	J	16.1	J	3	16	7.21

py_Log Date 6/28/2021 16:01

Notes:

1. RSD = Relative Standard Deviation

Table C-30 Fish Tissue #1 Native Concentration

	L33358-1		L33358-3		L33358-4		Number of Detections	Mean (µg/kg ww [1])	RSD [2] (%)
	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier			
PFBA	0.593	U	0.593	U	0.59	U	0	NA	NA
PFPeA	0.825		0.804		0.776	J	3	0.8	3.07
PFHxA	0.163	J	0.096	U	0.0955	U	1	0.16	NA
PFHpA	0.088	U	0.088	U	0.0876	U	0	NA	NA
PFOA	0.086	U	0.086	U	0.099	J	1	0.1	NA
PFNA	0.413		0.422		0.41		3	0.41	1.5
PFDA	0.316	J	0.289	J	0.28	J	3	0.29	6.35
PFUnA	0.535		0.477		0.475		3	0.5	6.88
PFDoA	0.178	J	0.164	J	0.138	J	3	0.16	12.69
PFTrDA	0.41		0.422		0.365	J	3	0.4	7.53
PFTeDA	0.185	U	0.185	U	0.184	U	0	NA	NA
PFBS	0.07	U	0.07	U	0.0697	U	0	NA	NA
PFPeS	0.032	U	0.032	U	0.0318	U	0	NA	NA
PFHxS	0.118	JQ	0.093	J	0.108	JQ	3	0.11	11.83
PFHpS	0.043	U	0.043	U	0.0428	U	0	NA	NA
PFOS	1.32		1.39		1.32		3	1.34	3.01
PFNS	0.114	U	0.114	U	0.113	U	0	NA	NA
PFDS	0.101	U	0.101	U	0.1	U	0	NA	NA
PFDoS	0.177	U	0.177	U	0.176	U	0	NA	NA
4:2 FTS	0.74	U	0.74	U	0.736	U	0	NA	NA
6:2 FTS	3.02		1.15	U	1.14	U	1	3.02	NA
8:2 FTS	0.373	U	0.373	U	0.371	U	0	NA	NA
PFOSA	0.479		0.474		0.45		3	0.47	3.31
N-MeFOSA	0.161	U	0.161	U	0.16	U	0	NA	NA
N-EtFOSA	0.169	U	0.169	U	0.168	U	0	NA	NA
N-MeFOSAA	0.093	U	0.093	U	0.0925	U	0	NA	NA
N-EtFOSAA	0.138	U	0.138	U	0.137	U	0	NA	NA
N-MeFOSE	9.98	U	9.98	U	9.93	U	0	NA	NA

Table C-30 Fish Tissue #1 Native Concentration

	L33358-1		L33358-3		L33358-4		Number of Detections	Mean (µg/kg ww [1])	RSD [2] (%)
	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier			
N-EtFOSE	3.03	Q	4.92	Q	5.13	Q	3	4.36	26.53
HFPO-DA	0.161	U	0.161	U	0.16	U	0	NA	NA
ADONA	0.082	U	0.082	U	0.0816	U	0	NA	NA
PFMPA	0.07	U	0.07	U	0.0697	U	0	NA	NA
PFMBA	0.069	U	0.069	U	0.0687	U	0	NA	NA
NFDHA	0.294	U	0.294	U	0.293	U	0	NA	NA
9Cl-PF3ONS	0.152	U	0.152	U	0.151	U	0	NA	NA
11Cl-PF3OUdS	0.312	U	0.312	U	0.31	U	0	NA	NA
PFEESA	0.045	U	0.045	U	0.0448	U	0	NA	NA
3:3 FTCA	0.247	U	0.247	U	0.246	U	0	NA	NA
5:3 FTCA	1.54	U	1.54	U	1.53	U	0	NA	NA
7:3 FTCA	1.49	J	1.66	J	1.4	J	3	1.52	8.71

py_Log Date 6/28/2021 16:01

Notes:

1. µg/kg ww = micrograms/kilogram wet weight
2. RSD = Relative Standard Deviation

Table C-31 Fish Tissue #2 Native Concentration

Analyte	L33357-2		L33357-5		L33357-6		Number of Detections	Mean (µg/kg ww [1])	RSD [2] (%)
	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier			
PFBA	0.596	U	0.59	U	0.593	U	0	NA	NA
PFPeA	0.0834	U	0.0826	U	0.083	U	0	NA	NA
PFHxA	0.0965	U	0.0955	U	0.096	U	0	NA	NA
PFHpA	0.0884	U	0.0876	U	0.088	U	0	NA	NA
PFOA	0.0864	U	0.0856	U	0.086	U	0	NA	NA
PFNA	0.161	U	0.159	U	0.16	U	0	NA	NA
PFDA	1.02		0.76		0.71		3	0.83	20.05
PFUnA	1.62		1.43		1.46		3	1.5	6.79
PFDoA	0.563		0.633		0.701		3	0.63	10.91
PFTrDA	1.02		1.42		1.5		3	1.31	19.58
PFTeDA	0.344	J	0.468		0.446		3	0.42	15.78
PFBS	0.0704	U	0.0697	U	0.07	U	0	NA	NA
PFPeS	0.0322	U	0.0318	U	0.032	U	0	NA	NA
PFHxS	0.0834	U	0.0826	U	0.083	U	0	NA	NA
PFHpS	0.0432	U	0.0428	U	0.043	U	0	NA	NA
PFOS	11.7		9.36		9.9		3	10.32	11.87
PFNS	0.115	U	0.113	U	0.114	U	0	NA	NA
PFDS	0.653		0.252	J	0.333	J	3	0.41	51.38
PFDoS	0.178	U	0.176	U	0.177	U	0	NA	NA
4:2 FTS	0.744	U	0.736	U	0.74	U	0	NA	NA
6:2 FTS	1.15	U	1.14	U	1.15	U	0	NA	NA
8:2 FTS	0.375	U	0.371	U	0.373	U	0	NA	NA
PFOSA	0.0945	U	0.0935	U	0.094	U	0	NA	NA
N-MeFOSA	0.162	U	0.16	U	0.161	U	0	NA	NA
N-EtFOSA	0.17	U	0.168	U	0.169	U	0	NA	NA
N-MeFOSAA	0.0935	U	0.0925	U	0.093	U	0	NA	NA
N-EtFOSAA	0.139	U	0.137	U	0.138	U	0	NA	NA
N-MeFOSE	10	U	9.93	U	9.98	U	0	NA	NA
N-EtFOSE	1.89	J	1.49	U	1.5	U	1	1.89	NA
HFPO-DA	0.162	U	0.16	U	0.161	U	0	NA	NA

Table C-31 Fish Tissue #2 Native Concentration

Analyte	L33357-2		L33357-5		L33357-6		Number of Detections	Mean (µg/kg ww [1])	RSD [2] (%)
	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier			
ADONA	0.0824	U	0.0816	U	0.082	U	0	NA	NA
PFMPA	0.0704	U	0.0697	U	0.07	U	0	NA	NA
PFMBA	0.0694	U	0.0687	U	0.069	U	0	NA	NA
NFDHA	0.295	U	0.293	U	0.294	U	0	NA	NA
9Cl-PF3ONS	0.153	U	0.151	U	0.152	U	0	NA	NA
11Cl-PF3OUdS	0.314	U	0.31	U	0.312	U	0	NA	NA
PFEESA	0.0452	U	0.0448	U	0.045	U	0	NA	NA
3:3 FTCA	0.248	U	0.246	U	0.247	U	0	NA	NA
5:3 FTCA	1.54	U	1.53	U	1.54	U	0	NA	NA
7:3 FTCA	0.849	U	0.841	U	0.845	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. µg/kg ww = micrograms/kilogram wet weight
2. RSD = Relative Standard Deviation

Table C-32 Clam Tissue Native Concentration

Analyte	L33356-7		L33356-8		L33356-9		Number of Detections	Mean (µg/kg ww [1])	RSD [2] (%)
	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier			
PFBA	0.596	U	0.596	U	0.593	U	0	NA	NA
PFPeA	0.0834	U	0.0834	U	0.083	U	0	NA	NA
PFHxA	0.0965	U	0.0965	U	0.096	U	0	NA	NA
PFHpA	0.0884	U	0.0884	U	0.088	U	0	NA	NA
PFOA	0.0864	U	0.0864	U	0.086	U	0	NA	NA
PFNA	0.161	U	0.161	U	0.16	U	0	NA	NA
PFDA	0.125	U	0.125	U	0.124	U	0	NA	NA
PFUnA	0.153	U	0.153	U	0.152	U	0	NA	NA
PFDoA	0.131	U	0.131	U	0.13	U	0	NA	NA
PFTrDA	0.0864	U	0.0864	U	0.086	U	0	NA	NA
PFTeDA	0.186	U	0.186	U	0.185	U	0	NA	NA
PFBS	0.0704	U	0.0704	U	0.07	U	0	NA	NA
PFPeS	0.0322	U	0.0322	U	0.032	U	0	NA	NA
PFHxS	0.0834	U	0.0834	U	0.083	U	0	NA	NA
PFHpS	0.0432	U	0.0432	U	0.043	U	0	NA	NA
PFOS	0.295	U	0.295	U	0.294	U	0	NA	NA
PFNS	0.115	U	0.115	U	0.114	U	0	NA	NA
PFDS	0.102	U	0.102	U	0.101	U	0	NA	NA
PFDoS	0.178	U	0.178	U	0.177	U	0	NA	NA
4:2 FTS	0.744	U	0.744	U	0.74	U	0	NA	NA
6:2 FTS	1.15	U	1.15	U	12.5		1	12.5	NA
8:2 FTS	0.375	U	0.375	U	0.373	U	0	NA	NA
PFOSA	0.0945	U	0.0945	U	0.094	U	0	NA	NA
N-MeFOSA	0.162	U	0.162	U	0.161	U	0	NA	NA
N-EtFOSA	0.17	U	0.17	U	0.169	U	0	NA	NA
N-MeFOSAA	0.0935	U	0.0935	U	0.093	U	0	NA	NA
N-EtFOSAA	0.139	U	0.139	U	0.138	U	0	NA	NA
N-MeFOSE	10	U	10	U	9.98	U	0	NA	NA
N-EtFOSE	1.51	U	1.51	U	1.5	U	0	NA	NA
HFPO-DA	0.162	U	0.162	U	0.161	U	0	NA	NA

Table C-32 Clam Tissue Native Concentration

Analyte	L33356-7		L33356-8		L33356-9		Number of Detections	Mean (µg/kg ww [1])	RSD [2] (%)
	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier	Concentration (µg/kg ww [1])	Qualifier			
ADONA	0.0824	U	0.0824	U	0.082	U	0	NA	NA
PFMPA	0.0704	U	0.0704	U	0.07	U	0	NA	NA
PFMBA	0.0694	U	0.0694	U	0.069	U	0	NA	NA
NFDHA	0.295	U	0.295	U	0.294	U	0	NA	NA
9Cl-PF3ONS	0.153	U	0.153	U	0.152	U	0	NA	NA
11Cl-PF3OUdS	0.314	U	0.314	U	0.312	U	0	NA	NA
PFEESA	0.0452	U	0.0452	U	0.045	U	0	NA	NA
3:3 FTCA	0.248	U	0.248	U	0.247	U	0	NA	NA
5:3 FTCA	1.54	U	1.54	U	1.54	U	0	NA	NA
7:3 FTCA	0.849	U	0.849	U	0.845	U	0	NA	NA

py_Log Date 6/28/2021 16:01

Notes:

1. µg/kg ww = micrograms/kilogram wet weight
2. RSD = Relative Standard Deviation

Appendix D

Individual Sample Matrix Recovery

APPENDIX D. SAMPLE MATRIX RECOVERY TABLES

This appendix reports the mean spike concentration and mean percent recovery for the 40 PFAS analytes, from three subsamples, for each environmental sample. Spike concentration indicates the known amount of analyte added to the native sample, while percent recovery indicates the percent of the analyte detected compared with the known amount. The native concentration was subtracted from the spike concentration before calculating the percent recovery, for each subsample. Only positive percent recovery values are reported. Spike concentrations that were flagged by the lab as either U (where it was not detected and less than the limit of detection) or B (where the results were associated with a contaminated blank) were not used to calculate percent recoveries.

Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021). Briefly, the tables include the following information

- Analyte—40 PFAS analytes listed in Table 1 of the Single Lab Validation Study Report
- Native Mean Concentration—Mean native concentration calculated and reported in Appendix C. Native concentration for each of the three subsamples of the analyte from the EDD in µg/kg for solid matrices and tissues, ng/L for aqueous matrices.
- Number of Detections—Number of subsamples for the analyte that do not have a U or B flag for native concentration. Could be 0, 1, 2 or 3.
- Mean PFAS Spike Concentration - Mean spike concentration of the EDD entries that do not have a U or B flag. If all three subsamples have B-flags.
- % Recovery - Mean of the three subsamples' percent recoveries for spike concentrations that do not have a U or B flag. The native concentration is subtracted before calculating each of the three subsamples' percent recoveries. If the native concentration is NA, then a value of 0 was used instead. If mean native concentration is greater than the spike concentration, then NA was used. If three subsample values have a B-flag, then NA was used. If the mean percent recovery is less than 0, then NA was used.
- Mean—Mean native concentration of the subsamples that do not have a U or B flag. If all three subsamples have U or B, then the value is NA.
- RSD— Percent RSD of the mean native concentration. If the number of samples is less than two then NA is reported.

Table D-1 GW CO Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			High 2 Spike Recovery				All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	GW 4 Native Mean Concentration [1]	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	18.2	3	15	NA	3	29.9	109.89	3	44.7	103.36	4.86	1	199	99.74	3	104.33	4.93
PFPeA	22.2	3	7.5	NA	3	14.9	NA	3	22.4	117.45	5.81	1	99.7	106.14	2	111.8	7.15
PFHxA	19.6	3	3.75	NA	3	7.47	NA	3	11.2	NA	4.96	1	49.9	97.8	1	97.8	NA
PFHpA	7.47	3	3.75	NA	3	7.47	102.93	3	11.2	107.45	1.98	1	49.9	101.12	3	103.84	3.14
PFOA	23	3	3.75	NA	3	7.47	NA	3	11.2	NA	5.74	1	49.9	97.05	1	97.05	NA
PFNA	2.08	3	3.75	90.56	3	7.47	102.16	3	11.2	106.72	0.446	1	49.9	98.78	4	99.55	6.85
PFDA	0.556	3	3.75	96.23	3	7.47	110.57	3	11.2	109.63	NA	0	49.9	104.62	4	105.26	6.24
PFUnA	NA	0	3.75	91.09	0	7.47	110.87	0	11.2	108.32	NA	0	49.9	99.95	4	102.56	8.74
PFDoA	NA	0	3.75	82.26	0	7.47	107.12	0	11.2	107.21	NA	0	49.9	101.31	4	99.47	11.87
PFTrDA	NA	0	3.75	89.23	0	7.47	112.42	0	11.2	108.95	NA	0	49.9	96.85	4	101.86	10.55
PFTeDA	NA	0	3.75	91.86	0	7.47	110.83	0	11.2	108.06	NA	0	49.9	99.35	4	102.53	8.42
PFBS	20.6	3	3.75	NA	3	7.47	NA	3	11.2	NA	5.05	1	49.9	103.84	1	103.84	NA
PFPeS	8.06	3	3.76	NA	3	7.49	NA	3	11.2	111.29	1.99	1	50	94.23	2	102.76	11.74
PFHxS	48.1	3	3.75	NA	3	7.47	NA	3	11.2	NA	11.1	1	49.9	107.14	1	107.14	NA
PFHpS	1.3	3	3.76	91.96	3	7.49	108.63	3	11.2	105.61	0.261	1	50	93.22	4	99.86	8.51
PFOS	124	3	3.75	NA	3	7.47	NA	3	11.2	NA	20.1	1	49.9	108.59	1	108.59	NA
PFNS	NA	0	3.76	83.08	0	7.48	97.51	0	11.2	108.03	NA	0	50	97.6	4	96.55	10.62
PFDS	NA	0	3.75	78.04	0	7.47	91.76	0	11.2	93.74	NA	0	49.9	92.26	4	88.95	8.23
PFDoS	NA	0	3.75	74.59	0	7.48	94.04	0	11.2	91.69	NA	0	49.9	88.49	4	87.2	9.99
4:2 FTS	NA	0	15	87.74	0	29.9	105.43	0	44.7	109.69	NA	0	199	98.19	4	100.26	9.58
6:2 FTS	NA	0	13.5	108.06	0	26.9	120.16	0	40.3	115.16	NA	0	180	102.43	4	111.46	7
8:2 FTS	NA	0	15	108.2	0	29.9	125.28	0	44.7	124.31	NA	0	199	114.93	4	118.18	6.88
PFOSA	5.6	3	3.75	NA	3	7.47	130.25	3	11.2	147.49	0.968	1	49.9	102.61	3	126.78	17.86
N-MeFOSA	NA	0	4.31	73.25	0	8.59	110.98	0	12.9	102.31	NA	0	57.4	98.03	4	96.14	16.83
N-EtFOSA	NA	0	9.38	73.76	0	18.7	105.67	0	28	100.34	NA	0	125	102.21	4	95.49	15.35
N-MeFOSAA	NA	0	3.75	90.25	0	7.47	118.67	0	11.2	144.62	NA	0	49.9	97.82	4	112.84	21.59
N-EtFOSAA	NA	0	3.75	87.55	0	7.47	114.16	0	11.2	127.37	NA	0	49.9	98.29	4	106.84	16.4
N-MeFOSE	NA	0	37.5	85.57	0	74.7	109.08	0	112	106.26	NA	0	499	105.44	4	101.59	10.62
N-EtFOSE	NA	0	28.1	87.64	0	56	115.45	0	83.9	109.89	NA	0	374	108.82	4	105.45	11.59
HFPO-DA	NA	0	14.2	92.23	0	28.4	105.74	0	42.5	106.87	NA	0	190	100.14	4	101.25	6.61
ADONA	NA	0	15	84.22	0	29.9	99.66	0	44.9	106.91	NA	0	200	84.12	4	93.73	12.19
PFMPA	NA	0	7.5	95.42	0	14.9	112.49	0	22.4	100.15	NA	0	99.7	100.02	4	102.02	7.17
PFMBA	NA	0	3.75	84.43	0	7.47	110.31	0	11.2	100.31	NA	0	49.9	99.83	4	98.72	10.82
NFDHA	NA	0	7.5	83.42	0	14.9	105.37	0	22.4	92.83	NA	0	99.7	82.88	4	91.13	11.56
9Cl-PF3ONS	NA	0	15	82.45	0	29.9	102.55	0	44.8	104.81	NA	0	200	89.07	4	94.72	11.33
11Cl-PF3OUdS	NA	0	15	72.26	0	29.9	94.43	0	44.8	99.02	NA	0	200	88.26	4	88.49	13.2
PFEESA	NA	0	3.75	91.34	0	7.47	106.35	0	11.2	104.17	NA	0	49.9	97.44	4	99.82	6.82
3:3 FTCA	NA	0	15	91.75	0	29.9	109.8	0	44.7	102.01	NA	0	199	96.8	4	100.09	7.7
5:3 FTCA	NA	0	93.8	85.84	0	187	97.1	0	280	96.18	NA	0	1250	85.42	4	91.14	6.99
7:3 FTCA	NA	0	93.8	83.5	0	187	96.76	0	280	92.61	NA	0	1250	93.37	4	91.56	6.19

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-2 GW WL378 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			High 2 Spike Recovery				All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	GW 5 Native Mean Concentration [1]	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	10.5	3	15.6	99.34	3	30.7	116.73	3	46.5	113.98	2.85	1	400	99.21	4	107.31	8.71
PFPeA	13.6	3	7.78	NA	3	15.4	118.19	3	23.3	112.44	3.64	1	200	104.01	3	111.55	6.39
PFHxA	49.8	3	3.89	NA	3	7.67	NA	3	11.6	NA	12.2	1	99.9	99.9	1	99.9	NA
PFHpA	7.17	3	3.89	NA	3	7.67	115.22	3	11.6	108.02	1.91	1	99.9	97.66	3	106.97	8.26
PFOA	41.3	3	3.89	NA	3	7.67	NA	3	11.6	NA	8.97	1	99.9	96.6	1	96.6	NA
PFNA	0.635	3	3.89	98.13	3	7.67	112.85	3	11.6	112.89	NA	0	99.9	98.75	4	105.66	7.89
PFDA	0.435	2	3.89	101.11	2	7.67	124.71	2	11.6	120.61	NA	0	99.9	104.11	4	112.64	10.44
PFUnA	NA	0	3.89	107.84	0	7.67	119.07	0	11.6	117.2	NA	0	99.9	99.78	4	110.97	8.05
PFDoA	NA	0	3.89	106.51	0	7.67	117.09	0	11.6	120.89	NA	0	99.9	98.45	4	110.73	9.21
PFTrDA	NA	0	3.89	106.57	0	7.67	124.49	0	11.6	126.37	NA	0	99.9	91.45	4	112.22	14.68
PFTeDA	NA	0	3.89	105.72	0	7.67	121.26	0	11.6	118.62	NA	0	99.9	97.47	4	110.77	10.08
PFBS	20.1	3	3.89	NA	3	7.67	NA	3	11.6	NA	5.09	1	99.9	103.68	1	103.68	NA
PFPeS	18.4	3	3.9	NA	3	7.69	NA	3	11.7	NA	4.44	1	100	93.36	1	93.36	NA
PFHxS	236	3	3.89	NA	3	7.67	NA	3	11.6	NA	54.3	1	99.9	104.81	1	104.81	NA
PFHpS	5.44	3	3.9	NA	3	7.68	131.5	3	11.7	123.37	1.03	1	100	89.6	3	114.82	19.35
PFOS	346	3	3.89	NA	3	7.67	NA	3	11.6	NA	57.2	1	99.9	110.92	1	110.92	NA
PFNS	NA	0	3.9	119.48	0	7.68	127.21	0	11.7	122.82	NA	0	100	96.7	4	116.55	11.68
PFDS	NA	0	3.89	96.77	0	7.67	111.79	0	11.6	107.73	NA	0	99.9	89.69	4	101.49	9.96
PFDoS	NA	0	3.9	92.81	0	7.68	104.99	0	11.6	105.99	NA	0	100	84.22	4	97	10.74
4:2 FTS	NA	0	15.6	101.68	0	30.7	119	0	46.5	119.66	NA	0	400	99.26	4	109.9	9.95
6:2 FTS	NA	0	14.03*	NA	0	27.6	130.47	0	41.9	119.22	NA	0	360	97.04	3	115.58	14.72
8:2 FTS	NA	0	15.6	109.61	0	30.7	125.35	0	46.5	124.26	NA	0	400	104.19	4	115.85	9.13
PFOSA	4.72	3	3.89	NA	3	7.67	110.16	3	11.6	115.32	0.63	1	99.9	101.87	3	109.12	6.21
N-MeFOSA	NA	0	4.47	95.22	0	8.81	113.75	0	13.4	114.44	NA	0	115	98.09	4	105.37	9.62
N-EtFOSA	NA	0	9.72	93.22	0	19.2	113.73	0	29.1	110.08	NA	0	250	102.57	4	104.9	8.64
N-MeFOSAA	NA	0	3.89	106.85	0	7.67	151.29	0	11.6	140.32	NA	0	99.9	93.6	4	123.02	22.14
N-EtFOSAA	NA	0	3.89	101.26	0	7.67	137.41	0	11.6	126.91	NA	0	99.9	95.64	4	115.31	17.4
N-MeFOSE	NA	0	38.9	100.42	0	76.7	117.66	0	116	117.21	NA	0	999	104.99	4	110.07	7.91
N-EtFOSE	NA	0	29.1	101.01	0	57.5	122.13	0	87.2	121.9	NA	0	749	106.9	4	112.98	9.47
HFPO-DA	NA	0	14.8	105.66	0	29.1	125.39	0	44.2	124.5	NA	0	380	103.76	4	114.83	10.2
ADONA	NA	0	15.6	92.72	0	30.8	115.56	0	46.7	136.2	NA	0	401	83.75	4	107.06	22.04
PFMPA	NA	0	7.78	106.44	0	15.4	120.45	0	23.3	115.47	NA	0	200	98.08	4	110.11	8.99
PFMBA	NA	0	3.89	104.09	0	7.67	116.45	0	11.6	114.32	NA	0	99.9	100.12	4	108.75	7.25
NFDHA	NA	0	7.78	100.19	0	15.4	109.38	0	23.3	112.67	NA	0	200	138.81	4	115.26	14.37
9Cl-PF3ONS	NA	0	15.6	95.31	0	30.7	115.94	0	46.6	142.53	NA	0	400	88.8	4	110.64	21.87
11Cl-PF3OUdS	NA	0	15.6	91.46	0	30.7	106.36	0	46.6	125.93	NA	0	400	85.54	4	102.32	17.6
PFEESA	NA	0	3.89	97.34	0	7.67	116.87	0	11.6	118.04	NA	0	99.9	101.26	4	108.38	9.79
3:3 FTCA	NA	0	15.6	104.49	0	30.7	119.89	0	46.5	111.54	NA	0	400	94.98	4	107.73	9.81
5:3 FTCA	NA	0	97.2	101.94	0	192	117.88	0	291	114.78	NA	0	2500	91.13	4	106.43	11.57
7:3 FTCA	NA	0	97.2	106.25	0	192	120.89	0	291	116.28	NA	0	2500	99.76	4	110.8	8.63

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-3 GW Wurtsmith AFB Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			High 2 Spike Recovery				All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	GW 6 Native Mean Concentration [1]	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	3.84	3	16	101.65	3	30.9	110.34	3	47.5	107.28	1.11	1	399	100.05	4	104.83	4.59
PFPeA	15.1	3	7.99	NA	3	15.5	112.89	3	23.7	114.58	3.61	1	199	104.71	3	110.73	4.77
PFHxA	38.2	3	4	NA	3	7.73	NA	3	11.9	NA	9.25	1	99.7	98.35	1	98.35	NA
PFHpA	7.7	3	4	NA	3	7.73	105.64	3	11.9	107.29	1.86	1	99.7	98.2	3	103.71	4.67
PFOA	64.3	3	4	NA	3	7.73	NA	3	11.9	NA	15.2	1	99.7	96.05	1	96.05	NA
PFNA	1.25	3	4	105.39	3	7.73	108.8	3	11.9	105.76	0.337	1	99.7	99.36	4	104.83	3.77
PFDA	NA	0	4	112.83	0	7.73	115.97	0	11.9	114.57	NA	0	99.7	104.61	4	112	4.54
PFUnA	NA	0	4	100.62	0	7.73	108.41	0	11.9	110.06	NA	0	99.7	100	4	104.77	4.96
PFDoA	NA	0	4	96.95	0	7.73	116.74	0	11.9	102.76	NA	0	99.7	99.28	4	103.93	8.53
PFTrDA	NA	0	4	105.1	0	7.73	114.96	0	11.9	108.66	NA	0	99.7	92.41	4	105.28	9.03
PFTeDA	NA	0	4	102.1	0	7.73	114.36	0	11.9	111.8	NA	0	99.7	98	4	106.56	7.3
PFBS	1.9	3	4	101.13	3	7.73	113.28	3	11.9	111.97	0.41	1	99.7	104.53	4	107.73	5.43
PFPeS	3.39	3	4.01	94.14	3	7.75	121.36	3	11.9	115.26	0.694	1	99.9	91.43	4	105.55	14.2
PFHxS	149	3	4	NA	3	7.73	NA	3	11.9	NA	32.4	1	99.7	108.21	1	108.21	NA
PFHpS	4.76	3	4.01	NA	3	7.75	115.3	3	11.9	122.52	1.1	1	99.9	93.99	3	110.61	13.41
PFOS	381	3	4	NA	3	7.73	NA	3	11.9	NA	100	1	99.7	NA	0	NA	NA
PFNS	NA	0	4	123.2	0	7.74	126.75	0	11.9	130.47	NA	0	99.8	104.2	4	121.16	9.65
PFDS	NA	0	4	106.13	0	7.73	115.08	0	11.9	107.83	NA	0	99.7	98.13	4	106.79	6.51
PFDoS	NA	0	4	98.83	0	7.74	98.39	0	11.9	101.86	NA	0	99.8	87.96	4	96.76	6.27
4:2 FTS	NA	0	16	106.96	0	30.9	112.94	0	47.5	109.45	NA	0	399	98.51	4	106.96	5.75
6:2 FTS	47.1	3	14.4	NA	3	27.9	NA	3	42.8	NA	11.7	1	360	99.44	1	99.44	NA
8:2 FTS	16.5	3	16	NA	3	30.9	134	3	47.5	123.48	4.6	1	399	108.45	3	121.98	10.53
PFOSA	23.4	3	4	NA	3	7.73	NA	3	11.9	NA	6.36	1	99.7	101.57	1	101.57	NA
N-MeFOSA	NA	0	4.6	95.73	0	8.89	101.08	0	13.6	115.11	NA	0	115	98.08	4	102.5	8.47
N-EtFOSA	NA	0	9.97	91.26	0	19.3	95.56	0	29.7	112.49	NA	0	249	104.71	4	101.01	9.4
N-MeFOSAA	NA	0	4	97.35	0	7.73	118.97	0	11.9	113.89	NA	0	99.7	95.89	4	106.53	10.92
N-EtFOSAA	NA	0	4	97.09	0	7.73	119.07	0	11.9	112.62	NA	0	99.7	95.86	4	106.16	10.83
N-MeFOSE	NA	0	40	103.23	0	77.3	112.25	0	119	112.62	NA	0	997	105.5	4	108.4	4.38
N-EtFOSE	NA	0	29.9	104.64	0	58	115.3	0	89	115.27	NA	0	748	107.66	4	110.72	4.89
HFPO-DA	NA	0	15.2	100.58	0	29.4	123.76	0	45.1	111.99	NA	0	379	104.9	4	110.31	9.18
ADONA	NA	0	16.1	104.58	0	31	117.66	0	47.6	102.79	NA	0	400	84.72	4	102.44	13.22
PFMPA	NA	0	7.99	105.66	0	15.5	112.46	0	23.7	111.91	NA	0	199	96.9	4	106.73	6.79
PFMBA	NA	0	4	110.73	0	7.73	109.26	0	11.9	107.53	NA	0	99.7	98.21	4	106.43	5.3
NFDHA	NA	0	7.99	111.24	0	15.5	106.05	0	23.7	111.11	NA	0	199	99.97	4	107.09	4.98
9Cl-PF3ONS	NA	0	16	111.52	0	31	120.91	0	47.6	105.89	NA	0	400	90.77	4	107.27	11.77
11Cl-PF3OUdS	NA	0	16	106.35	0	31	112.73	0	47.6	96.36	NA	0	400	90.45	4	101.48	9.82
PFEESA	NA	0	4	107.22	0	7.73	110.54	0	11.9	112.18	NA	0	99.7	101.8	4	107.93	4.24
3:3 FTCA	NA	0	16	101.33	0	30.9	115.95	0	47.5	113.7	NA	0	399	93.8	4	106.19	9.86
5:3 FTCA	NA	0	99.7	106.66	0	193	118.28	0	297	114.89	NA	0	2490	89.23	4	107.27	12.09
7:3 FTCA	NA	0	99.7	107.92	0	193	122.44	0	297	118.4	NA	0	2490	99.63	4	112.1	9.21

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-4 WW#3 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	3.86	3	15.2	104.87	3	32.1	118.89	3	46.6	114.6	3	112.78	6.37
PFPeA	2.05	3	7.59	107.19	3	16	125.52	3	23.3	125.8	3	119.5	8.92
PFHxA	1.83	3	3.8	97.8	3	8.03	112.46	3	11.6	114.33	3	108.2	8.36
PFHpA	1.55	3	3.8	95.53	3	8.03	115.05	3	11.6	115.32	3	108.63	10.45
PFOA	NA	0	3.8	108.01	0	8.03	119.23	0	11.6	112.01	3	113.08	5.03
PFNA	NA	0	3.8	107.48	0	8.03	120.61	0	11.6	118.05	3	115.38	6.03
PFDA	NA	0	3.8	110.19	0	8.03	122.52	0	11.6	113.73	3	115.48	5.49
PFUnA	NA	0	3.8	105.17	0	8.03	118.55	0	11.6	115.18	3	112.97	6.16
PFDoA	NA	0	3.8	117.68	0	8.03	111.75	0	11.6	115.73	3	115.05	2.62
PFTrDA	NA	0	3.8	113.89	0	8.03	118.62	0	11.6	119.51	3	117.34	2.57
PFTeDA	NA	0	3.8	109.18	0	8.03	122.38	0	11.6	122.1	3	117.89	6.4
PFBS	0.645	3	3.8	107.6	3	8.03	120.06	3	11.6	115.64	3	114.44	5.52
PFPeS	NA	0	3.81	102.06	0	8.05	116.33	0	11.7	118.01	3	112.13	7.81
PFHxS	0.251	1	3.8	111.17	1	8.03	125.4	1	11.6	124.76	3	120.44	6.67
PFHpS	0.24	3	3.81	129.66	3	8.05	128.77	3	11.6	132.05	3	130.16	1.3
PFOS	372	3	3.8	NA	3	8.03	NA	3	11.6	NA	0	NA	NA
PFNS	NA	0	3.8	83.91	0	8.04	95.53	0	11.6	96.6	3	92.01	7.65
PFDS	NA	0	3.8	57.66	0	8.03	70.81	0	11.6	59.69	3	62.72	11.29
PFDoS	NA	0	nan	NA	0	8.04	28.89	0	11.6	24.85	2	26.87	10.63
4:2 FTS	6.67	3	15.2	105.88	3	32.1	117.95	3	46.6	116.65	3	113.49	5.84
6:2 FTS	9690	3	13.7	NA	3	29	NA	3	42	NA	0	NA	NA
8:2 FTS	16.3	3	15.2	NA	3	32.1	137.22	3	46.6	139.39	2	138.31	1.11
PFOSA	0.644	3	3.8	92.68	3	8.03	123.61	3	11.6	112.79	3	109.7	14.31
N-MeFOSA	NA	0	4.37	101.11	0	9.23	111.94	0	13.4	132.85	3	115.3	14
N-EtFOSA	NA	0	9.49	101.49	0	20.1	135.08	0	29.1	120.79	3	119.12	14.15
N-MeFOSAA	NA	0	3.8	85.62	0	8.03	118.95	0	11.6	120.03	3	108.2	18.08
N-EtFOSAA	NA	0	3.8	92.95	0	8.03	125.73	0	11.6	124.07	3	114.25	16.16
N-MeFOSE	NA	0	38	109.71	0	80.3	123.75	0	116	118.62	3	117.36	6.05
N-EtFOSE	NA	0	28.4	123.67	0	60.2	135.86	0	87.3	127.09	3	128.87	4.88
HFPO-DA	NA	0	14.4	110.11	0	30.5	116.41	0	44.3	115.15	3	113.89	2.93
ADONA	NA	0	15.2	99.8	0	32.2	110.95	0	46.7	120.46	3	110.4	9.36
PFMPA	NA	0	7.59	101.67	0	16	125.36	0	23.3	135.68	3	120.91	14.42
PFMBA	NA	0	3.8	101.43	0	8.03	119.85	0	11.6	134.95	3	118.74	14.14
NFDHA	NA	0	7.59	95.47	0	16	118.99	0	23.3	105.81	3	106.76	11.04
9Cl-PF3ONS	NA	0	15.2	49.8	0	32.2	77.47	0	46.7	87.24	3	71.5	27.16
11Cl-PF3OUdS	NA	0	15.2	30.92	0	32.2	45.5	0	46.6	48	3	41.47	22.23
PFEESA	NA	0	3.8	94.88	0	8.03	108.75	0	11.6	113.17	3	105.6	9.04
3:3 FTCA	NA	0	15.2	88.87	0	32.1	122.45	0	46.6	131.73	3	114.35	19.72
5:3 FTCA	NA	0	94.9	83.5	0	201	117.54	0	291	113.38	3	104.81	17.71
7:3 FTCA	NA	0	94.9	60.18	0	201	106.29	0	291	112.81	3	93.09	30.82

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-5 WW#5 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	7.86	3	16.1	86.44	3	32.8	100.61	3	49.4	98.2	3	95.08	7.97
PFPeA	10.9	3	8.06	NA	3	16.4	106.49	3	24.7	101.25	2	103.87	3.57
PFHxA	16	3	4.03	NA	3	8.21	NA	3	12.3	NA	0	NA	NA
PFHpA	4.53	3	4.03	NA	3	8.21	103.2	3	12.3	97.37	2	100.29	4.11
PFOA	8.53	3	4.03	NA	3	8.21	NA	3	12.3	93.5	1	93.5	NA
PFNA	2.88	3	4.03	79.75	3	8.21	101.33	3	12.3	99.58	3	93.55	12.81
PFDA	0.696	3	4.03	78.41	3	8.21	107.6	3	12.3	100.48	3	95.5	15.94
PFUnA	NA	0	4.03	89.49	0	8.21	102.13	0	12.3	101.65	3	97.75	7.33
PFDoA	NA	0	4.03	87.95	0	8.21	101.98	0	12.3	97.57	3	95.83	7.49
PFTrDA	NA	0	4.03	92.09	0	8.21	107.42	0	12.3	105.69	3	101.74	8.25
PFTeDA	NA	0	4.03	90.7	0	8.21	102.9	0	12.3	103	3	98.87	7.15
PFBS	12.2	3	4.03	NA	3	8.21	NA	3	12.3	108.13	1	108.13	NA
PFPeS	0.759	3	4.04	89.5	3	8.24	100.86	3	12.4	98.21	3	96.19	6.18
PFHxS	7.91	3	4.03	NA	3	8.21	105.91	3	12.3	100.2	2	103.05	3.92
PFHpS	0.325	3	4.04	84.43	3	8.23	103.95	3	12.3	100.08	3	96.15	10.75
PFOS	12.6	3	4.03	NA	3	8.21	NA	3	12.3	NA	0	NA	NA
PFNS	NA	0	4.04	76.66	0	8.23	106.74	0	12.3	105.18	3	96.19	17.6
PFDS	NA	0	4.03	68.93	0	8.21	89.59	0	12.3	88.63	3	82.38	14.15
PFDoS	NA	0	4.04	56.82	0	8.22	72.67	0	12.3	73.61	3	67.7	13.94
4:2 FTS	NA	0	16.1	87.32	0	32.8	106.07	0	49.4	97.92	3	97.1	9.68
6:2 FTS	NA	0	14.5	111.27	0	29.6	111.81	0	44.5	104.79	3	109.29	3.57
8:2 FTS	NA	0	16.1	105.02	0	32.8	122.57	0	49.4	119.19	3	115.59	8.05
PFOSA	0.323	3	4.03	89.35	3	8.21	111.61	3	12.3	112	3	104.32	12.43
N-MeFOSA	NA	0	4.63	79.36	0	9.44	97.49	0	14.2	106.07	3	94.3	14.46
N-EtFOSA	NA	0	10.1	82.32	0	20.5	101.3	0	30.8	103.18	3	95.6	12.07
N-MeFOSAA	0.844	1	4.03	107.23	1	8.21	171.35	1	12.3	186.2	3	154.92	27.09
N-EtFOSAA	0.357	1	4.03	99.03	1	8.21	152.72	1	12.3	154.63	3	135.46	23.3
N-MeFOSE	NA	0	40.3	82.11	0	82.1	99.27	0	123	95.92	3	92.43	9.84
N-EtFOSE	NA	0	30.2	85.49	0	61.5	101.75	0	92.5	100.22	3	95.82	9.37
HFPO-DA	NA	0	15.3	89.83	0	31.2	99.61	0	46.9	98.69	3	96.04	5.62
ADONA	NA	0	16.1	83.83	0	32.9	95.45	0	49.5	98.25	3	92.51	8.27
PFMPA	NA	0	8.06	97.36	0	16.4	105.27	0	24.7	101.06	3	101.23	3.91
PFMBA	NA	0	4.03	91.57	0	8.21	99.9	0	12.3	91.28	3	94.25	5.2
NFDHA	NA	0	8.06	68.36	0	16.4	109.98	0	24.7	75.48	3	84.61	26.31
9Cl-PF3ONS	NA	0	16.1	83.79	0	32.9	91.98	0	49.4	100.13	3	91.97	8.89
11Cl-PF3OUdS	NA	0	16.1	71.18	0	32.9	89.36	0	49.4	89.24	3	83.26	12.57
PFEESA	NA	0	4.03	87.63	0	8.21	101.46	0	12.3	103.22	3	97.44	8.76
3:3 FTCA	NA	0	16.1	108.21	0	32.8	36.57	0	49.4	51.53	3	65.44	57.75
5:3 FTCA	NA	0	101	101.22	0	205	88.33	0	308	87.6	3	92.38	8.29
7:3 FTCA	NA	0	101	94.25	0	205	98.85	0	308	98.66	3	97.25	2.68

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-6: WW #6 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	4.31	3	15.8	96.96	3	30.9	98.89	3	47.2	98.99	3	98.28	1.16
PFPeA	6.99	3	7.92	102.72	3	15.5	106.95	3	23.6	105.4	3	105.02	2.04
PFHxA	6.55	3	3.96	NA	3	7.72	112.42	3	11.8	111.41	2	111.92	0.64
PFHpA	3.65	3	3.96	91.86	3	7.72	101.29	3	11.8	100.71	3	97.95	5.39
PFOA	10.3	3	3.96	NA	3	7.72	NA	3	11.8	102.91	1	102.91	NA
PFNA	1.4	3	3.96	101.4	3	7.72	99.82	3	11.8	98.84	3	100.02	1.29
PFDA	0.839	3	3.96	99.04	3	7.72	112.48	3	11.8	106.45	3	105.99	6.35
PFUnA	0.34	2	3.96	97.46	2	7.72	96.1	2	11.8	96.56	3	96.71	0.72
PFDoA	NA	0	3.96	88.6	0	7.72	105.45	0	11.8	109.32	3	101.12	10.89
PFTrDA	0.268	2	3.96	91.69	2	7.72	121.86	2	11.8	107.62	3	107.06	14.1
PFTeDA	NA	0	3.96	90.57	0	7.72	105.82	0	11.8	102.82	3	99.74	8.1
PFBS	4.99	3	3.96	NA	3	7.72	107.25	3	11.8	102.91	2	105.08	2.92
PFPeS	0.484	3	3.97	97.6	3	7.74	99.31	3	11.8	98.44	3	98.45	0.86
PFHxS	3.61	3	3.96	95.26	3	7.72	105.71	3	11.8	104.44	3	101.8	5.6
PFHpS	0.22	3	3.97	99.47	3	7.73	104.77	3	11.8	96.16	3	100.13	4.34
PFOS	11.5	3	3.96	NA	3	7.72	NA	3	11.8	105.08	1	105.08	NA
PFNS	NA	0	3.97	94.08	0	7.73	105.01	0	11.8	105.93	3	101.68	6.48
PFDS	NA	0	3.96	82.48	0	7.72	96.04	0	11.8	92.66	3	90.39	7.8
PFDoS	NA	0	3.97	55.44	0	7.73	57.67	0	11.8	50.79	3	54.63	6.42
4:2 FTS	NA	0	15.8	89.26	0	30.9	102.46	0	47.2	102.26	3	97.99	7.72
6:2 FTS	NA	0	14.3	118.42	0	27.8	109	0	42.5	104.78	3	110.73	6.31
8:2 FTS	NA	0	15.8	111.35	0	30.9	122.43	0	47.2	118.71	3	117.5	4.8
PFOSA	1.54	3	3.96	105.89	3	7.72	124.32	3	11.8	134.69	3	121.64	11.99
N-MeFOSA	NA	0	4.55	90.82	0	8.87	104.02	0	13.6	102.21	3	99.02	7.23
N-EtFOSA	NA	0	9.9	90.03	0	19.3	102.59	0	29.5	106.44	3	99.69	8.61
N-MeFOSAA	1.3	2	3.96	230.25	2	7.72	368.86	2	11.8	411.9	3	337	28.17
N-EtFOSAA	0.723	3	3.96	191.08	3	7.72	243.77	3	11.8	260.82	3	231.89	15.68
N-MeFOSE	1.86	2	39.6	76.32	2	77.2	77.73	2	118	79.24	3	77.77	1.87
N-EtFOSE	NA	0	29.7	91.11	0	57.8	98.11	0	88.4	93.82	3	94.35	3.74
HFPO-DA	NA	0	15	95.76	0	29.3	102.72	0	44.8	101.27	3	99.92	3.68
ADONA	NA	0	15.9	95.58	0	30.9	102.92	0	47.3	101.13	3	99.88	3.83
PFMPA	NA	0	7.92	112.3	0	15.5	108.85	0	23.6	107.06	3	109.4	2.43
PFMBA	NA	0	3.96	101.42	0	7.72	102.43	0	11.8	98.02	3	100.62	2.29
NFDHA	NA	0	7.92	66.27	0	15.5	86.06	0	23.6	88.28	3	80.2	15.1
9Cl-PF3ONS	NA	0	15.9	92.01	0	30.9	100.88	0	47.3	102.54	3	98.48	5.75
11Cl-PF3OUdS	NA	0	15.9	76.49	0	30.9	93.56	0	47.2	92.66	3	87.57	10.97
PFEESA	NA	0	3.96	98.89	0	7.72	98.41	0	11.8	101.41	3	99.57	1.62
3:3 FTCA	NA	0	15.8	135.53	0	30.9	104.88	0	47.2	105.58	3	115.33	15.17
5:3 FTCA	46.3	3	99	140.38	3	193	110.07	3	295	111.44	3	120.63	14.19
7:3 FTCA	NA	0	99	109.06	0	193	97.05	0	295	99.32	3	101.81	6.26

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-7 WW #7 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	1.22	3	16.2	93.11	3	31.9	96.71	3	47.9	98.07	3	95.96	2.67
PFPeA	0.833	3	8.11	101.41	3	16	99.37	3	24	100.71	3	100.5	1.03
PFHxA	1.28	3	4.06	83.88	3	7.98	95.62	3	12	91.56	3	90.35	6.6
PFHpA	0.369	3	4.06	94.66	3	7.98	96.06	3	12	96.08	3	95.6	0.86
PFOA	1.24	3	4.06	94.91	3	7.98	99.22	3	12	98.57	3	97.57	2.38
PFNA	0.412	3	4.06	93.76	3	7.98	99.28	3	12	98.76	3	97.27	3.13
PFDA	NA	0	4.06	98.36	0	7.98	104.83	0	12	104.62	3	102.6	3.58
PFUnA	NA	0	4.06	86.75	0	7.98	97.93	0	12	98.54	3	94.41	7.03
PFDoA	NA	0	4.06	97.07	0	7.98	104.19	0	12	109.41	3	103.56	5.98
PFTrDA	NA	0	4.06	136.24	0	7.98	135.75	0	12	135.44	3	135.81	0.3
PFTeDA	0.478	3	4.06	95.11	3	7.98	95.88	3	12	95.98	3	95.66	0.49
PFBS	0.671	3	4.06	98.99	3	7.98	102.83	3	12	104.09	3	101.97	2.6
PFPeS	1.15	3	4.07	88.86	3	8	98.6	3	12	97.52	3	94.99	5.62
PFHxS	0.37	3	4.06	117.02	3	7.98	113.81	3	12	111.36	3	114.06	2.49
PFHpS	NA	0	4.07	115.69	0	8	122.42	0	12	120.15	3	119.42	2.87
PFOS	0.579	3	4.06	96.47	3	7.98	98.57	3	12	99.3	3	98.11	1.5
PFNS	0.727	1	4.06	90.86	1	7.99	90.93	1	12	96.87	3	92.89	3.72
PFDS	0.76	1	4.06	73.4	1	7.98	61.42	1	12	65.45	3	66.76	9.13
PFDoS	NA	0	4.06	82.56	0	7.99	77.3	0	12	75.71	3	78.52	4.56
4:2 FTS	NA	0	16.2	99.84	0	31.9	99.22	0	47.9	101.85	3	100.3	1.37
6:2 FTS	NA	0	14.6	107.56	0	28.8	144.85	0	43.2	149.19	3	133.87	17.1
8:2 FTS	NA	0	16.2	110.41	0	31.9	109.39	0	47.9	114.34	3	111.38	2.34
PFOSA	NA	0	4.06	101.42	0	7.98	103.34	0	12	102.7	3	102.49	0.95
N-MeFOSA	NA	0	4.66	88.85	0	9.17	105.14	0	13.8	99.57	3	97.85	8.46
N-EtFOSA	0.564	1	10.1	88.4	1	20	93.29	1	29.9	102.42	3	94.7	7.52
N-MeFOSAA	NA	0	4.06	100.05	0	7.98	99.76	0	12	89.05	3	96.29	6.51
N-EtFOSAA	NA	0	4.06	54.83	0	7.98	74.67	0	12	118.26	3	82.58	39.29
N-MeFOSE	1.23	2	40.6	94.97	2	79.8	95.45	2	120	93.65	3	94.69	0.99
N-EtFOSE	1.31	3	30.4	105.63	3	59.8	104.97	3	89.7	102.3	3	104.3	1.69
HFPO-DA	NA	0	15.4	97.4	0	30.3	101.67	0	45.5	99.1	3	99.39	2.16
ADONA	NA	0	16.3	105.36	0	32	108.79	0	48	113.86	3	109.34	3.91
PFMPA	NA	0	8.11	97.88	0	16	104.39	0	24	103.33	3	101.87	3.43
PFMBA	NA	0	4.06	91.92	0	7.98	94.58	0	12	95.83	3	94.11	2.12
NFDHA	NA	0	8.11	78.07	0	16	42.25	0	24	50.54	3	56.95	32.92
9Cl-PF3ONS	NA	0	16.3	66.62	0	32	59.39	0	48	62.32	3	62.78	5.79
11Cl-PF3OUdS	NA	0	16.2	55.19	0	32	50.25	0	48	51.39	3	52.28	4.95
PFEESA	NA	0	4.06	105.72	0	7.98	116.25	0	12	108.89	3	110.29	4.9
3:3 FTCA	NA	0	16.2	27.22	0	31.9	23.76	0	47.9	29.68	3	26.89	11.06
5:3 FTCA	NA	0	101	76.63	0	200	68.65	0	299	73.23	3	72.84	5.5
7:3 FTCA	NA	0	101	79.38	0	200	72.29	0	299	77.4	3	76.36	4.79

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-8 WW #8 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	1.65	3	16.1	95.81	3	30.9	87.01	3	47.4	95.05	3	92.62	5.26
PFPeA	1.97	3	8.06	89.3	3	15.5	92.22	3	23.7	100.55	3	94.02	6.21
PFHxA	2.19	3	4.03	94.23	3	7.73	84.65	3	11.8	93.02	3	90.63	5.75
PFHpA	1.93	3	4.03	99.83	3	7.73	89.4	3	11.8	95	3	94.74	5.51
PFOA	4.57	3	4.03	NA	3	7.73	86.21	3	11.8	99.7	2	92.96	10.26
PFNA	1.66	3	4.03	92.9	3	7.73	86.64	3	11.8	95.81	3	91.78	5.1
PFDA	3.44	3	4.03	93.25	3	7.73	86.89	3	11.8	96.25	3	92.13	5.19
PFUnA	0.582	3	4.03	101.82	3	7.73	90.11	3	11.8	100.42	3	97.45	6.56
PFDoA	1.68	3	4.03	103.71	3	7.73	92.53	3	11.8	92.77	3	96.34	6.63
PFTrDA	0.24	2	4.03	102.31	2	7.73	95.09	2	11.8	102.72	3	100.04	4.29
PFTeDA	0.359	1	4.03	93.15	1	7.73	89.11	1	11.8	97.5	3	93.25	4.5
PFBS	1.61	3	4.03	95.48	3	7.73	93.97	3	11.8	101.56	3	97.01	4.14
PFPeS	NA	0	4.04	98.12	0	7.75	90.03	0	11.9	98.34	3	95.5	4.96
PFHxS	0.668	3	4.03	105.08	3	7.73	99.67	3	11.8	104.77	3	103.18	2.94
PFHpS	NA	0	4.04	98.07	0	7.75	94	0	11.9	98.03	3	96.7	2.42
PFOS	2.83	3	4.03	96.93	3	7.73	93.51	3	11.8	105.71	3	98.72	6.38
PFNS	NA	0	4.04	95.87	0	7.74	97.8	0	11.9	96.29	3	96.65	1.05
PFDS	NA	0	4.03	86.82	0	7.73	84.58	0	11.8	91.86	3	87.75	4.25
PFDoS	NA	0	4.04	80.67	0	7.74	75.94	0	11.9	82.95	3	79.86	4.48
4:2 FTS	NA	0	16.1	103.04	0	30.9	90.62	0	47.4	101.36	3	98.34	6.85
6:2 FTS	4.88	1	14.5	131.45	1	27.90*	NA	1	42.8	88.81	2	110.13	27.38
8:2 FTS	NA	0	16.1	118.74	0	30.9	105.82	0	47.4	119.69	3	114.75	6.75
PFOSA	1.8	3	4.03	104.13	3	7.73	96.47	3	11.8	108.47	3	103.02	5.9
N-MeFOSA	NA	0	4.64	90.13	0	8.89	85.26	0	13.6	97.79	3	91.06	6.93
N-EtFOSA	NA	0	10.1	84.26	0	19.3	80.51	0	29.6	94.46	3	86.41	8.35
N-MeFOSAA	NA	0	4.03	133.54	0	7.73	135.54	0	11.8	123.74	3	130.94	4.82
N-EtFOSAA	NA	0	4.03	111.4	0	7.73	116.68	0	11.8	118.54	3	115.54	3.2
N-MeFOSE	NA	0	40.3	96.04	0	77.3	88.67	0	118	97.74	3	94.15	5.12
N-EtFOSE	NA	0	30.2	96.39	0	58	93.16	0	88.9	99.82	3	96.46	3.45
HFPO-DA	NA	0	15.3	93.45	0	29.4	84.47	0	45	94.33	3	90.75	6.01
ADONA	NA	0	16.2	110.25	0	31	82.04	0	47.6	85.53	3	92.61	16.6
PFMPA	NA	0	8.06	101.06	0	15.5	86.64	0	23.7	94.37	3	94.02	7.68
PFMBA	NA	0	4.03	94.22	0	7.73	85.18	0	11.8	94.91	3	91.44	5.94
NFDHA	NA	0	8.06	79.94	0	15.5	78.86	0	23.7	89.56	3	82.79	7.11
9Cl-PF3ONS	NA	0	16.2	128.1	0	31	82.79	0	47.5	85.49	3	98.79	25.72
11Cl-PF3OUdS	NA	0	16.2	116.46	0	31	77.95	0	47.5	80.29	3	91.57	23.58
PFEESA	NA	0	4.03	107.19	0	7.73	85.34	0	11.8	96.3	3	96.28	11.35
3:3 FTCA	NA	0	16.1	95.48	0	30.9	88.35	0	47.4	97.66	3	93.83	5.19
5:3 FTCA	NA	0	101	90.68	0	193	91.55	0	296	97.83	3	93.35	4.18
7:3 FTCA	NA	0	101	93.66	0	193	85.17	0	296	93.9	3	90.91	5.47

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-9: WW #9 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	1.41	3	15.9	97.43	3	31	86.73	3	46.8	96.05	3	93.41	6.23
PFPeA	1.57	3	7.96	113.22	3	15.5	92.65	3	23.4	99.42	3	101.76	10.3
PFHxA	3.21	3	3.98	103.45	3	7.74	85.8	3	11.7	94.79	3	94.68	9.32
PFHpA	0.521	3	3.98	98.58	3	7.74	91.14	3	11.7	92.44	3	94.05	4.23
PFOA	1.97	3	3.98	91.27	3	7.74	83.56	3	11.7	93.71	3	89.51	5.92
PFNA	0.465	3	3.98	100.54	3	7.74	87.34	3	11.7	97.15	3	95.01	7.21
PFDA	0.404	2	3.98	100.42	2	7.74	89.21	2	11.7	99.95	3	96.53	6.57
PFUnA	0.29	1	3.98	101.43	1	7.74	87.74	1	11.7	96.33	3	95.17	7.27
PFDoA	NA	0	3.98	104.51	0	7.74	89.42	0	11.7	99.7	3	97.88	7.87
PFTrDA	NA	0	3.98	100.75	0	7.74	96.77	0	11.7	102.83	3	100.12	3.08
PFTeDA	0.348	2	3.98	98.91	2	7.74	88.67	2	11.7	98.42	3	95.34	6.06
PFBS	1.61	3	3.98	108.22	3	7.74	95.4	3	11.7	103.86	3	102.49	6.36
PFPeS	1.95	3	3.99	95.61	3	7.76	89.78	3	11.7	96.93	3	94.11	4.04
PFHxS	0.531	3	3.98	107.36	3	7.74	97.44	3	11.7	105.88	3	103.56	5.17
PFHpS	NA	0	3.99	98.59	0	7.75	89.31	0	11.7	97.93	3	95.28	5.43
PFOS	1.89	3	3.98	107.45	3	7.74	91.35	3	11.7	103.92	3	100.91	8.39
PFNS	NA	0	3.99	103.69	0	7.75	91.91	0	11.7	107.6	3	101.07	8.08
PFDS	NA	0	3.98	79.99	0	7.74	73.6	0	11.7	83	3	78.87	6.09
PFDoS	NA	0	3.98	47.53	0	7.75	41.9	0	11.7	42.33	3	43.92	7.13
4:2 FTS	NA	0	15.9	98.76	0	31	92.29	0	46.8	100.14	3	97.06	4.31
6:2 FTS	NA	0	14.3	115.85	0	27.90*	NA	0	42.2	101.02	2	108.43	9.67
8:2 FTS	NA	0	15.9	115.17	0	31	107.48	0	46.8	109.68	3	110.78	3.57
PFOSA	2.66	3	3.98	133.38	3	7.74	92.34	3	11.7	100.88	3	108.87	19.89
N-MeFOSA	NA	0	4.58	93.74	0	8.9	88.25	0	13.5	98.73	3	93.57	5.6
N-EtFOSA	NA	0	9.95	96.51	0	19.4	89.56	0	29.3	100.08	3	95.38	5.61
N-MeFOSAA	NA	0	3.98	294.42	0	7.74	243.97	0	11.7	240.94	3	259.78	11.56
N-EtFOSAA	0.457	3	3.98	241.43	3	7.74	178.91	3	11.7	194.89	3	205.08	15.84
N-MeFOSE	NA	0	39.8	90.6	0	77.4	81.3	0	117	91.49	3	87.8	6.43
N-EtFOSE	NA	0	29.8	89.89	0	58	85.23	0	87.8	95.99	3	90.37	5.97
HFPO-DA	NA	0	15.1	98.68	0	29.4	88.64	0	44.5	93.62	3	93.65	5.36
ADONA	NA	0	16	114.85	0	31	97.52	0	47	103.86	3	105.41	8.32
PFMPA	NA	0	7.96	133.61	0	15.5	118.49	0	23.4	132.33	3	128.14	6.54
PFMBA	NA	0	3.98	110.9	0	7.74	103.89	0	11.7	113.06	3	109.28	4.39
NFDHA	NA	0	7.96	62.88	0	15.5	56.67	0	23.4	75.14	3	64.9	14.49
9Cl-PF3ONS	NA	0	15.9	116.55	0	31	98.36	0	46.9	106.99	3	107.3	8.48
11Cl-PF3OUdS	NA	0	15.9	97.08	0	31	88.96	0	46.9	93.6	3	93.21	4.37
PFEESA	NA	0	3.98	115.33	0	7.74	106.16	0	11.7	113.91	3	111.8	4.42
3:3 FTCA	NA	0	15.9	100.43	0	31	96.55	0	46.8	109.7	3	102.23	6.61
5:3 FTCA	NA	0	99.5	163.17	0	194	148.11	0	293	158.7	3	156.66	4.94
7:3 FTCA	NA	0	99.5	112.56	0	194	109.36	0	293	120.6	3	114.17	5.07

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-10 WW #10 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	6.49	3	15.7	97.29	3	31.2	91.51	3	46.7	95.31	3	94.7	3.1
PFPeA	5.19	3	7.84	100.02	3	15.6	95.4	3	23.4	100.43	3	98.62	2.83
PFHxA	13.8	3	3.92	NA	3	7.81	NA	3	11.7	NA	0	NA	NA
PFHpA	1.64	3	3.92	102.46	3	7.81	90.87	3	11.7	91.69	3	95.01	6.81
PFOA	4.27	3	3.92	NA	3	7.81	98.17	3	11.7	91.92	2	95.04	4.65
PFNA	0.747	3	3.92	102.19	3	7.81	93.08	3	11.7	97.28	3	97.52	4.68
PFDA	1.33	3	3.92	103.32	3	7.81	100.23	3	11.7	99.8	3	101.12	1.9
PFUnA	NA	0	3.92	98.2	0	7.81	97.28	0	11.7	102.56	3	99.35	2.84
PFDoA	NA	0	3.92	100.85	0	7.81	94.22	0	11.7	97.4	3	97.49	3.4
PFTrDA	NA	0	3.92	106.48	0	7.81	92.57	0	11.7	92.27	3	97.11	8.36
PFTeDA	NA	0	3.92	116.33	0	7.81	98.65	0	11.7	99.44	3	104.81	9.53
PFBS	7.13	3	3.92	NA	3	7.81	104.76	3	11.7	108.3	2	106.53	2.35
PFPeS	0.228	1	3.93	97.84	1	7.83	95.16	1	11.7	99.47	3	97.49	2.23
PFHxS	2.01	3	3.92	118.28	3	7.81	100.2	3	11.7	103.02	3	107.17	9.08
PFHpS	NA	0	3.93	102.19	0	7.82	93.74	0	11.7	94.29	3	96.74	4.89
PFOS	16.1	3	3.92	NA	3	7.81	NA	3	11.7	NA	0	NA	NA
PFNS	NA	0	3.93	95.59	0	7.82	86.63	0	11.7	91.74	3	91.32	4.92
PFDS	NA	0	3.92	80.02	0	7.81	76.38	0	11.7	75.57	3	77.32	3.07
PFDoS	NA	0	3.93	59.08	0	7.82	41.07	0	11.7	40.99	3	47.05	22.15
4:2 FTS	NA	0	15.7	101.91	0	31.2	99.83	0	46.7	98.77	3	100.17	1.59
6:2 FTS	12.1	3	14.1	108.95	3	28.13*	NA	3	42.2	99.33	2	104.14	6.53
8:2 FTS	NA	0	15.7	129.25	0	31.2	114.7	0	46.7	116.38	3	120.11	6.63
PFOSA	1.02	3	3.92	112.75	3	7.81	97.13	3	11.7	102.67	3	104.18	7.6
N-MeFOSA	NA	0	4.51	93.59	0	8.97	91.38	0	13.4	98.04	3	94.34	3.6
N-EtFOSA	NA	0	9.8	93.44	0	19.5	93.21	0	29.2	95.44	3	94.03	1.31
N-MeFOSAA	1.74	3	3.92	149.64	3	7.81	161.85	3	11.7	141.51	3	151	6.78
N-EtFOSAA	0.801	3	3.92	135.69	3	7.81	141.86	3	11.7	136.44	3	138	2.44
N-MeFOSE	NA	0	39.2	96.94	0	78.1	92.93	0	117	97.72	3	95.86	2.68
N-EtFOSE	NA	0	29.4	98.97	0	58.5	97.98	0	87.6	102.13	3	99.69	2.18
HFPO-DA	NA	0	14.9	102.68	0	29.6	92.17	0	44.4	96.62	3	97.15	5.43
ADONA	NA	0	15.7	104.67	0	31.3	92.16	0	46.9	92.52	3	96.45	7.38
PFMPA	NA	0	7.84	110.34	0	15.6	104.53	0	23.4	105.27	3	106.71	2.96
PFMBA	NA	0	3.92	95.42	0	7.81	90.49	0	11.7	95.42	3	93.78	3.03
NFDHA	NA	0	7.84	47.88	0	15.6	55.88	0	23.4	72.84	3	58.87	21.66
9Cl-PF3ONS	NA	0	15.7	100.01	0	31.3	89.83	0	46.8	91.23	3	93.69	5.89
11Cl-PF3OUdS	NA	0	15.7	81.53	0	31.3	72.96	0	46.8	71.49	3	75.33	7.2
PFEESA	NA	0	3.92	102.3	0	7.81	96.34	0	11.7	97.42	3	98.69	3.22
3:3 FTCA	NA	0	15.7	127.19	0	31.2	128.53	0	46.7	130.06	3	128.59	1.12
5:3 FTCA	27.6	3	98	138.42	3	195	128.59	3	292	128.74	3	131.92	4.27
7:3 FTCA	NA	0	98	113.24	0	195	106.54	0	292	108.32	3	109.37	3.17

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-11 SW COP-SW01 TO 11 Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	3.97	3	15.4	91.46	3	31.3	110.2	3	46.7	96.71	3	99.46	9.72
PFPeA	3.4	3	7.68	98.51	3	15.7	113.95	3	23.3	108.66	3	107.04	7.33
PFHxA	5.33	3	3.84	NA	3	7.83	100.47	3	11.7	98.69	2	99.58	1.26
PFHpA	3.6	3	3.84	86.05	3	7.83	105.42	3	11.7	81.06	3	90.85	14.16
PFOA	19.3	3	3.84	NA	3	7.83	NA	3	11.7	NA	0	NA	NA
PFNA	2.06	3	3.84	90.69	3	7.83	105.86	3	11.7	99.42	3	98.66	7.72
PFDA	0.398	3	3.84	91.3	3	7.83	113.14	3	11.7	102.22	3	102.22	10.68
PFUnA	0.289	3	3.84	95.88	3	7.83	109.35	3	11.7	103.86	3	103.03	6.58
PFDoA	0.441	3	3.84	92.73	3	7.83	109.76	3	11.7	103.33	3	101.94	8.44
PFTrDA	0.963	3	3.84	98.1	3	7.83	115.65	3	11.7	103.14	3	105.63	8.55
PFTeDA	0.467	3	3.84	96.77	3	7.83	108.64	3	11.7	102.2	3	102.54	5.79
PFBS	7.22	3	3.84	NA	3	7.83	102.12	3	11.7	104.2	2	103.16	1.43
PFPeS	6.01	3	3.85	NA	3	7.85	105.94	3	11.7	104.11	2	105.02	1.23
PFHxS	48.8	3	3.84	NA	3	7.83	NA	3	11.7	NA	0	NA	NA
PFHpS	3.12	3	3.85	84.7	3	7.85	106.67	3	11.7	96.71	3	96.02	11.46
PFOS	96.8	3	3.84	NA	3	7.83	NA	3	11.7	NA	0	NA	NA
PFNS	NA	0	3.85	97.58	0	7.84	100.81	0	11.7	106.76	3	101.72	4.58
PFDS	NA	0	3.84	86.82	0	7.83	100.38	0	11.7	94.99	3	94.07	7.26
PFDoS	NA	0	3.84	59.08	0	7.84	66.59	0	11.7	68.87	3	64.85	7.9
4:2 FTS	NA	0	15.4	98.49	0	31.3	112.67	0	46.7	100.44	3	103.86	7.4
6:2 FTS	NA	0	13.8	141.26	0	28.2	137.78	0	42.1	110.57	3	129.87	12.94
8:2 FTS	NA	0	15.4	105.39	0	31.3	127.19	0	46.7	114.2	3	115.59	9.49
PFOSA	0.991	3	3.84	94.48	3	7.83	110.13	3	11.7	122.44	3	109.02	12.85
N-MeFOSA	NA	0	4.42	87.38	0	9.01	111.78	0	13.4	100.91	3	100.02	12.22
N-EtFOSA	NA	0	9.6	80.03	0	19.6	109.95	0	29.2	101.19	3	97.06	15.85
N-MeFOSAA	NA	0	3.84	192.11	0	7.83	198.45	0	11.7	204.78	3	198.45	3.19
N-EtFOSAA	NA	0	3.84	117.46	0	7.83	133.32	0	11.7	129.69	3	126.83	6.55
N-MeFOSE	NA	0	38.4	86.52	0	78.3	107.25	0	117	99.7	3	97.82	10.73
N-EtFOSE	NA	0	28.8	91.77	0	58.7	117.28	0	87.5	107.14	3	105.4	12.19
HFPO-DA	NA	0	14.6	99.19	0	29.8	114.74	0	44.4	100.11	3	104.68	8.33
ADONA	NA	0	15.4	97.35	0	31.4	107.16	0	46.8	103.14	3	102.55	4.81
PFMPA	NA	0	7.68	92.9	0	15.7	109.92	0	23.3	95.35	3	99.39	9.26
PFMBA	NA	0	3.84	85.76	0	7.83	100.64	0	11.7	97.67	3	94.69	8.32
NFDHA	NA	0	7.68	93.58	0	15.7	102.74	0	23.3	95.07	3	97.13	5.06
9Cl-PF3ONS	NA	0	15.4	102.09	0	31.4	107.08	0	46.8	106.22	3	105.13	2.54
11Cl-PF3OUdS	NA	0	15.4	89.03	0	31.3	105.65	0	46.8	99.93	3	98.2	8.6
PFEESA	NA	0	3.84	93.21	0	7.83	108.86	0	11.7	98.33	3	100.13	7.97
3:3 FTCA	NA	0	15.4	98.05	0	31.3	107.61	0	46.7	99.13	3	101.6	5.15
5:3 FTCA	NA	0	96	91.73	0	196	105.38	0	292	94	3	97.04	7.53
7:3 FTCA	NA	0	96	76.8	0	196	99.27	0	292	98.41	3	91.49	13.91

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-12 SW Marine Surface Water Matrix Spike Recovery (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	1.51	3	14.5	93.5	3	29.1	107.31	3	44.7	101.25	3	100.69	6.88
PFPeA	0.415	3	7.24	97.58	3	14.6	110.42	3	22.4	110.33	3	106.11	6.97
PFHxA	NA	0	3.62	93.37	0	7.28	106.09	0	11.2	101.75	3	100.41	6.44
PFHpA	NA	0	3.62	91.06	0	7.28	111.96	0	11.2	104.16	3	102.39	10.32
PFOA	0.271	1	3.62	88	1	7.28	102.87	1	11.2	102.57	3	97.81	8.69
PFNA	NA	0	3.62	93.54	0	7.28	107.24	0	11.2	106.03	3	102.27	7.42
PFDA	NA	0	3.62	94.65	0	7.28	110.58	0	11.2	109.8	3	105.01	8.55
PFUnA	NA	0	3.62	90.41	0	7.28	107.84	0	11.2	103.02	3	100.42	8.96
PFDoA	NA	0	3.62	86.28	0	7.28	103.94	0	11.2	99.12	3	96.45	9.46
PFTrDA	NA	0	3.62	87.19	0	7.28	108.61	0	11.2	105.12	3	100.31	11.46
PFTeDA	NA	0	3.62	87.2	0	7.28	110.77	0	11.2	105.99	3	101.32	12.3
PFBS	NA	0	3.62	94.37	0	7.28	102.06	0	11.2	109.9	3	102.11	7.6
PFPeS	NA	0	3.63	95.13	0	7.3	98.59	0	11.2	104.75	3	99.49	4.9
PFHxS	NA	0	3.62	97.34	0	7.28	111.63	0	11.2	111.89	3	106.96	7.79
PFHpS	NA	0	3.63	94.47	0	7.29	105.35	0	11.2	101.49	3	100.44	5.49
PFOS	NA	0	3.62	99.16	0	7.28	115.94	0	11.2	111.34	3	108.81	7.97
PFNS	NA	0	3.63	76.88	0	7.29	89.17	0	11.2	105.95	3	90.67	16.1
PFDS	NA	0	3.62	74.12	0	7.28	95.01	0	11.2	95.17	3	88.1	13.74
PFDoS	NA	0	3.62	67.99	0	7.29	91.08	0	11.2	89	3	82.69	15.45
4:2 FTS	NA	0	14.5	96.77	0	29.1	110.88	0	44.7	105.44	3	104.36	6.82
6:2 FTS	4.65	1	13.1	65.44	1	26.2	127.39	1	40.4	99.85	3	97.56	31.82
8:2 FTS	NA	0	14.5	100.45	0	29.1	119.24	0	44.7	115.02	3	111.57	8.83
PFOSA	0.456	3	3.62	88.51	3	7.28	112.02	3	11.2	133.2	3	111.25	20.1
N-MeFOSA	NA	0	4.16	79.01	0	8.37	105.22	0	12.9	104.91	3	96.38	15.61
N-EtFOSA	NA	0	9.05	80.54	0	18.2	106.04	0	28	102.88	3	96.49	14.41
N-MeFOSAA	NA	0	3.62	105.71	0	7.28	132.39	0	11.2	143.64	3	127.25	15.31
N-EtFOSAA	NA	0	3.62	100.81	0	7.28	140.17	0	11.2	159.22	3	133.4	22.33
N-MeFOSE	NA	0	36.2	87.84	0	72.8	107.28	0	112	105.67	3	100.26	10.76
N-EtFOSE	NA	0	27.1	90.9	0	54.6	112.59	0	83.9	108.26	3	103.91	11.04
HFPO-DA	NA	0	13.8	83.3	0	27.7	110.12	0	42.5	103.33	3	98.92	14.1
ADONA	NA	0	14.5	86.93	0	29.2	108.9	0	44.9	97.34	3	97.72	11.25
PFMPA	NA	0	7.24	92.95	0	14.6	103.66	0	22.4	96.68	3	97.76	5.56
PFMBA	NA	0	3.62	91.34	0	7.28	105.18	0	11.2	100.27	3	98.93	7.09
NFDHA	NA	0	7.24	79.02	0	14.6	113.77	0	22.4	139.28	3	110.69	27.32
9Cl-PF3ONS	NA	0	14.5	77.46	0	29.2	105.82	0	44.8	99.18	3	94.16	15.75
11Cl-PF3OUdS	NA	0	14.5	63.79	0	29.2	93.14	0	44.8	90.8	3	82.58	19.75
PFEESA	NA	0	3.62	94.56	0	7.28	107.28	0	11.2	100.58	3	100.81	6.31
3:3 FTCA	NA	0	14.5	87.55	0	29.1	99.2	0	44.7	97.03	3	94.59	6.55
5:3 FTCA	NA	0	90.5	80.99	0	182	91.94	0	280	91.13	3	88.02	6.93
7:3 FTCA	NA	0	90.5	74.95	0	182	88.83	0	280	88.8	3	84.19	9.51

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-13 SW RP-SW01 to 11 Matrix Spike Recovery (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	3.84	3	16.5	104.29	3	32	118.49	3	47	115.44	3	112.74	6.63
PFPeA	8.01	3	8.28	118.19	3	16	130.04	3	23.5	122.54	3	123.59	4.85
PFHxA	9.48	3	4.14	NA	3	8	NA	3	11.7	114.37	1	114.37	NA
PFHpA	6.95	3	4.14	NA	3	8	125.56	3	11.7	118.65	2	122.1	4
PFOA	12.7	3	4.14	NA	3	8	NA	3	11.7	NA	0	NA	NA
PFNA	3.92	3	4.14	111.51	3	8	123.85	3	11.7	121.43	3	118.93	5.5
PFDA	0.378	2	4.14	103.47	2	8	121.81	2	11.7	121.16	3	115.48	9.01
PFUnA	NA	0	4.14	103.26	0	8	119.96	0	11.7	119.85	3	114.36	8.4
PFDoA	NA	0	4.14	106.19	0	8	123.36	0	11.7	113.04	3	114.2	7.57
PFTrDA	NA	0	4.14	109.56	0	8	124.54	0	11.7	119.61	3	117.9	6.47
PFTeDA	NA	0	4.14	106.53	0	8	122.51	0	11.7	117.93	3	115.66	7.12
PFBS	4.76	3	4.14	NA	3	8	123.82	3	11.7	115.97	2	119.89	4.63
PFPeS	5.23	3	4.15	NA	3	8.03	125.46	3	11.8	112.71	2	119.09	7.57
PFHxS	60.7	3	4.14	NA	3	8	NA	3	11.7	NA	0	NA	NA
PFHpS	2.32	3	4.15	109.13	3	8.02	123.33	3	11.8	121.43	3	117.96	6.53
PFOS	208	3	4.14	NA	3	8	NA	3	11.7	NA	0	NA	NA
PFNS	NA	0	4.15	113.86	0	8.02	123.16	0	11.8	119.18	3	118.73	3.93
PFDS	NA	0	4.14	94.55	0	8	102.57	0	11.7	106.91	3	101.34	6.19
PFDoS	NA	0	4.15	92.95	0	8.02	94.4	0	11.8	101.71	3	96.35	4.87
4:2 FTS	NA	0	16.5	101.75	0	32	122.34	0	47	121.88	3	115.32	10.2
6:2 FTS	NA	0	14.93*	NA	0	28.9	129.49	0	42.4	120.72	2	125.11	4.95
8:2 FTS	NA	0	16.5	104.64	0	32	127.91	0	47	123.7	3	118.75	10.44
PFOSA	0.532	3	4.14	141.32	3	8	125.43	3	11.7	126.05	3	130.93	6.87
N-MeFOSA	NA	0	4.76	94.97	0	9.2	118.48	0	13.5	112.56	3	108.67	11.26
N-EtFOSA	NA	0	10.4	93.08	0	20	113.43	0	29.4	111.14	3	105.89	10.53
N-MeFOSAA	NA	0	4.14	117.99	0	8	164.31	0	11.7	157.35	3	146.55	17.05
N-EtFOSAA	NA	0	4.14	109.56	0	8	139.88	0	11.7	125.9	3	125.11	12.13
N-MeFOSE	NA	0	41.4	101.6	0	80	119.46	0	117	117.05	3	112.7	8.6
N-EtFOSE	NA	0	31	103.63	0	60	124.21	0	88.1	123.29	3	117.04	9.93
HFPO-DA	NA	0	15.7	108.14	0	30.4	120.55	0	44.7	106.43	3	111.71	6.9
ADONA	NA	0	16.6	91.92	0	32.1	112.32	0	47.1	117.64	3	107.29	12.65
PFMPA	NA	0	8.28	105.4	0	16	124.26	0	23.5	115.34	3	115	8.2
PFMBA	NA	0	4.14	98.37	0	8	119.53	0	11.7	113.91	3	110.6	9.91
NFDHA	NA	0	8.28	124.96	0	16	110.9	0	23.5	105.5	3	113.79	8.83
9Cl-PF3ONS	NA	0	16.6	94.98	0	32.1	113.85	0	47.1	123.13	3	110.65	12.97
11Cl-PF3OUdS	NA	0	16.6	89.88	0	32.1	94.14	0	47.1	109.19	3	97.74	10.38
PFEESA	NA	0	4.14	100.61	0	8	101.1	0	11.7	116.74	3	106.15	8.64
3:3 FTCA	NA	0	16.5	103.85	0	32	120.65	0	47	113.67	3	112.72	7.49
5:3 FTCA	NA	0	104	101.57	0	200	115.19	0	294	108.02	3	108.26	6.29
7:3 FTCA	NA	0	104	105.21	0	200	113.18	0	294	114.27	3	110.89	4.46

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-14 LCH C & D Landfill Leachate Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	82.4	3	69	NA	3	142	100.64	3	207	106.88	2	103.76	4.25
PFPeA	437	3	34.5	NA	3	70.9	NA	3	103	NA	0	NA	NA
PFHxA	253	3	17.3	NA	3	35.5	NA	3	51.7	NA	0	NA	NA
PFHpA	74.9	3	17.3	NA	3	35.5	NA	3	51.7	NA	0	NA	NA
PFOA	125	3	17.3	NA	3	35.5	NA	3	51.7	NA	0	NA	NA
PFNA	6.9	3	17.3	98.79	3	35.5	98.1	3	51.7	108.98	3	101.96	5.98
PFDA	2.19	3	17.3	109.4	3	35.5	112.36	3	51.7	110.43	3	110.73	1.36
PFUnA	NA	0	17.3	99.34	0	35.5	111.2	0	51.7	114.97	3	108.5	7.52
PFDoA	NA	0	17.3	100.6	0	35.5	110.11	0	51.7	117.68	3	109.47	7.82
PFTrDA	NA	0	17.3	106.21	0	35.5	102.97	0	51.7	110.31	3	106.5	3.45
PFTeDA	NA	0	17.3	118.37	0	35.5	109.12	0	51.7	106.98	3	111.49	5.43
PFBS	45.6	3	17.3	NA	3	35.5	NA	3	51.7	117.91	1	117.91	NA
PFPeS	3.8	3	17.3	100.7	3	35.5	111.39	3	51.9	113.73	3	108.61	6.4
PFHxS	33.1	3	17.3	NA	3	35.5	103.56	3	51.7	115.54	2	109.55	7.74
PFHpS	1.37	3	17.3	104.72	3	35.5	104.35	3	51.8	113.6	3	107.56	4.87
PFOS	42.9	3	17.3	NA	3	35.5	NA	3	51.7	123.36	1	123.36	NA
PFNS	NA	0	17.3	90.25	0	35.5	105.23	0	51.8	109.08	3	101.52	9.8
PFDS	NA	0	17.3	77.62	0	35.5	85.32	0	51.7	86	3	82.98	5.61
PFDoS	NA	0	17.3	58.64	0	35.5	54.65	0	51.8	59.56	3	57.62	4.53
4:2 FTS	NA	0	69	108.33	0	142	107.98	0	207	108.69	3	108.33	0.32
6:2 FTS	29.5	3	62.2	105.39	3	128	101.85	3	187	106.02	3	104.42	2.15
8:2 FTS	NA	0	69	124.37	0	142	115.88	0	207	126.07	3	122.1	4.47
PFOSA	4.13	3	17.3	178.31	3	35.5	118.61	3	51.7	117.03	3	137.98	25.32
N-MeFOSA	NA	0	19.8	109.33	0	40.7	109.37	0	59.5	111.14	3	109.95	0.94
N-EtFOSA	NA	0	43.1	111.92	0	88.6	109.16	0	130	113.91	3	111.66	2.14
N-MeFOSAA	2.54	2	17.3	220.51	2	35.5	170.99	2	51.7	151.93	3	181.14	19.54
N-EtFOSAA	NA	0	17.3	155.67	0	35.5	134.96	0	51.7	132.2	3	140.94	9.1
N-MeFOSE	NA	0	173	108.74	0	355	106.05	0	517	112.2	3	109	2.83
N-EtFOSE	NA	0	130	109.34	0	266	110.94	0	387	116.16	3	112.15	3.18
HFPO-DA	NA	0	65.5	107.6	0	135	108.58	0	196	108.35	3	108.18	0.47
ADONA	NA	0	69.2	124.48	0	142	128.47	0	207	130.53	3	127.83	2.41
PFMPA	NA	0	34.5	163.81	0	70.9	187.36	0	103	197.46	3	182.88	9.44
PFMBA	NA	0	17.3	112.7	0	35.5	124.86	0	51.7	126.07	3	121.21	6.1
NFDHA	NA	0	34.5	50.48	0	70.9	37.7	0	103	36.86	3	41.68	18.31
9Cl-PF3ONS	NA	0	69.1	121.89	0	142	128.28	0	207	126.27	3	125.48	2.6
11Cl-PF3OUdS	NA	0	69.1	92.21	0	142	107.16	0	207	106.69	3	102.02	8.33
PFEESA	NA	0	17.3	129	0	35.5	135.21	0	51.7	139.42	3	134.54	3.89
3:3 FTCA	NA	0	69	176.81	0	142	200.98	0	207	210.24	3	196.01	8.81
5:3 FTCA	102	3	431	207.72	3	886	219.25	3	1300	218.96	3	215.31	3.05
7:3 FTCA	NA	0	431	133.1	0	886	167.59	0	1300	166.76	3	155.81	12.63

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-15 LCH MSW Landfill Leachate Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	1610	3	65.3	NA	3	128	NA	3	214	NA	0	NA	NA
PFPeA	1250	3	32.7	NA	3	63.9	NA	3	107	NA	0	NA	NA
PFHxA	3190	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
PFHpA	627	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
PFOA	1340	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
PFNA	82.2	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
PFDA	119	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
PFUnA	12.6	3	16.4	94.76	3	31.9	103.31	3	53.3	105.94	3	101.34	5.77
PFDoA	31	3	16.4	NA	3	31.9	97.83	3	53.3	129.86	2	113.85	19.89
PFTrDA	7.07	3	16.4	99.27	3	31.9	99.9	3	53.3	108.96	3	102.71	5.28
PFTeDA	15.8	3	16.4	104.22	3	31.9	114.91	3	53.3	111.96	3	110.36	5
PFBS	1490	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
PFPeS	22.4	3	16.4	NA	3	32	114.76	3	53.5	124.31	2	119.53	5.65
PFHxS	780	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
PFHpS	4.29	3	16.4	109.81	3	32	106.26	3	53.5	118.62	3	111.57	5.7
PFOS	217	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
PFNS	NA	0	16.4	96.54	0	32	92.54	0	53.4	87.55	3	92.21	4.88
PFDS	4.96	3	16.4	69.26	3	31.9	55.96	3	53.3	63.89	3	63.03	10.62
PFDoS	NA	0	16.4	39.87	0	32	22.91	0	53.4	28.97	3	30.58	28.1
4:2 FTS	NA	0	65.3	107.75	0	128	108.28	0	214	110.07	3	108.7	1.12
6:2 FTS	183	3	58.9	NA	3	115	NA	3	192	105.82	1	105.82	NA
8:2 FTS	60.1	3	65.3	122.76	3	128	123.85	3	214	132.49	3	126.36	4.22
PFOSA	34.1	3	16.4	NA	3	31.9	NA	3	53.3	116.16	1	116.16	NA
N-MeFOSA	12.3	3	18.8	110.08	3	36.8	111.33	3	61.4	119.4	3	113.6	4.45
N-EtFOSA	23.5	3	40.8	103.15	3	79.9	115.23	3	133	116.52	3	111.63	6.6
N-MeFOSAA	311	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
N-EtFOSAA	250	3	16.4	NA	3	31.9	NA	3	53.3	NA	0	NA	NA
N-MeFOSE	2680	3	164	NA	3	319	NA	3	533	NA	0	NA	NA
N-EtFOSE	2150	3	122	NA	3	240	NA	3	400	NA	0	NA	NA
HFPO-DA	11.1	3	62.1	106.9	3	122	110.14	3	203	108.75	3	108.6	1.49
ADONA	NA	0	65.5	184.25	0	128	191.14	0	214	196.71	3	190.7	3.27
PFMPA	0.796	1	32.7	64.16	1	63.9	133.16	1	107	134.91	3	110.74	36.44
PFMBA	1.84	3	16.4	161.87	3	31.9	166.11	3	53.3	164.31	3	164.1	1.3
NFDHA	6.43	1	32.7	17.3	1	63.9	28.54	1	107	35.37	3	27.07	33.7
9Cl-PF3ONS	NA	0	65.4	192.02	0	128	198.03	0	214	200.22	3	196.76	2.16
11Cl-PF3OUdS	NA	0	65.4	141.74	0	128	131.71	0	214	139.67	3	137.71	3.85
PFEESA	8.94	3	16.4	196.16	3	31.9	258.84	3	53.3	274.32	3	243.11	17.02
3:3 FTCA	38.9	3	65.3	29.12	3	128	93.37	3	214	96.07	3	72.85	52.02
5:3 FTCA	8230	3	408	NA	3	799	NA	3	1330	NA	0	NA	NA
7:3 FTCA	1850	3	408	NA	3	799	NA	3	1330	NA	0	NA	NA

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-16 LCH MSW Incineration Ash Landfill Leachate Matrix Spike Recoveries (ng/L)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	51.9	3	63.3	102.77	3	131	109.03	3	191	107.87	3	106.56	3.12
PFPeA	54.1	3	31.7	NA	3	65.3	117.4	3	95.5	112.68	2	115.04	2.9
PFHxA	53.8	3	15.9	NA	3	32.7	NA	3	47.7	NA	0	NA	NA
PFHpA	4.65	3	15.9	105.38	3	32.7	110.12	3	47.7	109.15	3	108.22	2.32
PFOA	5.55	3	15.9	104.3	3	32.7	107.14	3	47.7	107.86	3	106.43	1.77
PFNA	NA	0	15.9	100.18	0	32.7	105.92	0	47.7	110.47	3	105.52	4.89
PFDA	NA	0	15.9	107.86	0	32.7	113.26	0	47.7	112.44	3	111.18	2.62
PFUnA	NA	0	15.9	101.72	0	32.7	110.43	0	47.7	115.21	3	109.12	6.27
PFDoA	NA	0	15.9	106.1	0	32.7	109.46	0	47.7	111.89	3	109.15	2.67
PFTrDA	NA	0	15.9	114.38	0	32.7	113.58	0	47.7	114.02	3	113.99	0.35
PFTeDA	NA	0	15.9	120.07	0	32.7	112.94	0	47.7	111.69	3	114.9	3.93
PFBS	52.9	3	15.9	NA	3	32.7	NA	3	47.7	NA	0	NA	NA
PFPeS	NA	0	15.9	105.96	0	32.8	118.69	0	47.8	116.03	3	113.56	5.92
PFHxS	3.34	3	15.9	116.92	3	32.7	118.54	3	47.7	116.98	3	117.48	0.78
PFHpS	NA	0	15.9	101.07	0	32.7	118.07	0	47.8	119.05	3	112.73	8.97
PFOS	1.43	1	15.9	107.1	1	32.7	116.85	1	47.7	118.54	3	114.16	5.41
PFNS	NA	0	15.9	104.12	0	32.7	108.47	0	47.8	104.06	3	105.55	2.4
PFDS	NA	0	15.9	90.33	0	32.7	94.89	0	47.7	91.02	3	92.08	2.67
PFDoS	NA	0	15.9	75.72	0	32.7	76.09	0	47.8	77.6	3	76.47	1.3
4:2 FTS	NA	0	63.3	109.3	0	131	110.18	0	191	112.09	3	110.53	1.29
6:2 FTS	NA	0	57.1	112.18	0	118	110.11	0	172	111.37	3	111.22	0.94
8:2 FTS	NA	0	63.3	115.17	0	131	114.29	0	191	122.53	3	117.33	3.86
PFOSA	NA	0	15.9	102.22	0	32.7	105.69	0	47.7	106.4	3	104.77	2.13
N-MeFOSA	NA	0	18.2	94.28	0	37.6	111	0	54.9	110.78	3	105.35	9.1
N-EtFOSA	NA	0	39.6	91.92	0	81.7	104.81	0	119	109.21	3	101.98	8.81
N-MeFOSAA	2.38	1	15.9	87.39	1	32.7	100.9	1	47.7	110.25	3	99.51	11.55
N-EtFOSAA	1.48	2	15.9	87.72	2	32.7	111.03	2	47.7	119.78	3	106.17	15.61
N-MeFOSE	NA	0	159	101.75	0	327	111.04	0	477	112.83	3	108.54	5.48
N-EtFOSE	NA	0	119	105.37	0	245	116.38	0	357	115.66	3	112.47	5.47
HFPO-DA	NA	0	60.2	116.33	0	124	118.41	0	181	109.3	3	114.68	4.16
ADONA	NA	0	63.5	113.21	0	131	110.66	0	191	103.6	3	109.16	4.56
PFMPA	NA	0	31.7	111.03	0	65.3	113.95	0	95.5	112.12	3	112.37	1.32
PFMBA	NA	0	15.9	109.93	0	32.7	112.65	0	47.7	110.03	3	110.87	1.39
NFDHA	NA	0	31.7	95.57	0	65.3	90.91	0	95.5	102.96	3	96.48	6.3
9Cl-PF3ONS	NA	0	63.5	99.21	0	131	101.97	0	191	92.47	3	97.88	4.99
11Cl-PF3OUdS	NA	0	63.4	89.57	0	131	93.59	0	191	82.93	3	88.7	6.07
PFEESA	NA	0	15.9	105.31	0	32.7	111.27	0	47.7	111.9	3	109.49	3.32
3:3 FTCA	NA	0	63.3	111.46	0	131	113.43	0	191	116.09	3	113.66	2.04
5:3 FTCA	NA	0	396	109.67	0	817	117.44	0	1190	115.06	3	114.06	3.49
7:3 FTCA	NA	0	396	85.99	0	817	106.46	0	1190	108.44	3	100.3	12.4

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-17 Soil 2014-107 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration (1)	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n (2)	Mean Spike Concentration	% Recovery	n (2)	Mean Spike Concentration	% Recovery	n (2)	Mean Spike Concentration	% Recovery	n (3)	% Mean Recovery	RSD (4)
PFBA	NA	0	2.58	106.85	0	3.87	107.07	0	6.44	106.89	3	106.93	0.11
PFPeA	0.531	3	1.29	101.73	3	1.93	100.98	3	3.22	108.35	3	103.69	3.91
PFHxA	0.454	3	0.644	99.79	3	0.966	96.86	3	1.61	102.24	3	99.63	2.7
PFHpA	0.349	3	0.644	99.95	3	0.966	100.1	3	1.61	105.42	3	101.83	3.06
PFOA	1.89	3	0.644	NA	3	0.966	NA	3	1.61	NA	0	NA	NA
PFNA	0.61	3	0.644	100.98	3	0.966	99.38	3	1.61	102.09	3	100.82	1.35
PFDA	2.38	3	0.644	NA	3	0.966	NA	3	1.61	NA	0	NA	NA
PFUnA	0.244	3	0.644	104.87	3	0.966	98.9	3	1.61	103.46	3	102.41	3.05
PFDoA	0.648	3	0.644	NA	3	0.966	90.57	3	1.61	103.44	2	97	9.38
PFTrDA	0.079	3	0.644	110.14	3	0.966	114.28	3	1.61	109.79	3	111.41	2.24
PFTeDA	0.182	3	0.644	93.43	3	0.966	103.97	3	1.61	104.22	3	100.54	6.13
PFBS	NA	0	0.644	104.19	0	0.966	105.93	0	1.61	104.55	3	104.89	0.88
PFPeS	NA	0	0.646	105.52	0	0.968	105.95	0	1.62	104.95	3	105.47	0.48
PFHxS	0.178	3	0.644	107.61	3	0.966	109.24	3	1.61	114.84	3	110.57	3.43
PFHpS	NA	0	0.646	98.66	0	0.968	102.72	0	1.61	102.69	3	101.36	2.31
PFOS	1.1	3	0.644	NA	3	0.966	NA	3	1.61	106.83	1	106.83	NA
PFNS	NA	0	0.646	109.6	0	0.967	108.26	0	1.61	104.76	3	107.54	2.32
PFDS	0.181	3	0.644	91.87	3	0.966	101.69	3	1.61	100.58	3	98.05	5.48
PFDoS	NA	0	0.645	82.64	0	0.967	89.25	0	1.61	88.2	3	86.69	4.1
4:2 FTS	NA	0	2.58	101.42	0	3.87	108.36	0	6.44	102.23	3	104	3.65
6:2 FTS	3.49	2	2.32*	NA	2	3.49	NA	2	5.81	48.56	1	48.56	NA
8:2 FTS	NA	0	2.58	114.86	0	3.87	123.11	0	6.44	121.08	3	119.68	3.59
PFOSA	NA	0	0.644	102.02	0	0.966	109.69	0	1.61	108.7	3	106.8	3.91
N-MeFOSA	NA	0	0.741	106.34	0	1.11	107.81	0	1.85	107.03	3	107.06	0.68
N-EtFOSA	NA	0	1.61	112.63	0	2.42	115.31	0	4.03	114.99	3	114.31	1.28
N-MeFOSAA	NA	0	0.644	98.03	0	0.966	105.66	0	1.61	101.86	3	101.85	3.74
N-EtFOSAA	0.0617	3	0.644	108.9	3	0.966	106.07	3	1.61	111.7	3	108.89	2.58
N-MeFOSE	NA	0	6.44	104.3	0	9.66	108.66	0	16.1	108.9	3	107.29	2.42
N-EtFOSE	NA	0	4.83	109.59	0	7.24	112.61	0	12.1	112.95	3	111.72	1.65
HFPO-DA	NA	0	2.45	99.05	0	3.67	115.89	0	6.12	107.36	3	107.43	7.84
ADONA	NA	0	2.58	96.12	0	3.88	109.37	0	6.46	97.62	3	101.04	7.18
PFMPA	NA	0	1.29	110.85	0	1.93	107.94	0	3.22	103.11	3	107.3	3.65
PFMBA	NA	0	0.644	115.37	0	0.966	112.45	0	1.61	90.68	3	106.17	12.71
NFDHA	NA	0	1.29	82.71	0	1.93	83.42	0	3.22	125.78	3	97.3	25.34
9Cl-PF3ONS	NA	0	2.58	102.58	0	3.87	118.43	0	6.45	102.06	3	107.69	8.64
11Cl-PF3OUdS	NA	0	2.58	101.29	0	3.87	114.56	0	6.45	101.29	3	105.71	7.25
PFEESA	NA	0	0.644	104.71	0	0.966	99.97	0	1.61	100.62	3	101.77	2.53
3:3 FTCA	NA	0	2.58	111.5	0	3.87	107.67	0	6.44	102.95	3	107.37	3.99
5:3 FTCA	NA	0	16.1	106.63	0	24.2	99.59	0	40.3	104.56	3	103.59	3.49
7:3 FTCA	NA	0	16.1	103.52	0	24.2	100.55	0	40.3	108.69	3	104.26	3.95

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a "No") and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a "No") and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-18 Soil 2016-106 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.65	105.65	0	3.97	106.96	0	6.62	105.79	3	106.13	0.68
PFPeA	0.0347	3	1.33	103.42	3	1.99	107.99	3	3.31	108.91	3	106.78	2.75
PFHxA	0.0387	3	0.663	99	3	0.993	101.68	3	1.66	101.68	3	100.79	1.54
PFHpA	NA	0	0.663	105.33	0	0.993	100.33	0	1.66	107.24	3	104.3	3.42
PFOA	0.0503	3	0.663	97.79	3	0.993	101.34	3	1.66	101.79	3	100.31	2.18
PFNA	NA	0	0.663	104.01	0	0.993	104.39	0	1.66	105.83	3	104.75	0.92
PFDA	0.036	1	0.663	99.3	1	0.993	101.78	1	1.66	106.48	3	102.52	3.56
PFUnA	NA	0	0.663	102.92	0	0.993	108.42	0	1.66	104.42	3	105.26	2.7
PFDoA	NA	0	0.663	101.21	0	0.993	107.76	0	1.66	116.27	3	108.41	6.96
PFTrDA	NA	0	0.663	105.78	0	0.993	113.45	0	1.66	113.67	3	110.97	4.05
PFTeDA	NA	0	0.663	100.4	0	0.993	104.74	0	1.66	106.43	3	103.86	2.99
PFBS	NA	0	0.663	102.16	0	0.993	107.75	0	1.66	109.65	3	106.52	3.65
PFPeS	NA	0	0.665	102.71	0	0.996	102.74	0	1.66	103.83	3	103.09	0.62
PFHxS	NA	0	0.663	109.4	0	0.993	111.11	0	1.66	110.86	3	110.46	0.84
PFHpS	NA	0	0.665	97.14	0	0.995	97.96	0	1.66	99.79	3	98.3	1.38
PFOS	0.123	3	0.663	106.38	3	0.993	112.83	3	1.66	109.68	3	109.63	2.94
PFNS	NA	0	0.664	92.43	0	0.995	95.7	0	1.66	101.62	3	96.58	4.82
PFDS	NA	0	0.663	101.51	0	0.993	102.57	0	1.66	100.4	3	101.49	1.07
PFDoS	NA	0	0.664	91.02	0	0.994	92.14	0	1.66	94.36	3	92.51	1.84
4:2 FTS	NA	0	2.65	100.13	0	3.97	103.68	0	6.62	104.09	3	102.63	2.12
6:2 FTS	NA	0	2.39*	NA	0	3.58	103.35	0	5.97	117.98	2	110.66	9.35
8:2 FTS	NA	0	2.65	113.31	0	3.97	121.64	0	6.62	122.51	3	119.15	4.26
PFOSA	NA	0	0.663	102.46	0	0.993	108.76	0	1.66	106.04	3	105.75	2.99
N-MeFOSA	NA	0	0.763	111.37	0	1.14	111.95	0	1.91	106.82	3	110.04	2.55
N-EtFOSA	0.094	3	1.66	111.62	3	2.48	111.27	3	4.14	112.78	3	111.89	0.7
N-MeFOSAA	NA	0	0.663	97.43	0	0.993	99.64	0	1.66	102.82	3	99.96	2.71
N-EtFOSAA	NA	0	0.663	107.73	0	0.993	113.81	0	1.66	114.07	3	111.87	3.21
N-MeFOSE	NA	0	6.63	104.32	0	9.93	109.77	0	16.6	108.04	3	107.38	2.59
N-EtFOSE	NA	0	4.97	107.24	0	7.44	109.68	0	12.4	114.25	3	110.39	3.22
HFPO-DA	NA	0	2.52	112.96	0	3.77	105.91	0	6.29	103.49	3	107.45	4.58
ADONA	NA	0	2.66	123.47	0	3.98	102.09	0	6.64	94.87	3	106.81	13.93
PFMPA	NA	0	1.33	107.29	0	1.99	108.4	0	3.31	102.82	3	106.17	2.78
PFMBA	NA	0	0.663	111.41	0	0.993	111.79	0	1.66	103.23	3	108.81	4.45
NFDHA	NA	0	1.33	71.83	0	1.99	113.43	0	3.31	100.87	3	95.38	22.37
9Cl-PF3ONS	NA	0	2.66	128.74	0	3.98	109.04	0	6.63	97.18	3	111.65	14.28
11Cl-PF3OUdS	NA	0	2.66	124.97	0	3.98	106.78	0	6.63	98.08	3	109.95	12.48
PFEESA	NA	0	0.663	98.84	0	0.993	99.7	0	1.66	96.78	3	98.44	1.52
3:3 FTCA	NA	0	2.65	103.01	0	3.97	105.88	0	6.62	100.26	3	103.05	2.73
5:3 FTCA	NA	0	16.6	100.19	0	24.8	93.03	0	41.4	101.21	3	98.15	4.54
7:3 FTCA	NA	0	16.6	100.19	0	24.8	99.05	0	41.4	104.91	3	101.39	3.06

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

TableD-19 Soil 2017-111 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.62	104.07	0	3.94	108.55	0	6.57	104.41	3	105.68	2.36
PFPeA	0.027	1	1.31	112.7	1	1.97	111	1	3.28	109.63	3	111.11	1.38
PFHxA	0.0235	2	0.656	95.71	2	0.984	104.02	2	1.64	102.23	3	100.65	4.35
PFHpA	NA	0	0.656	105.34	0	0.984	104.38	0	1.64	105.49	3	105.07	0.57
PFOA	0.0565	2	0.656	99.67	2	0.984	101.68	2	1.64	97.98	3	99.78	1.86
PFNA	NA	0	0.656	105.85	0	0.984	109.8	0	1.64	103.46	3	106.37	3.01
PFDA	NA	0	0.656	102.24	0	0.984	112.17	0	1.64	106.5	3	106.97	4.66
PFUnA	NA	0	0.656	104.93	0	0.984	108.79	0	1.64	104.27	3	106	2.3
PFDoA	NA	0	0.656	105.69	0	0.984	105.37	0	1.64	106.71	3	105.92	0.66
PFTrDA	NA	0	0.656	115.04	0	0.984	116.23	0	1.64	108.74	3	113.34	3.55
PFTeDA	NA	0	0.656	102.65	0	0.984	108.79	0	1.64	105.49	3	105.64	2.91
PFBS	NA	0	0.656	107.02	0	0.984	109.45	0	1.64	106.3	3	107.59	1.53
PFPeS	NA	0	0.658	101.83	0	0.987	107.78	0	1.65	100	3	103.2	3.94
PFHxS	NA	0	0.656	104.88	0	0.984	114.88	0	1.64	108.74	3	109.5	4.61
PFHpS	NA	0	0.657	98.17	0	0.986	103.28	0	1.65	98.99	3	100.15	2.74
PFOS	NA	0	0.656	113.78	0	0.984	118.6	0	1.64	111.99	3	114.79	2.98
PFNS	NA	0	0.657	88.89	0	0.986	99.76	0	1.64	110.34	3	99.66	10.76
PFDS	NA	0	0.656	98.37	0	0.984	101.32	0	1.64	100.2	3	99.97	1.49
PFDoS	NA	0	0.657	76.7	0	0.985	86.12	0	1.64	82.35	3	81.73	5.8
4:2 FTS	NA	0	2.62	96.06	0	3.94	106.01	0	6.57	107.91	3	103.33	6.16
6:2 FTS	2.69	1	2.36*	NA	1	3.55	37.5	1	5.92	61.96	2	49.73	34.78
8:2 FTS	NA	0	2.62	121.35	0	3.94	127.01	0	6.57	121.4	3	123.25	2.64
PFOSA	NA	0	0.656	105.13	0	0.984	109.79	0	1.64	105.08	3	106.67	2.54
N-MeFOSA	NA	0	0.754	109.85	0	1.13	108.85	0	1.89	111.29	3	110	1.11
N-EtFOSA	0.0867	3	1.64	108.74	3	2.46	115.83	3	4.1	114.06	3	112.88	3.27
N-MeFOSAA	NA	0	0.656	101.53	0	0.984	97.63	0	1.64	103.66	3	100.94	3.03
N-EtFOSAA	NA	0	0.656	110.17	0	0.984	112.16	0	1.64	111.79	3	111.37	0.95
N-MeFOSE	NA	0	6.56	104.98	0	9.84	112.51	0	16.4	108.74	3	108.74	3.46
N-EtFOSE	NA	0	4.91	109.43	0	7.37	113.56	0	12.3	111.38	3	111.46	1.85
HFPO-DA	NA	0	2.49	109.9	0	3.74	119.7	0	6.24	100.75	3	110.12	8.61
ADONA	NA	0	2.63	112.8	0	3.95	119.98	0	6.59	91.91	3	108.23	13.48
PFMPA	NA	0	1.31	116.03	0	1.97	116.95	0	3.28	102.74	3	111.91	7.1
PFMBA	NA	0	0.656	122.27	0	0.984	116.56	0	1.64	105.69	3	114.84	7.33
NFDHA	NA	0	1.31	97.2	0	1.97	88.15	0	3.28	99.09	3	94.81	6.16
9Cl-PF3ONS	NA	0	2.63	120.53	0	3.94	128.09	0	6.58	96.26	3	114.96	14.47
11Cl-PF3OUdS	NA	0	2.63	114.2	0	3.94	121.27	0	6.58	93.93	3	109.8	12.93
PFEESA	NA	0	0.656	99.95	0	0.984	94.93	0	1.64	96.75	3	97.21	2.61
3:3 FTCA	NA	0	2.62	106.1	0	3.94	112.86	0	6.57	101.17	3	106.71	5.5
5:3 FTCA	NA	0	16.4	97.56	0	24.6	92.44	0	41	96.83	3	95.61	2.9
7:3 FTCA	NA	0	16.4	97.36	0	24.6	99.87	0	41	102.19	3	99.81	2.42

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

TableD-20 Soil 2018-105 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.58	104.27	0	3.88	108.24	0	6.44	105.02	3	105.84	1.99
PFPeA	0.0255	2	1.29	115.34	2	1.94	113.96	2	3.23	109.75	3	113.02	2.57
PFHxA	0.025	1	0.644	97.15	1	0.971	106.25	1	1.61	101.76	3	101.72	4.47
PFHpA	NA	0	0.644	101.14	0	0.971	105.64	0	1.61	100.63	3	102.47	2.69
PFOA	NA	0	0.644	103.16	0	0.971	106.42	0	1.61	102.48	3	104.02	2.02
PFNA	NA	0	0.644	101.55	0	0.971	110.54	0	1.61	105.38	3	105.82	4.26
PFDA	NA	0	0.644	106.62	0	0.971	110.54	0	1.61	105.59	3	107.58	2.43
PFUnA	NA	0	0.644	105.28	0	0.971	108.82	0	1.61	102.28	3	105.46	3.1
PFDoA	NA	0	0.644	96.96	0	0.971	108.13	0	1.61	102.69	3	102.59	5.45
PFTrDA	NA	0	0.644	108.43	0	0.971	113.63	0	1.61	105.58	3	109.21	3.73
PFTeDA	NA	0	0.644	106.93	0	0.971	110.2	0	1.61	103.93	3	107.02	2.93
PFBS	NA	0	0.644	108.43	0	0.971	106.21	0	1.61	104.14	3	106.26	2.02
PFPeS	NA	0	0.646	106.65	0	0.974	105.41	0	1.61	99.79	3	103.95	3.51
PFHxS	0.021	1	0.644	106.88	1	0.971	111.47	1	1.61	106.34	3	108.23	2.6
PFHpS	NA	0	0.646	99.17	0	0.973	102.3	0	1.61	96.69	3	99.39	2.83
PFOS	NA	0	0.644	108.22	0	0.971	113.28	0	1.61	107.44	3	109.65	2.89
PFNS	NA	0	0.646	94.11	0	0.973	102.61	0	1.61	99.58	3	98.77	4.36
PFDS	NA	0	0.644	101.81	0	0.971	106.42	0	1.61	98.97	3	102.4	3.67
PFDoS	NA	0	0.645	100.1	0	0.972	96.44	0	1.61	93.18	3	96.57	3.59
4:2 FTS	NA	0	2.58	105.05	0	3.88	110.38	0	6.44	107.66	3	107.7	2.48
6:2 FTS	5.66	1	2.32*	NA	1	3.5	NA	1	5.81	11.24	1	11.24	NA
8:2 FTS	NA	0	2.58	131.95	0	3.88	129.78	0	6.44	122.98	3	128.24	3.65
PFOSA	NA	0	0.644	107.03	0	0.971	110.54	0	1.61	105.58	3	107.72	2.37
N-MeFOSA	NA	0	0.741	109.59	0	1.12	111.05	0	1.85	108.63	3	109.76	1.11
N-EtFOSA	NA	0	1.61	109.52	0	2.43	117.03	0	4.03	111.91	3	112.82	3.4
N-MeFOSAA	NA	0	0.644	97.3	0	0.971	107.45	0	1.61	105.99	3	103.58	5.3
N-EtFOSAA	NA	0	0.644	114.53	0	0.971	106.77	0	1.61	111.16	3	110.82	3.51
N-MeFOSE	NA	0	6.44	106.78	0	9.71	112.25	0	16.1	108.27	3	109.1	2.6
N-EtFOSE	NA	0	4.83	110.08	0	7.28	113.23	0	12.1	111.57	3	111.63	1.41
HFPO-DA	NA	0	2.45	101.5	0	3.69	110.86	0	6.13	105.82	3	106.06	4.42
ADONA	NA	0	2.59	95.49	0	3.89	116.09	0	6.46	92.68	3	101.42	12.61
PFMPA	NA	0	1.29	114.99	0	1.94	116.8	0	3.23	104.34	3	112.04	6.01
PFMBA	NA	0	0.644	121.47	0	0.971	122.21	0	1.61	103.51	3	115.73	9.15
NFDHA	NA	0	1.29	116.54	0	1.94	99.29	0	3.23	110.98	3	108.94	8.08
9Cl-PF3ONS	NA	0	2.58	101.68	0	3.89	130.43	0	6.46	98.81	3	110.31	15.85
11Cl-PF3OUdS	NA	0	2.58	102.84	0	3.89	127.26	0	6.46	97.47	3	109.19	14.54
PFEESA	NA	0	0.644	103.93	0	0.971	104.25	0	1.61	100.83	3	103	1.84
3:3 FTCA	NA	0	2.58	109.19	0	3.88	112.36	0	6.44	100.46	3	107.34	5.74
5:3 FTCA	NA	0	16.1	96.48	0	24.3	93.82	0	40.3	91.39	3	93.9	2.71
7:3 FTCA	NA	0	16.1	97.31	0	24.3	99.59	0	40.3	98.26	3	98.39	1.16

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

TableD-21 Soil 2018-116 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.66	106.63	0	4	108.09	0	6.67	105.9	3	106.87	1.04
PFPeA	0.028	1	1.33	100.16	1	2	101.26	1	3.34	111.55	3	104.32	6.03
PFHxA	0.0307	3	0.666	101.45	3	0.999	105.08	3	1.67	101.36	3	102.63	2.07
PFHpA	0.04	1	0.666	97.25	1	0.999	101.81	1	1.67	99.2	3	99.42	2.3
PFOA	0.0843	3	0.666	98.8	3	0.999	102.04	3	1.67	100.54	3	100.46	1.61
PFNA	NA	0	0.666	109.32	0	0.999	106.14	0	1.67	104.81	3	106.76	2.17
PFDA	NA	0	0.666	106.25	0	0.999	110.15	0	1.67	107.01	3	107.8	1.92
PFUnA	NA	0	0.666	91.73	0	0.999	106.15	0	1.67	104.2	3	100.69	7.77
PFDoA	NA	0	0.666	108.95	0	0.999	110.15	0	1.67	101.01	3	106.7	4.65
PFTrDA	NA	0	0.666	113.26	0	0.999	112.82	0	1.67	107.41	3	111.16	2.93
PFTeDA	NA	0	0.666	105.61	0	0.999	107.81	0	1.67	105.2	3	106.21	1.32
PFBS	0.0213	3	0.666	99.21	3	0.999	109.02	3	1.67	107.52	3	105.25	5.02
PFPeS	NA	0	0.668	101.84	0	1	103.84	0	1.67	110.76	3	105.48	4.44
PFHxS	0.0287	3	0.666	107.41	3	0.999	113.28	3	1.67	111.89	3	110.86	2.77
PFHpS	NA	0	0.668	98.6	0	1	102.7	0	1.67	99.8	3	100.37	2.1
PFOS	0.263	3	0.666	107.79	3	0.999	112.21	3	1.67	106.05	3	108.69	2.92
PFNS	NA	0	0.667	108.69	0	0.999	100.29	0	1.67	88.02	3	99	10.5
PFDS	NA	0	0.666	103.65	0	0.999	105.14	0	1.67	95.61	3	101.47	5.05
PFDoS	NA	0	0.667	94.44	0	0.999	98.83	0	1.67	95.21	3	96.16	2.44
4:2 FTS	NA	0	2.66	99.75	0	4	105.5	0	6.67	107.7	3	104.32	3.94
6:2 FTS	0.172	1	2.4	102.83	1	3.6	108.64	1	6.01	102.97	3	104.81	3.16
8:2 FTS	NA	0	2.66	118.63	0	4	122.27	0	6.67	121.84	3	120.91	1.64
PFOSA	NA	0	0.666	105.3	0	0.999	108.48	0	1.67	106.41	3	106.73	1.51
N-MeFOSA	NA	0	0.766	102.26	0	1.15	109.57	0	1.92	110.09	3	107.3	4.08
N-EtFOSA	NA	0	1.67	111.99	0	2.5	114.39	0	4.17	111.91	3	112.76	1.25
N-MeFOSAA	NA	0	0.666	100.46	0	0.999	110.15	0	1.67	107.8	3	106.14	4.76
N-EtFOSAA	NA	0	0.666	107.82	0	0.999	113.49	0	1.67	112.21	3	111.17	2.67
N-MeFOSE	NA	0	6.66	104.81	0	9.99	109.15	0	16.7	107	3	106.99	2.03
N-EtFOSE	NA	0	4.99	107.41	0	7.49	111.43	0	12.5	110.67	3	109.84	1.94
HFPO-DA	NA	0	2.53	99.58	0	3.8	87.79	0	6.34	113.65	3	100.34	12.9
ADONA	NA	0	2.67	107.82	0	4.01	97.34	0	6.69	108.16	3	104.44	5.89
PFMPA	NA	0	1.33	114.01	0	2	109.83	0	3.34	99.3	3	107.71	7.04
PFMBA	NA	0	0.666	117.84	0	0.999	112.14	0	1.67	98.8	3	109.59	8.91
NFDHA	NA	0	1.33	75.54	0	2	70.63	0	3.34	108.49	3	84.89	24.25
9Cl-PF3ONS	NA	0	2.67	116.47	0	4	107.66	0	6.68	115.4	3	113.18	4.25
11Cl-PF3OUdS	NA	0	2.67	112.72	0	4	105.09	0	6.68	118.15	3	111.99	5.86
PFEESA	NA	0	0.666	104.96	0	0.999	97.88	0	1.67	102	3	101.61	3.5
3:3 FTCA	NA	0	2.66	108.9	0	4	101.5	0	6.67	92.31	3	100.9	8.24
5:3 FTCA	NA	0	16.7	96.6	0	25	92.01	0	41.7	86.49	3	91.7	5.52
7:3 FTCA	NA	0	16.7	97.8	0	25	94.27	0	41.7	90.65	3	94.24	3.8

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-22 Soil 2019-107 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.62	108.65	0	3.94	105.75	0	6.55	106.56	3	106.99	1.4
PFPeA	0.0357	3	1.31	109.49	3	1.97	101.75	3	3.28	111.02	3	107.42	4.63
PFHxA	0.033	3	0.656	100.36	3	0.985	97.53	3	1.63	101.65	3	99.84	2.11
PFHpA	0.0325	2	0.656	96.88	2	0.985	97.86	2	1.63	99.84	3	98.19	1.54
PFOA	0.122	3	0.656	94.97	3	0.985	97.23	3	1.63	100.08	3	97.42	2.63
PFNA	0.092	2	0.656	98.27	2	0.985	98.58	2	1.63	103.14	3	100	2.72
PFDA	0.037	3	0.656	99.9	3	0.985	100.61	3	1.63	108.95	3	103.16	4.88
PFUnA	0.042	3	0.656	95.48	3	0.985	102.27	3	1.63	101.51	3	99.75	3.73
PFDoA	NA	0	0.656	103.51	0	0.985	105.13	0	1.63	103.06	3	103.9	1.05
PFTrDA	NA	0	0.656	114.54	0	0.985	117.72	0	1.63	113.46	3	115.24	1.92
PFTeDA	NA	0	0.656	104.68	0	0.985	102.78	0	1.63	105.31	3	104.25	1.26
PFBS	NA	0	0.656	102.65	0	0.985	107.01	0	1.63	108.16	3	105.94	2.74
PFPeS	NA	0	0.658	102.89	0	0.988	99.86	0	1.64	106.49	3	103.08	3.22
PFHxS	0.02	1	0.656	104.73	1	0.985	105.88	1	1.63	109.59	3	106.73	2.38
PFHpS	NA	0	0.657	95.18	0	0.988	95.22	0	1.64	101.82	3	97.41	3.92
PFOS	0.24	3	0.656	103.5	3	0.985	105.52	3	1.63	113.44	3	107.49	4.89
PFNS	NA	0	0.657	105.33	0	0.987	93.61	0	1.64	96.92	3	98.62	6.13
PFDS	NA	0	0.656	97.31	0	0.985	98.66	0	1.63	103.66	3	99.87	3.35
PFDoS	NA	0	0.657	92.59	0	0.987	88.9	0	1.64	93.5	3	91.66	2.66
4:2 FTS	NA	0	2.62	97.33	0	3.94	101.85	0	6.55	106.25	3	101.81	4.38
6:2 FTS	NA	0	2.37	111.12	0	3.55	147.45	0	5.9	105.31	3	121.3	18.83
8:2 FTS	NA	0	2.62	119.34	0	3.94	118.34	0	6.55	124.36	3	120.68	2.67
PFOSA	NA	0	0.656	103.1	0	0.985	102.98	0	1.63	105.91	3	104	1.6
N-MeFOSA	NA	0	0.754	109.46	0	1.13	110.01	0	1.88	107.96	3	109.14	0.97
N-EtFOSA	NA	0	1.64	108.13	0	2.47	109.86	0	4.1	113.34	3	110.44	2.4
N-MeFOSAA	NA	0	0.656	106.76	0	0.985	102.51	0	1.63	97.15	3	102.14	4.71
N-EtFOSAA	NA	0	0.656	109.25	0	0.985	104.75	0	1.63	109.18	3	107.73	2.39
N-MeFOSE	NA	0	6.56	104.07	0	9.85	105.31	0	16.3	109.58	3	106.32	2.72
N-EtFOSE	NA	0	4.92	106.3	0	7.39	107.18	0	12.3	112.2	3	108.56	2.93
HFPO-DA	NA	0	2.49	110.84	0	3.75	95.18	0	6.22	111.74	3	105.92	8.8
ADONA	NA	0	2.63	107.1	0	3.95	101.76	0	6.56	114.16	3	107.67	5.78
PFMPA	NA	0	1.31	116.28	0	1.97	110.82	0	3.28	100.3	3	109.14	7.44
PFMBA	NA	0	0.656	112.66	0	0.985	114.34	0	1.63	103.67	3	110.22	5.2
NFDHA	NA	0	1.31	120.87	0	1.97	90.23	0	3.28	99.08	3	103.39	15.25
9Cl-PF3ONS	NA	0	2.63	118.18	0	3.95	111.21	0	6.56	121.67	3	117.02	4.55
11Cl-PF3OUdS	NA	0	2.63	117.42	0	3.95	109.11	0	6.56	127.26	3	117.93	7.7
PFEESA	NA	0	0.656	104.52	0	0.985	99.73	0	1.63	100.2	3	101.49	2.6
3:3 FTCA	NA	0	2.62	109.8	0	3.94	106.16	0	6.55	93.9	3	103.28	8.07
5:3 FTCA	NA	0	16.4	94.31	0	24.7	91.35	0	41	89.75	3	91.8	2.52
7:3 FTCA	NA	0	16.4	97.36	0	24.7	94.59	0	41	96.99	3	96.31	1.56

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

TableD-23 Soil 2019-110 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.57	107.67	0	3.86	101.56	0	6.43	104.87	3	104.7	2.92
PFPeA	0.0337	3	1.29	106.74	3	1.93	97.74	3	3.22	108.69	3	104.39	5.6
PFHxA	0.042	3	0.642	105.35	3	0.965	97.55	3	1.61	96.98	3	99.96	4.68
PFHpA	0.0415	2	0.642	100.45	2	0.965	97.29	2	1.61	98.25	3	98.66	1.64
PFOA	0.065	3	0.642	98.91	3	0.965	94.2	3	1.61	100.93	3	98.01	3.53
PFNA	0.207	3	0.642	100.32	3	0.965	98.76	3	1.61	100.79	3	99.96	1.06
PFDA	0.044	3	0.642	105.14	3	0.965	100.1	3	1.61	107	3	104.08	3.43
PFUnA	0.203	3	0.642	101.98	3	0.965	97.51	3	1.61	103.77	3	101.09	3.19
PFDoA	NA	0	0.642	103.22	0	0.965	112.01	0	1.61	112.63	3	109.28	4.82
PFTrDA	0.0623	3	0.642	116.2	3	0.965	114.84	3	1.61	119.32	3	116.78	1.97
PFTeDA	NA	0	0.642	112.88	0	0.965	106.7	0	1.61	105.38	3	108.32	3.69
PFBS	NA	0	0.642	105.6	0	0.965	99.38	0	1.61	104.76	3	103.25	3.27
PFPeS	NA	0	0.644	103.83	0	0.967	98.11	0	1.61	102.89	3	101.61	3.02
PFHxS	NA	0	0.642	110.23	0	0.965	107.81	0	1.61	108.7	3	108.91	1.13
PFHpS	NA	0	0.643	97.05	0	0.967	94	0	1.61	98.96	3	96.67	2.59
PFOS	0.135	3	0.642	105.51	3	0.965	102.49	3	1.61	108.2	3	105.4	2.71
PFNS	NA	0	0.643	111.46	0	0.966	98.54	0	1.61	106.42	3	105.47	6.17
PFDS	NA	0	0.642	98.55	0	0.965	100.28	0	1.61	103.93	3	100.92	2.72
PFDoS	NA	0	0.643	83.61	0	0.966	86.99	0	1.61	88.41	3	86.34	2.85
4:2 FTS	NA	0	2.57	104.94	0	3.86	101.2	0	6.43	104.61	3	103.58	2
6:2 FTS	NA	0	2.32	109.08	0	3.48	103.65	0	5.8	107.52	3	106.75	2.62
8:2 FTS	NA	0	2.57	121.82	0	3.86	113.81	0	6.43	119.94	3	118.53	3.53
PFOSA	NA	0	0.642	105.82	0	0.965	103.77	0	1.61	106.83	3	105.47	1.48
N-MeFOSA	NA	0	0.738	113.86	0	1.11	106.31	0	1.85	106.67	3	108.94	3.91
N-EtFOSA	NA	0	1.61	114.74	0	2.41	112.3	0	4.02	114	3	113.68	1.1
N-MeFOSAA	NA	0	0.642	102.07	0	0.965	109.7	0	1.61	107.87	3	106.55	3.74
N-EtFOSAA	NA	0	0.642	105.05	0	0.965	101.21	0	1.61	105.8	3	104.02	2.36
N-MeFOSE	NA	0	6.42	108.52	0	9.65	107.81	0	16.1	109.11	3	108.48	0.6
N-EtFOSE	NA	0	4.81	109.77	0	7.23	105.49	0	12.1	112.12	3	109.13	3.08
HFPO-DA	NA	0	2.44	98.09	0	3.67	97.35	0	6.11	98.63	3	98.02	0.66
ADONA	NA	0	2.58	102.07	0	3.87	98.43	0	6.45	95.76	3	98.76	3.21
PFMPA	NA	0	1.29	114.78	0	1.93	103.28	0	3.22	98.34	3	105.47	8
PFMBA	NA	0	0.642	118.44	0	0.965	105.21	0	1.61	99.59	3	107.75	8.98
NFDHA	NA	0	1.29	103.03	0	1.93	94.65	0	3.22	84.78	3	94.15	9.7
9Cl-PF3ONS	NA	0	2.58	110.61	0	3.87	106.46	0	6.45	102.85	3	106.64	3.64
11Cl-PF3OUdS	NA	0	2.58	106.08	0	3.86	106.63	0	6.44	109.67	3	107.46	1.8
PFEESA	NA	0	0.642	106.75	0	0.965	96.61	0	1.61	98.55	3	100.64	5.35
3:3 FTCA	NA	0	2.57	111.44	0	3.86	99.23	0	6.43	93.73	3	101.47	8.93
5:3 FTCA	NA	0	16.1	108.3	0	24.1	96.41	0	40.2	95.77	3	100.16	7.04
7:3 FTCA	NA	0	16.1	107.89	0	24.1	96.41	0	40.2	98.17	3	100.82	6.13

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-24 Marine Sediment Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.56	97.66	0	3.83	104.61	0	6.41	108.49	3	103.58	5.3
PFPeA	0.023	1	1.28	99.77	1	1.92	108	1	3.2	111.87	3	106.55	5.8
PFHxA	NA	0	0.64	96.36	0	0.958	101.01	0	1.6	103.54	3	100.3	3.63
PFHpA	NA	0	0.64	94.59	0	0.958	101.22	0	1.6	102.92	3	99.57	4.42
PFOA	NA	0	0.64	95.06	0	0.958	104.52	0	1.6	106.25	3	101.94	5.91
PFNA	NA	0	0.64	96.2	0	0.958	103.93	0	1.6	109.79	3	103.31	6.6
PFDA	NA	0	0.64	100.99	0	0.958	102.68	0	1.6	108.54	3	104.07	3.81
PFUnA	NA	0	0.64	97.77	0	0.958	104.94	0	1.6	108.96	3	103.89	5.46
PFDoA	NA	0	0.64	100.78	0	0.958	105.08	0	1.6	112.5	3	106.12	5.59
PFTrDA	NA	0	0.64	103.81	0	0.958	111.3	0	1.6	112.92	3	109.34	4.45
PFTeDA	NA	0	0.64	97.55	0	0.958	107.13	0	1.6	105.62	3	103.44	4.98
PFBS	NA	0	0.64	98.18	0	0.958	101.88	0	1.6	108.96	3	103.01	5.32
PFPeS	NA	0	0.641	99.07	0	0.961	104.26	0	1.6	106.86	3	103.4	3.84
PFHxS	0.0195	2	0.64	107.99	2	0.958	118.31	2	1.6	120.66	3	115.65	5.83
PFHpS	NA	0	0.641	91.69	0	0.961	99.58	0	1.6	103.33	3	98.2	6.05
PFOS	NA	0	0.64	105.52	0	0.958	111.65	0	1.6	115.21	3	110.79	4.42
PFNS	NA	0	0.641	87.99	0	0.96	95.38	0	1.6	112.69	3	98.69	12.85
PFDS	NA	0	0.64	94.69	0	0.958	100.7	0	1.6	110.62	3	102	7.89
PFDoS	NA	0	0.641	93.04	0	0.96	96.7	0	1.6	100.42	3	96.72	3.82
4:2 FTS	NA	0	2.56	96.74	0	3.83	103.82	0	6.41	106.35	3	102.31	4.87
6:2 FTS	0.58	1	2.31	76.33	1	3.45	87.94	1	5.77	100.7	3	88.32	13.8
8:2 FTS	NA	0	2.56	110.16	0	3.83	120.34	0	6.41	126.75	3	119.08	7.03
PFOSA	NA	0	0.64	98.23	0	0.958	106.43	0	1.6	110.21	3	104.96	5.83
N-MeFOSA	NA	0	0.736	104.53	0	1.1	110.61	0	1.84	105.43	3	106.86	3.07
N-EtFOSA	NA	0	1.6	102.71	0	2.39	112.25	0	4	106.66	3	107.21	4.47
N-MeFOSAA	NA	0	0.64	90.89	0	0.958	94.74	0	1.6	107.5	3	97.71	8.9
N-EtFOSAA	NA	0	0.64	99.9	0	0.958	109.14	0	1.6	108.54	3	105.86	4.89
N-MeFOSE	NA	0	6.4	96	0	9.58	105.74	0	16	109.79	3	103.84	6.83
N-EtFOSE	NA	0	4.8	101.32	0	7.18	110.53	0	12	114.17	3	108.67	6.09
HFPO-DA	NA	0	2.43	90.26	0	3.64	113.98	0	6.09	106.96	3	103.73	11.75
ADONA	NA	0	2.57	93	0	3.84	108.75	0	6.42	104.05	3	101.93	7.93
PFMPA	NA	0	1.28	94.01	0	1.92	96.53	0	3.2	100	3	96.85	3.11
PFMBA	NA	0	0.64	97.66	0	0.958	93.43	0	1.6	103.75	3	98.28	5.28
NFDHA	NA	0	1.28	86.77	0	1.92	85.07	0	3.2	107.58	3	93.14	13.46
9Cl-PF3ONS	NA	0	2.56	101.31	0	3.84	114.99	0	6.42	111.48	3	109.26	6.5
11Cl-PF3OUdS	NA	0	2.56	102.35	0	3.84	116.32	0	6.42	114.81	3	111.16	6.9
PFEESA	NA	0	0.64	91.46	0	0.958	101.5	0	1.6	102.29	3	98.42	6.13
3:3 FTCA	NA	0	2.56	95.96	0	3.83	96.7	0	6.41	100.16	3	97.61	2.3
5:3 FTCA	NA	0	16	106.04	0	23.9	96.12	0	40	103.66	3	101.94	5.08
7:3 FTCA	NA	0	16	111.25	0	23.9	107.11	0	40	109.66	3	109.34	1.91

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-25 Sediment #2 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveris		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.62	103.55	0	3.93	105.01	0	6.55	105.9	3	104.82	1.13
PFPeA	0.032	3	1.31	103.66	3	1.96	108.39	3	3.28	110.11	3	107.39	3.11
PFHxA	0.027	2	0.655	98.63	2	0.982	101.7	2	1.64	99.57	3	99.97	1.57
PFHpA	NA	0	0.655	96.44	0	0.982	102.61	0	1.64	101.22	3	100.09	3.24
PFOA	NA	0	0.655	103.46	0	0.982	101.8	0	1.64	103.86	3	103.04	1.06
PFNA	NA	0	0.655	100.91	0	0.982	106.96	0	1.64	104.47	3	104.11	2.92
PFDA	NA	0	0.655	107.89	0	0.982	108.32	0	1.64	105.89	3	107.37	1.21
PFUnA	NA	0	0.655	100.81	0	0.982	104.25	0	1.64	106.71	3	103.92	2.85
PFDoA	NA	0	0.655	97.71	0	0.982	103.47	0	1.64	104.67	3	101.95	3.65
PFTrDA	NA	0	0.655	114.4	0	0.982	111.38	0	1.64	110.57	3	112.11	1.8
PFTeDA	NA	0	0.655	103.56	0	0.982	108.32	0	1.64	106.71	3	106.2	2.28
PFBS	NA	0	0.655	103.16	0	0.982	105.2	0	1.64	106.71	3	105.02	1.7
PFPeS	NA	0	0.657	100.97	0	0.985	106.3	0	1.65	105.46	3	104.24	2.75
PFHxS	NA	0	0.655	109.67	0	0.982	111.04	0	1.64	109.15	3	109.95	0.89
PFHpS	NA	0	0.657	98.58	0	0.984	95.73	0	1.64	97.15	3	97.16	1.46
PFOS	NA	0	0.655	105.24	0	0.982	109.68	0	1.64	111.38	3	108.77	2.91
PFNS	NA	0	0.657	101.57	0	0.983	96.45	0	1.64	110.37	3	102.79	6.85
PFDS	NA	0	0.655	98.21	0	0.982	98.78	0	1.64	103.46	3	100.15	2.87
PFDoS	NA	0	0.656	91.2	0	0.983	94.03	0	1.64	91.46	3	92.23	1.7
4:2 FTS	NA	0	2.62	102.79	0	3.93	104.85	0	6.55	102.39	3	103.34	1.28
6:2 FTS	0.23	2	2.36	97.04	2	3.54	126.38	2	5.91	105.86	3	109.76	13.71
8:2 FTS	NA	0	2.62	118.17	0	3.93	126.75	0	6.55	128.13	3	124.35	4.34
PFOSA	NA	0	0.655	103.76	0	0.982	109.68	0	1.64	107.93	3	107.12	2.84
N-MeFOSA	NA	0	0.754	104.33	0	1.13	110.03	0	1.89	109.89	3	108.08	3.01
N-EtFOSA	NA	0	1.64	108.94	0	2.45	116.03	0	4.1	111.55	3	112.18	3.2
N-MeFOSAA	NA	0	0.655	92.52	0	0.982	98.84	0	1.64	104.27	3	98.54	5.97
N-EtFOSAA	NA	0	0.655	114.96	0	0.982	112.73	0	1.64	106.5	3	111.4	3.93
N-MeFOSE	NA	0	6.55	111.29	0	9.82	115.45	0	16.4	117.68	3	114.81	2.82
N-EtFOSE	NA	0	4.91	106.99	0	7.36	112.09	0	12.3	113.01	3	110.7	2.93
HFPO-DA	NA	0	2.49	108.84	0	3.73	110.37	0	6.23	101.13	3	106.78	4.64
ADONA	NA	0	2.63	119.42	0	3.94	113.12	0	6.57	101.02	3	111.18	8.41
PFMPA	NA	0	1.31	97.71	0	1.96	96.27	0	3.28	98.68	3	97.55	1.24
PFMBA	NA	0	0.655	100.96	0	0.982	100.65	0	1.64	102.24	3	101.28	0.83
NFDHA	NA	0	1.31	68.24	0	1.96	115.78	0	3.28	99.38	3	94.47	25.56
9Cl-PF3ONS	NA	0	2.63	127.54	0	3.93	120.52	0	6.57	108.67	3	118.91	8.02
11Cl-PF3OUdS	NA	0	2.63	130.84	0	3.93	123.06	0	6.56	109.6	3	121.17	8.87
PFEESA	NA	0	0.655	92.41	0	0.982	100.17	0	1.64	103.25	3	98.61	5.66
3:3 FTCA	NA	0	2.62	93.77	0	3.93	94.65	0	6.55	95.98	3	94.8	1.18
5:3 FTCA	NA	0	16.4	104.27	0	24.5	102.45	0	41	98.29	3	101.67	3.01
7:3 FTCA	NA	0	16.4	113.62	0	24.5	109.11	0	41	106.59	3	109.77	3.24

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-26 Sediment #3 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	2.64	105.94	0	3.96	107.16	0	6.61	106.21	3	106.44	0.6
PFPeA	0.034	3	1.32	102.98	3	1.98	109.06	3	3.3	109.06	3	107.03	3.28
PFHxA	0.0303	3	0.66	95.7	3	0.989	102.46	3	1.65	101.39	3	99.85	3.64
PFHpA	0.0365	2	0.66	95.32	2	0.989	100.83	2	1.65	103.65	3	99.93	4.24
PFOA	0.107	3	0.66	97.58	3	0.989	100.81	3	1.65	98.99	3	99.13	1.63
PFNA	NA	0	0.66	102.17	0	0.989	106.17	0	1.65	105.25	3	104.53	2
PFDA	0.0617	3	0.66	99.54	3	0.989	102.09	3	1.65	107.17	3	102.94	3.77
PFUnA	0.0477	3	0.66	97.27	3	0.989	101.05	3	1.65	106.81	3	101.71	4.72
PFDoA	NA	0	0.66	112.58	0	0.989	110.59	0	1.65	108.69	3	110.62	1.76
PFTrDA	NA	0	0.66	114.54	0	0.989	112.61	0	1.65	111.31	3	112.82	1.44
PFTeDA	NA	0	0.66	102.57	0	0.989	107.55	0	1.65	105.45	3	105.19	2.38
PFBS	NA	0	0.66	104.8	0	0.989	106.2	0	1.65	109.49	3	106.83	2.26
PFPeS	NA	0	0.662	100.05	0	0.992	106.22	0	1.65	103.03	3	103.1	2.99
PFHxS	0.0237	3	0.66	110.81	3	0.989	115.61	3	1.65	114.53	3	113.65	2.22
PFHpS	NA	0	0.661	94.2	0	0.991	99.16	0	1.65	98.99	3	97.45	2.89
PFOS	0.685	3	0.66	NA	3	0.989	110.72	3	1.65	111.6	2	111.16	0.56
PFNS	NA	0	0.661	92.37	0	0.991	98.18	0	1.65	112.5	3	101.02	10.26
PFDS	0.042	1	0.66	94.39	1	0.989	99.19	1	1.65	100.08	3	97.89	3.12
PFDoS	NA	0	0.661	90.12	0	0.99	93.2	0	1.65	91.74	3	91.69	1.68
4:2 FTS	NA	0	2.64	100.63	0	3.96	100.76	0	6.61	106.56	3	102.65	3.3
6:2 FTS	0.182	1	2.38	98.37	1	3.57	103.02	1	5.95	103.66	3	101.69	2.84
8:2 FTS	NA	0	2.64	114.66	0	3.96	117.28	0	6.61	118.11	3	116.68	1.54
PFOSA	NA	0	0.66	105.2	0	0.989	107.55	0	1.65	108.28	3	107.01	1.5
N-MeFOSA	NA	0	0.759	105.67	0	1.14	108.19	0	1.9	111.23	3	108.36	2.57
N-EtFOSA	NA	0	1.65	109.7	0	2.47	112.28	0	4.13	113.4	3	111.79	1.7
N-MeFOSAA	NA	0	0.66	97.74	0	0.989	104.31	0	1.65	104.44	3	102.16	3.75
N-EtFOSAA	0.131	3	0.66	104.59	3	0.989	100.37	3	1.65	106.2	3	103.72	2.9
N-MeFOSE	NA	0	6.6	101.87	0	9.89	107.89	0	16.5	108.08	3	105.95	3.34
N-EtFOSE	NA	0	4.95	108.69	0	7.42	111.55	0	12.4	112.9	3	111.05	1.94
HFPO-DA	NA	0	2.51	98.81	0	3.76	127.3	0	6.28	101.85	3	109.32	14.31
ADONA	NA	0	2.65	98.87	0	3.97	118.14	0	6.62	103.79	3	106.93	9.36
PFMPA	NA	0	1.32	97.47	0	1.98	97.47	0	3.3	100.4	3	98.45	1.72
PFMBA	NA	0	0.66	100.2	0	0.989	94.84	0	1.65	103.23	3	99.43	4.27
NFDHA	NA	0	1.32	89.39	0	1.98	104.71	0	3.3	93.11	3	95.74	8.35
9Cl-PF3ONS	NA	0	2.64	105.29	0	3.97	124.61	0	6.62	111.44	3	113.78	8.67
11Cl-PF3OUdS	NA	0	2.64	108.83	0	3.96	129.21	0	6.62	113.1	3	117.05	9.18
PFEESA	NA	0	0.66	101.06	0	0.989	95.58	0	1.65	103.84	3	100.16	4.19
3:3 FTCA	NA	0	2.64	91.02	0	3.96	89.38	0	6.61	88.45	3	89.62	1.45
5:3 FTCA	NA	0	16.5	78.79	0	24.7	81.38	0	41.3	80.13	3	80.1	1.62
7:3 FTCA	NA	0	16.5	102.83	0	24.7	100.94	0	41.3	94.5	3	99.43	4.39

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-27 Biosolid #1 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	5.3	2	25.1	74.22	2	37.7	105.67	2	62.4	98.05	3	92.64	17.71
PFPeA	0.96	3	12.6	89.97	3	18.8	113.96	3	31.2	108.77	3	104.23	12.11
PFHxA	5.78	3	6.28	85.23	3	9.41	105.37	3	15.6	94.97	3	95.19	10.58
PFHpA	0.359	1	6.28	83.64	1	9.41	103.44	1	15.6	97.67	3	94.92	10.73
PFOA	2.52	3	6.28	84.74	3	9.41	105.6	3	15.6	96.44	3	95.59	10.94
PFNA	1.15	3	6.28	87.1	3	9.41	107.09	3	15.6	99.89	3	98.03	10.33
PFDA	4.84	3	6.28	70.74	3	9.41	97.64	3	15.6	95.9	3	88.09	17.08
PFUnA	1.46	3	6.28	87.3	3	9.41	110.11	3	15.6	101.7	3	99.7	11.57
PFDoA	2.52	3	6.28	89.31	3	9.41	156.18	3	15.6	106.87	3	117.46	29.52
PFTrDA	0.808	3	6.28	108.45	3	9.41	149.62	3	15.6	127.29	3	128.45	16.05
PFTeDA	1.46	3	6.28	84.03	3	9.41	104.17	3	15.6	105.39	3	97.86	12.25
PFBS	2.37	3	6.28	109.72	3	9.41	70.22	3	15.6	81.64	3	87.2	23.31
PFPeS	1.64	1	6.29	101.33	1	9.44	109.66	1	15.7	108.45	3	106.48	4.23
PFHxS	0.943	3	6.28	116.26	3	9.41	134.03	3	15.6	122.55	3	124.28	7.25
PFHpS	NA	0	6.29	91.1	0	9.44	105.22	0	15.6	97.22	3	97.85	7.23
PFOS	33.4	3	6.28	NA	3	9.41	NA	3	15.6	NA	0	NA	NA
PFNS	NA	0	6.29	87.44	0	9.43	105.59	0	15.6	97.22	3	96.75	9.39
PFDS	1.68	3	6.28	56.78	3	9.41	74.08	3	15.6	73.43	3	68.1	14.4
PFDoS	NA	0	6.29	31.66	0	9.43	29.89	0	15.6	29.11	3	30.22	4.32
4:2 FTS	NA	0	25.1	89.91	0	37.7	109.06	0	62.4	102.33	3	100.43	9.68
6:2 FTS	11.1	3	22.6	52.2	3	33.9	87.71	3	56.3	90.37	3	76.76	27.76
8:2 FTS	NA	0	25.1	105.33	0	37.7	134.24	0	62.4	126.26	3	121.94	12.24
PFOSA	1.53	3	6.28	92.35	3	9.41	112.23	3	15.6	103.85	3	102.81	9.71
N-MeFOSA	0.753	3	7.22	92.03	3	10.8	111.77	3	17.9	108.81	3	104.2	10.21
N-EtFOSA	0.491	3	15.7	94.72	3	23.5	121.5	3	39	111.63	3	109.28	12.39
N-MeFOSAA	11.6	3	6.28	NA	3	9.41	NA	3	15.6	103.39	1	103.39	NA
N-EtFOSAA	4.44	3	6.28	88.45	3	9.41	115.26	3	15.6	106.54	3	103.42	13.22
N-MeFOSE	15.4	3	62.8	93.73	3	94.1	121.71	3	156	113.41	3	109.62	13.11
N-EtFOSE	3.24	3	47.1	102.96	3	70.6	137.44	3	117	131.11	3	123.84	14.82
HFPO-DA	NA	0	23.8	81.76	0	35.8	116.93	0	59.3	102.29	3	100.33	17.61
ADONA	NA	0	25.2	87.69	0	37.8	116.17	0	62.6	108.07	3	103.98	14.11
PFMPA	NA	0	12.6	82.24	0	18.8	33.45	0	31.2	24.44	3	46.71	66.57
PFMBA	NA	0	6.28	87.72	0	9.41	108.48	0	15.6	113.43	3	103.21	13.22
NFDHA	NA	0	12.6	28.99	0	18.8	50.85	0	31.2	42.69	3	40.85	27.05
9Cl-PF3ONS	NA	0	25.2	86.25	0	37.7	116.74	0	62.6	110.37	3	104.45	15.4
11Cl-PF3OUdS	NA	0	25.1	61.55	0	37.7	97.58	0	62.5	95.98	3	85.04	23.94
PFEESA	NA	0	6.28	92.58	0	9.41	102.39	0	15.6	97.41	3	97.46	5.03
3:3 FTCA	NA	0	25.1	74.11	0	37.7	27.85	0	62.4	18.19	3	40.05	74.63
5:3 FTCA	18.1	3	157	82.44	3	235	129.98	3	390	104.66	3	105.69	22.5
7:3 FTCA	9.12	3	157	70.17	3	235	115.08	3	390	106.18	3	97.14	24.48

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-28 D-28 Biosolid #2 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	26.3	92.64	0	38.8	109.2	0	63.9	104.07	3	101.97	8.31
PFPeA	0.413	3	13.1	92.52	3	19.4	109.4	3	31.9	107.96	3	103.29	9.06
PFHxA	1.09	3	6.56	85.84	3	9.71	102.09	3	16	96.15	3	94.69	8.68
PFHpA	NA	0	6.56	90.66	0	9.71	103.28	0	16	94.97	3	96.3	6.66
PFOA	0.524	3	6.56	83.59	3	9.71	99.28	3	16	99.49	3	94.12	9.69
PFNA	NA	0	6.56	92.67	0	9.71	103.85	0	16	104.03	3	100.18	6.5
PFDA	0.726	3	6.56	88.29	3	9.71	107.16	3	16	102.5	3	99.31	9.9
PFUnA	NA	0	6.56	91.76	0	9.71	107.45	0	16	104.24	3	101.15	8.19
PFDoA	NA	0	6.56	102.06	0	9.71	112.93	0	16	120.21	3	111.73	8.17
PFTrDA	0.554	3	6.56	88.91	3	9.71	112.73	3	16	116.66	3	106.1	14.16
PFTeDA	0.353	2	6.56	91.9	2	9.71	104.5	2	16	107.42	3	101.27	8.14
PFBS	0.38	3	6.56	101.64	3	9.71	107.98	3	16	108.5	3	106.04	3.6
PFPeS	0.15	1	6.58	89.24	1	9.74	106.55	1	16	100.97	3	98.92	8.93
PFHxS	0.196	1	6.56	98.61	1	9.71	115.36	1	16	109.22	3	107.73	7.86
PFHpS	NA	0	6.58	81.92	0	9.73	96.9	0	16	95.22	3	91.35	8.99
PFOS	1.25	3	6.56	96.23	3	9.71	109.4	3	16	108.09	3	104.57	6.94
PFNS	NA	0	6.58	82.1	0	9.73	101.81	0	16	101.8	3	95.23	11.95
PFDS	NA	0	6.56	75.06	0	9.71	103.15	0	16	97.12	3	91.78	16.11
PFDoS	NA	0	6.57	54.54	0	9.73	59.34	0	16	47.69	3	53.86	10.88
4:2 FTS	NA	0	26.3	91.15	0	38.8	103.52	0	63.9	104.31	3	99.66	7.4
6:2 FTS	23	1	23.7	NA	1	35	63.89	1	57.6	68.07	2	65.98	4.48
8:2 FTS	NA	0	26.3	102.08	0	38.8	130.44	0	63.9	122.09	3	118.2	12.33
PFOSA	NA	0	6.56	93.52	0	9.71	106.74	0	16	107.74	3	102.66	7.73
N-MeFOSA	NA	0	7.55	95.5	0	11.2	107.75	0	18.4	105.69	3	102.98	6.37
N-EtFOSA	NA	0	16.4	97.78	0	24.3	112.6	0	39.9	109.76	3	106.71	7.37
N-MeFOSAA	1.34	3	6.56	86.58	3	9.71	106.69	3	16	106.61	3	99.96	11.59
N-EtFOSAA	0.533	3	6.56	98.67	3	9.71	112.62	3	16	106.23	3	105.84	6.6
N-MeFOSE	NA	0	65.6	94.01	0	97.1	109.47	0	160	107.13	3	103.54	8.05
N-EtFOSE	NA	0	49.2	101.03	0	72.8	118.04	0	120	118.71	3	112.59	8.9
HFPO-DA	NA	0	24.9	92.03	0	36.9	109.93	0	60.7	98.66	3	100.21	9.03
ADONA	NA	0	26.3	96.59	0	39	118.12	0	64.1	99.75	3	104.82	11.09
PFMPA	NA	0	13.1	83.73	0	19.4	54.77	0	31.9	67.66	3	68.72	21.12
PFMBA	NA	0	6.56	86.22	0	9.71	98.32	0	16	96.11	3	93.55	6.89
NFDHA	NA	0	13.1	96.01	0	19.4	110.24	0	31.9	99.3	3	101.85	7.31
9Cl-PF3ONS	NA	0	26.3	101.14	0	38.9	126.96	0	64	106.03	3	111.38	12.31
11Cl-PF3OUdS	NA	0	26.3	86.47	0	38.9	125.54	0	64	104.7	3	105.57	18.52
PFEESA	NA	0	6.56	89.03	0	9.71	103.96	0	16	97.15	3	96.72	7.73
3:3 FTCA	NA	0	26.3	77.7	0	38.8	50.67	0	63.9	59.32	3	62.56	22.06
5:3 FTCA	9.96	3	164	83.16	3	243	98.23	3	399	89.02	3	90.14	8.43
7:3 FTCA	NA	0	164	81.93	0	243	103.15	0	399	90.8	3	91.96	11.59

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-29: Biosolid #3 Matrix Spike Recoveries (µg/kg)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	15.9	3	27.4	36.96	3	41.4	95.89	3	67.7	93.35	3	75.4	44.18
PFPeA	3.32	3	13.7	95.43	3	20.7	120.88	3	33.9	115.04	3	110.45	12.07
PFHxA	5.11	3	6.86	81.01	3	10.3	104.1	3	16.9	104.59	3	96.57	13.96
PFHpA	0.308	1	6.86	87.6	1	10.3	91.01	1	16.9	103.25	3	93.95	8.76
PFOA	1.57	3	6.86	81.4	3	10.3	100	3	16.9	102.15	3	94.52	12.07
PFNA	3.63	3	6.86	86.54	3	10.3	103.57	3	16.9	105.7	3	98.6	10.65
PFDA	2.03	3	6.86	87.52	3	10.3	107.14	3	16.9	109.69	3	101.45	11.96
PFUnA	2.13	3	6.86	87.96	3	10.3	96.13	3	16.9	103.42	3	95.84	8.07
PFDoA	2.83	3	6.86	78.21	3	10.3	99.42	3	16.9	123.04	3	100.22	22.38
PFTrDA	1.5	3	6.86	114.3	3	10.3	155.36	3	16.9	174.79	3	148.15	20.85
PFTeDA	1.67	3	6.86	81.34	3	10.3	104.78	3	16.9	106.82	3	97.65	14.5
PFBS	2.7	3	6.86	55.94	3	10.3	117.74	3	16.9	68.99	3	80.89	40.27
PFPeS	NA	0	6.88	142.6	0	10.4	143.01	0	17	125.64	3	137.08	7.23
PFHxS	40.5	3	6.86	NA	3	10.3	NA	3	16.9	NA	0	NA	NA
PFHpS	NA	0	6.87	178.7	0	10.3	175.87	0	17	124.08	3	159.55	19.27
PFOS	13.5	3	6.86	NA	3	10.3	NA	3	16.9	178.73	1	178.73	NA
PFNS	3.08	1	6.87	77.83	1	10.3	93.47	1	17	103.56	3	91.62	14.15
PFDS	4.9	3	6.86	113.32	3	10.3	115.12	3	16.9	83.73	3	104.06	16.94
PFDoS	NA	0	6.87	43.52	0	10.3	36.74	0	17	33.09	3	37.79	14.01
4:2 FTS	NA	0	27.4	84.77	0	41.4	100.25	0	67.7	102.82	3	95.95	10.17
6:2 FTS	1.91	3	24.8	96.3	3	37.3	109.32	3	61	111.45	3	105.69	7.76
8:2 FTS	NA	0	27.4	117.54	0	41.4	130.34	0	67.7	124.42	3	124.1	5.16
PFOSA	1.22	3	6.86	86.03	3	10.3	104.35	3	16.9	106.54	3	98.97	11.38
N-MeFOSA	NA	0	7.88	102.21	0	11.9	117.92	0	19.5	114.58	3	111.57	7.42
N-EtFOSA	NA	0	17.1	105.24	0	25.9	119.17	0	42.3	116.54	3	113.65	6.51
N-MeFOSAA	4.85	3	6.86	89.5	3	10.3	110.15	3	16.9	117.02	3	105.55	13.57
N-EtFOSAA	9.42	3	6.86	NA	3	10.3	108.86	3	16.9	115.25	2	112.06	4.03
N-MeFOSE	11.8	3	68.6	128.23	3	103	107.96	3	169	109.16	3	115.12	9.88
N-EtFOSE	4.49	3	51.4	79.56	3	77.6	96.23	3	127	97.76	3	91.18	11.07
HFPO-DA	NA	0	26.1	88.88	0	32.2	141.72	0	53.2	140.61	3	123.74	24.4
ADONA	NA	0	27.5	93.83	0	41.5	118.55	0	67.9	126.07	3	112.82	14.95
PFMPA	NA	0	13.7	88.82	0	20.7	16.69	0	33.9	17.94	3	41.15	100.35
PFMBA	NA	0	6.86	89.54	0	10.3	146.51	0	16.9	135.74	3	123.93	24.42
NFDHA	NA	0	13.7	49.19	0	20.7	61.87	0	33.9	66.5	3	59.19	15.14
9Cl-PF3ONS	NA	0	27.5	92.36	0	41.5	117.77	0	67.9	128.01	3	112.71	16.28
11Cl-PF3OUdS	NA	0	27.5	74.63	0	41.5	102.43	0	67.8	105.76	3	94.27	18.13
PFEESA	NA	0	6.86	94.06	0	10.3	112.89	0	16.9	117.98	3	108.31	11.63
3:3 FTCA	NA	0	27.4	89.65	0	41.4	10.06	0	67.7	10.49	3	36.73	124.76
5:3 FTCA	53.5	3	171	120.33	3	259	122.7	3	423	116.81	3	119.95	2.47
7:3 FTCA	16	3	171	94.52	3	259	128.97	3	423	135.71	3	119.73	18.45

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-30 Fish Tissue #1 Matrix Spike Recoveries (µg/kg wet weight)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	6.43	107.05	0	9.68	107.06	0	16.1	100.62	3	104.91	3.54
PFPeA	0.802	3	3.22	117.77	3	4.84	112.09	3	8.03	104.8	3	111.55	5.83
PFHxA	0.163	1	1.61	99.04	1	2.42	100.15	1	4.01	92.12	3	97.1	4.48
PFHpA	NA	0	1.61	115.94	0	2.42	105.24	0	4.01	107.48	3	109.55	5.15
PFOA	0.099	1	1.61	108.76	1	2.42	102.39	1	4.01	97.94	3	103.03	5.28
PFNA	0.415	3	1.61	106.1	3	2.42	103.52	3	4.01	98.8	3	102.81	3.6
PFDA	0.295	3	1.61	106.13	3	2.42	108.9	3	4.01	102.28	3	105.77	3.14
PFUnA	0.496	3	1.61	107.53	3	2.42	105.55	3	4.01	98.86	3	103.98	4.37
PFDoA	0.16	3	1.61	121.14	3	2.42	113.24	3	4.01	94.53	3	109.64	12.46
PFTrDA	0.399	3	1.61	132.76	3	2.42	131.36	3	4.01	111.07	3	125.06	9.71
PFTeDA	NA	0	1.61	112.02	0	2.42	107.73	0	4.01	99.92	3	106.55	5.76
PFBS	NA	0	1.61	108.48	0	2.42	107.31	0	4.01	102.66	3	106.15	2.9
PFPeS	NA	0	1.61	98.76	0	2.43	98.76	0	4.02	95.76	3	97.76	1.77
PFHxS	0.106	3	1.61	150.76	3	2.42	135.46	3	4.01	122.59	3	136.27	10.35
PFHpS	NA	0	1.61	106.84	0	2.43	103.57	0	4.02	95.03	3	101.81	5.99
PFOS	1.34	3	1.61	109.71	3	2.42	112.96	3	4.01	99.33	3	107.34	6.63
PFNS	NA	0	1.61	83.43	0	2.43	88.59	0	4.02	74.07	3	82.03	8.97
PFDS	NA	0	1.61	96.91	0	2.42	97.52	0	4.01	87.05	3	93.83	6.26
PFDoS	NA	0	1.61	85.92	0	2.43	87.78	0	4.02	83.09	3	85.59	2.76
4:2 FTS	NA	0	6.43	110	0	9.68	103.85	0	16.1	100.62	3	104.82	4.55
6:2 FTS	3.02	1	5.8	64.77	1	8.73	77.57	1	14.5	80.97	3	74.44	11.48
8:2 FTS	NA	0	6.43	124.47	0	9.68	126	0	16.1	114.11	3	121.52	5.32
PFOSA	0.468	3	1.61	112.37	3	2.42	112.1	3	4.01	103.55	3	109.34	4.59
N-MeFOSA	NA	0	1.85	127.05	0	2.78	127.59	0	4.61	130.07	3	128.24	1.26
N-EtFOSA	NA	0	4.02	111.04	0	6.05	109.58	0	10.1	95.03	3	105.22	8.41
N-MeFOSAA	NA	0	1.61	105.61	0	2.42	115.17	0	4.01	105.48	3	108.76	5.11
N-EtFOSAA	NA	0	1.61	125.92	0	2.42	116.56	0	4.01	117.43	3	119.97	4.31
N-MeFOSE	NA	0	16.1	150.53	0	24.2	131.57	0	40.1	126.94	3	136.35	9.17
N-EtFOSE	4.36	3	12.1	197.17	3	18.2	342.83	3	30.1	216.01	3	252	31.44
HFPO-DA	NA	0	6.11	89.5	0	9.2	106.86	0	15.3	101.95	3	99.43	9
ADONA	NA	0	6.45	117.01	0	9.7	113.36	0	16.1	107.07	3	112.48	4.47
PFMPA	NA	0	3.22	143.51	0	4.84	140.1	0	8.03	133.32	3	138.98	3.73
PFMBA	NA	0	1.61	116.56	0	2.42	115.87	0	4.01	103.92	3	112.12	6.34
NFDHA	NA	0	3.22	75.98	0	4.84	50.57	0	8.03	50.31	3	58.95	25.02
9Cl-PF3ONS	NA	0	6.44	142.29	0	9.7	143.59	0	16.1	137.79	3	141.22	2.15
11Cl-PF3OUdS	NA	0	6.44	158.53	0	9.69	155.78	0	16.1	146.71	3	153.67	4.02
PFEESA	NA	0	1.61	124.83	0	2.42	128	0	4.01	116.22	3	123.02	4.96
3:3 FTCA	NA	0	6.43	74.51	0	9.68	68.7	0	16.1	84.04	3	75.75	10.22
5:3 FTCA	NA	0	40.2	172.97	0	60.5	165.62	0	101	136.64	3	158.41	12.13
7:3 FTCA	1.52	3	40.2	273.25	3	60.5	276.83	3	101	251.17	3	267.09	5.2

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-31 Fish Tissue #2 Matrix Spike Recoveries (µg/kg wet weight)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	6.42	109.29	0	9.68	106.71	0	16	101.26	3	105.75	3.88
PFPeA	NA	0	3.21	106.64	0	4.84	115.23	0	8	107.84	3	109.9	4.23
PFHxA	NA	0	1.61	110.37	0	2.42	106.9	0	4	97.85	3	105.04	6.15
PFHpA	NA	0	1.61	116.37	0	2.42	108.69	0	4	109.66	3	111.57	3.75
PFOA	NA	0	1.61	111.61	0	2.42	107.59	0	4	100.84	3	106.68	5.1
PFNA	NA	0	1.61	113.28	0	2.42	112.14	0	4	104.66	3	110.03	4.25
PFDA	0.83	3	1.61	101.45	3	2.42	104.27	3	4	96.51	3	100.74	3.9
PFUnA	1.5	3	1.61	107.92	3	2.42	105.25	3	4	95.59	3	102.92	6.3
PFDoA	0.632	3	1.61	139.53	3	2.42	113.57	3	4	106.43	3	119.84	14.54
PFTrDA	1.31	3	1.61	146.96	3	2.42	126.21	3	4	108.6	3	127.26	15.09
PFTeDA	0.419	3	1.61	110.23	3	2.42	105.82	3	4	100.61	3	105.55	4.56
PFBS	NA	0	1.61	112.44	0	2.42	108.97	0	4	103.27	3	108.23	4.28
PFPeS	NA	0	1.61	87.97	0	2.43	96.71	0	4.01	87.06	3	90.58	5.88
PFHxS	NA	0	1.61	100.43	0	2.42	108.97	0	4	100.7	3	103.36	4.69
PFHpS	NA	0	1.61	99.79	0	2.43	104.26	0	4.01	97.67	3	100.57	3.34
PFOS	10.3	3	1.61	NA	3	2.42	NA	3	4	NA	0	NA	NA
PFNS	NA	0	1.61	116.79	0	2.43	113.87	0	4.01	96.03	3	108.9	10.32
PFDS	0.413	3	1.61	94.64	3	2.42	97.81	3	4	84.34	3	92.26	7.64
PFDoS	NA	0	1.61	94.4	0	2.43	56.01	0	4.01	33.41	3	61.28	50.32
4:2 FTS	NA	0	6.42	108.2	0	9.68	102.62	0	16	97.1	3	102.64	5.41
6:2 FTS	NA	0	5.79	118.02	0	8.73	112.84	0	14.4	106.24	3	112.36	5.25
8:2 FTS	NA	0	6.42	120.87	0	9.68	125.3	0	16	120.61	3	122.26	2.15
PFOSA	NA	0	1.61	113.28	0	2.42	110.62	0	4	104.84	3	109.58	3.94
N-MeFOSA	NA	0	1.85	111.54	0	2.78	98.43	0	4.6	109.44	3	106.47	6.61
N-EtFOSA	NA	0	4.01	118.27	0	6.05	120.71	0	10	116.12	3	118.37	1.94
N-MeFOSAA	NA	0	1.61	116.6	0	2.42	107.31	0	4	99.25	3	107.72	8.06
N-EtFOSAA	NA	0	1.61	114.31	0	2.42	114.49	0	4	107.01	3	111.94	3.81
N-MeFOSE	NA	0	16.1	179.67	0	24.2	186.4	0	40	129.55	3	165.21	18.8
N-EtFOSE	1.89	1	12.1	92.99	1	18.2	168.53	1	30	105.24	3	122.25	33.16
HFPO-DA	NA	0	6.1	115.83	0	9.2	109.71	0	15.2	94.75	3	106.76	10.16
ADONA	NA	0	6.44	151.24	0	9.7	124.35	0	16	106.66	3	127.42	17.62
PFMPA	NA	0	3.21	110.06	0	4.84	115.57	0	8	106.07	3	110.56	4.31
PFMBA	NA	0	1.61	110.37	0	2.42	109.25	0	4	103.01	3	107.54	3.69
NFDHA	NA	0	3.21	73.23	0	4.84	74.23	0	8	74.64	3	74.03	0.98
9Cl-PF3ONS	NA	0	6.43	160.19	0	9.7	136.72	0	16	113.32	3	136.74	17.14
11Cl-PF3OUdS	NA	0	6.43	153.86	0	9.69	134.43	0	16	110.24	3	132.84	16.45
PFEESA	NA	0	1.61	136.93	0	2.42	126.2	0	4	113.11	3	125.41	9.51
3:3 FTCA	NA	0	6.42	111.98	0	9.68	118.06	0	16	103.78	3	111.28	6.44
5:3 FTCA	NA	0	40.1	233.54	0	60.5	217.05	0	100	186.13	3	212.24	11.34
7:3 FTCA	NA	0	40.1	196.57	0	60.5	223.09	0	100	172.19	3	197.28	12.9

py_Log Date 7/21/2021 2:39:01 PM

Notes:
(1) Values from Appendix C: Individual Sample Native Concentrations
(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery
(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery
(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Table D-32 Clam Tissue Matrix Spike Recoveries (µg/kg wet weight)

Analyte	Native Mean Concentration [1]	Low Spike Recovery			Medium Spike Recovery			High Spike Recovery			All Spike Recoveries		
		n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [2]	Mean Spike Concentration	% Recovery	n [3]	% Mean Recovery	RSD [4]
PFBA	NA	0	6.42	107.95	0	9.55	106.11	0	16.2	99.59	3	104.55	4.2
PFPeA	NA	0	3.21	112.15	0	4.78	113.47	0	8.07	106.28	3	110.64	3.46
PFHxA	NA	0	1.61	102.48	0	2.39	105.6	0	4.03	95.78	3	101.29	4.95
PFHpA	NA	0	1.61	105.82	0	2.39	107.13	0	4.03	97.85	3	103.6	4.84
PFOA	NA	0	1.61	106.01	0	2.39	106	0	4.03	100.57	3	104.19	3.01
PFNA	NA	0	1.61	109.34	0	2.39	106.42	0	4.03	98.76	3	104.84	5.21
PFDA	NA	0	1.61	108.71	0	2.39	107.26	0	4.03	99.58	3	105.18	4.66
PFUnA	NA	0	1.61	106.86	0	2.39	107.39	0	4.03	101.74	3	105.33	2.97
PFDoA	NA	0	1.61	110.92	0	2.39	110.57	0	4.03	105.62	3	109.04	2.72
PFTrDA	NA	0	1.61	106.83	0	2.39	103.78	0	4.03	99.83	3	103.48	3.39
PFTeDA	NA	0	1.61	107.06	0	2.39	107.39	0	4.03	100.58	3	105.01	3.65
PFBS	NA	0	1.61	111	0	2.39	109.08	0	4.03	102.06	3	107.38	4.38
PFPeS	NA	0	1.61	106.02	0	2.4	100.29	0	4.04	95.8	3	100.7	5.09
PFHxS	NA	0	1.61	125.91	0	2.39	119.53	0	4.03	112.32	3	119.25	5.7
PFHpS	NA	0	1.61	102.28	0	2.39	99.19	0	4.04	94.23	3	98.56	4.12
PFOS	NA	0	1.61	107.06	0	2.39	107.26	0	4.03	100.25	3	104.86	3.81
PFNS	NA	0	1.61	75.36	0	2.39	84.25	0	4.04	88.05	3	82.55	7.89
PFDS	NA	0	1.61	72.44	0	2.39	72.53	0	4.03	73.39	3	72.78	0.73
PFDoS	NA	0	1.61	38.91	0	2.39	46.18	0	4.04	39.41	3	41.5	9.78
4:2 FTS	NA	0	6.42	110.96	0	9.55	106.03	0	16.2	100.62	3	105.87	4.88
6:2 FTS	12.5	1	5.79	NA	1	8.61	NA	1	14.6	17.62	1	17.62	NA
8:2 FTS	NA	0	6.42	125.44	0	9.55	130.88	0	16.2	117.52	3	124.62	5.39
PFOSA	NA	0	1.61	110.58	0	2.39	109.91	0	4.03	103.39	3	107.96	3.68
N-MeFOSA	NA	0	1.85	117.52	0	2.75	103.28	0	4.64	111.79	3	110.86	6.46
N-EtFOSA	NA	0	4.01	137.28	0	5.97	148.61	0	10.1	114.52	3	133.47	13.01
N-MeFOSAA	NA	0	1.61	108.91	0	2.39	110.74	0	4.03	100.83	3	106.83	4.94
N-EtFOSAA	NA	0	1.61	115.56	0	2.39	109.33	0	4.03	104.05	3	109.65	5.25
N-MeFOSE	NA	0	16.1	126.12	0	23.9	129.7	0	40.3	104.63	3	120.15	11.29
N-EtFOSE	NA	0	12	113.58	0	17.9	138.75	0	30.2	89.2	3	113.84	21.76
HFPO-DA	NA	0	6.1	118.48	0	9.08	103.4	0	15.4	91.08	3	104.32	13.15
ADONA	NA	0	6.44	135.17	0	9.58	110.3	0	16.2	102.47	3	115.98	14.72
PFMPA	NA	0	3.21	106.67	0	4.78	102.3	0	8.07	97.97	3	102.31	4.25
PFMBA	NA	0	1.61	99.78	0	2.39	107.26	0	4.03	103.13	3	103.39	3.62
NFDHA	NA	0	3.21	111.28	0	4.78	70.4	0	8.07	73.43	3	85.03	26.79
9Cl-PF3ONS	NA	0	6.43	124.62	0	9.57	105.82	0	16.2	101.85	3	110.76	10.98
11Cl-PF3OUdS	NA	0	6.43	98.49	0	9.57	83.01	0	16.2	88.25	3	89.91	8.76
PFEESA	NA	0	1.61	106.84	0	2.39	97.67	0	4.03	94.47	3	99.66	6.45
3:3 FTCA	NA	0	6.42	88.34	0	9.55	94.22	0	16.2	85.76	3	89.44	4.85
5:3 FTCA	NA	0	40.1	142.35	0	59.7	147.77	0	101	129.7	3	139.94	6.63
7:3 FTCA	NA	0	40.1	116.95	0	59.7	125.64	0	101	114.19	3	118.93	5.02

py_Log Date 7/21/2021 2:39:01 PM

Notes:

(1) Values from Appendix C: Individual Sample Native Concentrations

(2) Number of spike replicates for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(3) Number of spike categories for the analyte where the mean native concentration is NOT greater than the mean spike concentration (i.e. a “No”) and when there is a numeric value for percent recovery

(4) Relative Standard Deviation (RSD) of the mean percent recoveries. Where n < 1, the RSD cannot be calculated and a value of NA is reported. The RSD is the standard deviation divided by the mean, times 100.

Appendix E

Matrices Type Matrix Recovery

APPENDIX E. SUMMARY MATRIX SPIKE RECOVERY TABLES

This appendix *summarizes* those results presented in Appendix D for each of the 8 environmental matrices. Spike sample matrix recoveries for the four aqueous matrices (groundwater, wastewater, surface water, landfill leachate), the three solid matrices (soil, sediment, biosolids), and tissues. The first eight tables (E1 – E8) report the mean of the sample mean percent recovery, percent RSD, and SD for the 40 PFAS analytes, for the individual matrices from the tables in Appendix D. Two additional summary tables are presented for the combined aqueous media, and for the combined solid media (Tables E-9, E-10).

Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021). Briefly, the tables include the following information.

For Headings: Low, Medium, and High Spike Recovery

- Analyte -The 40 PFAS analytes listed in Table 1 of the Single Lab Validation Study Report
- "n" = The number of samples for a matrix, for spike concentrations, that do not have U or B flagged values. Can range from 0 to 7.
- % Mean Recovery-Mean of the sample mean percent recoveries, for a spike concentration, for the analyte, that does not have U or B flagged values (from the sample matrix recovery tables in section D of the digital appendix, column % Recovery). If all percent recoveries are NA, (i.e., if n = 0 in or mean percent recovery < 0), then NA is reported.
- RSD - Percent RSD of the mean of the sample mean percent recoveries, used to calculate column % Recovery. If the number of samples is less than two, then NA was used.
- SD – Standard deviation of the mean of sample mean percent recoveries. If the number of samples is less than two, then NA is reported.

For Headings: ALL SPIKE RECOVERIES

- "n" - Number of spike categories with percent recovery values, without NA in column "% Recovery" for the analyte.
- % Mean Recovery - Mean of the matrix mean percent recoveries, for the analyte. If the number of spike categories is less than two, NA is reported.
- Lowest % Recovery - Lowest percent recovery of the mean of the three subsamples' percent recoveries for the matrix for values not flagged with U or B and non-negative values from the sample matrix recovery tables in Appendix D of the digital appendix column "% Recovery".
- Highest % Recovery - Highest percent recovery of the mean of the three subsamples' percent recoveries for the matrix for values not flagged with U or B and non-negative values from the sample matrix recovery tables in Appendix D of the digital appendix column "% Recovery".
- RSD - Percent RSD of the matrix mean percent recoveries from columns "% Recovery". If the number of samples is less than two, NA is reported.
- SD – Standard deviation of the matrix mean percent recoveries from columns "% Recovery". If the number of samples is less than two, NA is reported.

Table E-1. All Groundwater Sample Matrix Recovery Table

Data from Tables D1 - D3

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				High 2 Spike Recovery (groundwater only)				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	2	100.50	1.63	1.63	3	112.32	3.40	3.82	3	108.21	4.96	5.37	3	99.67	0.43	0.43	4	105.17	99.21	116.73	5.82	6.12
PFPeA	0	0.00	NA	NA	2	115.54	3.25	3.75	3	114.82	2.19	2.51	3	104.95	1.03	1.09	3	111.77	104.01	118.19	5.29	5.92
PFHxA	0	NA	NA	NA	0	NA	NA	NA	0	NA	NA	NA	3	98.68	1.10	1.09	1	98.68	97.80	99.90	NA	NA
PFHpA	0	NA	NA	NA	3	107.93	5.98	6.46	3	107.59	0.36	0.39	3	98.99	1.88	1.86	3	104.84	97.66	115.22	4.83	5.06
PFOA	0	NA	NA	NA	0	NA	NA	NA	0	NA	NA	NA	3	96.56	0.52	0.50	1	96.56	96.05	97.05	NA	NA
PFNA	3	98.03	7.56	7.41	3	107.94	5.00	5.40	3	108.46	3.56	3.87	3	98.96	0.35	0.35	4	103.34	90.56	112.89	5.44	5.62
PFDA	3	103.39	8.25	8.53	3	117.09	6.09	7.13	3	114.94	4.78	5.50	3	104.45	0.28	0.29	4	109.97	96.23	124.71	6.41	7.05
PFUnA	3	99.85	8.42	8.40	3	112.78	4.95	5.58	3	111.86	4.21	4.71	3	99.91	0.12	0.12	4	106.10	91.09	119.07	6.78	7.19
PFDoA	3	95.24	12.82	12.21	3	113.65	4.98	5.66	3	110.28	8.57	9.45	3	99.68	1.47	1.47	4	104.71	82.26	120.89	8.29	8.68
PFTrDA	3	100.30	9.59	9.62	3	117.29	5.42	6.36	3	114.66	8.85	10.14	3	93.57	3.08	2.88	4	106.45	89.23	126.37	10.69	11.38
PFTeDA	3	99.89	7.20	7.19	3	115.48	4.59	5.31	3	112.83	4.75	5.36	3	98.28	0.99	0.97	4	106.62	91.86	121.26	8.25	8.79
PFBS	1	101.13	NA	NA	1	113.28	NA	NA	1	111.97	NA	NA	3	104.02	0.43	0.45	4	107.60	101.13	113.28	5.53	5.95
PFPeS	1	94.14	NA	NA	1	121.36	NA	NA	2	113.27	2.48	2.81	3	93.01	1.54	1.43	4	105.45	91.43	121.36	13.38	14.11
PFHxS	0	NA	NA	NA	0	NA	NA	NA	0	NA	NA	NA	3	106.72	1.63	1.74	1	106.72	104.81	108.21	NA	NA
PFHpS	1	91.96	NA	NA	3	118.48	9.93	11.76	3	117.17	8.55	10.01	3	92.27	2.54	2.34	4	104.97	89.60	131.50	14.15	14.85
PFOS	0	NA	NA	NA	0	NA	NA	NA	0	NA	NA	NA	2	109.76	1.50	1.64	1	109.76	108.59	110.92	NA	NA
PFNS	3	108.59	20.41	22.17	3	117.16	14.53	17.02	3	120.44	9.47	11.41	3	99.50	4.12	4.10	4	111.42	83.08	130.47	8.43	9.39
PFDS	3	93.64	15.28	14.30	3	106.21	11.88	12.62	3	103.10	7.86	8.11	3	93.36	4.63	4.33	4	99.08	78.04	115.08	6.63	6.56
PFDoS	3	88.74	14.23	12.62	3	99.14	5.56	5.51	3	99.85	7.37	7.36	3	86.89	2.68	2.33	4	93.66	74.59	105.99	7.25	6.79
4:2 FTS	3	98.79	10.05	9.93	3	112.46	6.05	6.80	3	112.93	5.16	5.83	3	98.66	0.56	0.55	4	105.71	87.74	119.66	7.63	8.07
6:2 FTS	1	108.06	NA	NA	2	125.32	5.81	7.29	2	117.19	2.45	2.87	3	99.64	2.71	2.70	4	112.55	97.04	130.47	9.89	11.13
8:2 FTS	2	108.90	0.91	0.99	3	128.21	3.91	5.02	3	124.02	0.38	0.47	3	109.19	4.95	5.41	4	117.58	104.19	134.00	8.51	10.00
PFOSA	0	NA	NA	NA	2	120.21	11.81	14.20	2	131.40	17.31	22.75	3	102.02	0.52	0.53	3	117.88	101.57	147.49	12.58	14.83
N-MeFOSA	3	88.07	14.57	12.83	3	108.60	6.13	6.66	3	`	6.51	7.20	3	98.07	0.03	0.03	4	101.34	73.25	115.11	10.28	10.42
N-EtFOSA	3	86.08	12.45	10.71	3	104.99	8.67	9.10	3	107.64	5.98	6.44	3	103.16	1.31	1.35	4	100.47	73.76	113.73	9.72	9.77
N-MeFOSAA	3	98.15	8.49	8.33	3	129.64	14.46	18.75	3	132.94	12.51	16.64	3	95.77	2.20	2.11	4	114.13	90.25	151.29	17.43	19.89
N-EtFOSAA	3	95.30	7.38	7.03	3	123.55	9.92	12.25	3	122.30	6.86	8.39	3	96.60	1.52	1.47	4	109.44	87.55	137.41	14.25	15.59
N-MeFOSE	3	96.41	9.85	9.49	3	113.00	3.84	4.34	3	112.03	4.91	5.50	3	105.31	0.26	0.28	4	106.68	85.57	117.66	7.18	7.66
N-EtFOSE	3	97.76	9.16	8.95	3	117.62	3.32	3.90	3	115.69	5.20	6.02	3	107.79	0.90	0.97	4	109.72	87.64	122.13	8.23	9.03
HFPO-DA	3	99.49	6.82	6.78	3	118.30	9.22	10.91	3	114.45	7.92	9.07	3	102.94	2.41	2.48	4	108.79	92.23	125.39	8.28	9.00
ADONA	3	93.84	10.90	10.23	3	110.96	8.87	9.84	3	115.30	15.80	18.22	3	84.20	0.58	0.49	4	101.07	83.75	136.20	14.42	14.58
PFMPA	3	102.51	6.00	6.15	3	115.14	4.00	4.60	3	109.17	7.34	8.02	3	98.33	1.61	1.58	4	106.29	95.42	120.45	6.96	7.40
PFMBA	3	99.75	13.71	13.68	3	112.01	3.47	3.89	3	107.39	6.53	7.01	3	99.39	1.04	1.03	4	104.63	84.43	116.45	5.87	6.15
NFDHA	3	98.28	14.25	14.01	3	106.93	2.01	2.15	3	105.54	10.45	11.03	3	107.22	26.73	28.66	4	104.49	82.88	138.81	4.02	4.20
9Cl-PF3ONS	3	96.42	15.11	14.57	3	113.13	8.39	9.50	3	117.74	18.24	21.47	3	89.55	1.19	1.07	4	104.21	82.45	142.53	12.86	13.40
11Cl-PF3OUdS	3	90.03	18.98	17.09	3	104.51	8.89	9.29	3	107.10	15.27	16.36	3	88.09	2.79	2.46	4	97.43	72.26	125.93	10.02	9.76
PFEESA	3	98.63	8.13	8.02	3	111.25	4.76	5.30	3	111.46	6.25	6.96	3	100.17	2.37	2.38	4	105.38	91.34	118.04	6.58	6.93
3:3 FTCA	3	99.19	6.69	6.63	3	115.21	4.41	5.09	3	109.09	5.70	6.22	3	95.19	1.59	1.51	4	104.67	91.75	119.89	8.73	9.14
5:3 FTCA	3	98.15	11.12	10.91	3	111.09	10.90	12.11	3	108.62	9.91	10.77	3	88.59	3.28	2.91	4	101.61	85.42	118.28	10.17	10.33
7:3 FTCA	3	99.22	13.75	13.64	3	113.36	12.70	14.40	3	109.10	13.12	14.32	3	97.59	3.74	3.65	4	104.82	83.50	122.44	7.28	7.64

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations based on detected values (not flagged at "U" or "B". Can range from 0-7)

(2) Mean sample mean percent recoveries for detected values values from Appendix D Individual Sample Tables. If n = 0 from Appendix D “NA” is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , ‘NA’ is reported.

(5) n= the number from all spike categories with percent recovery values

(6) mean of the mean percent recoveries with numerical values. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), ‘NA’ was is reported.

Table E-2. All Surface Water Sample Matrix Recovery Table

Data from Tables D11 - D13

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				ALL SPIKE RECOVERIES					
Analyte	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	3	96.42	7.15	6.90	3	112.00	5.18	5.80	3	104.47	9.35	9.77	3	104.29	91.46	118.49	7.47	7.79
PFPeA	3	104.76	11.11	11.64	3	118.14	8.85	10.46	3	113.85	6.65	7.58	3	112.25	97.58	130.04	6.09	6.83
PFHxA	1	93.37	NA	NA	2	103.28	3.85	3.97	3	104.94	7.92	8.31	3	100.53	93.37	114.37	6.22	6.25
PFHpA	2	88.55	3.99	3.54	3	114.31	8.99	10.27	3	101.29	18.72	18.96	3	101.39	81.06	125.56	12.70	12.88
PFOA	1	88.00	NA	NA	1	102.87	NA	NA	1	102.57	NA	NA	3	97.81	88.00	102.87	8.69	8.50
PFNA	3	98.58	11.45	11.29	3	112.32	8.91	10.01	3	108.96	10.36	11.29	3	106.62	90.69	123.85	6.72	7.16
PFDA	3	96.47	6.51	6.28	3	115.18	5.11	5.89	3	111.06	8.58	9.53	3	107.57	91.30	121.81	9.14	9.83
PFUnA	3	96.52	6.68	6.45	3	112.38	5.88	6.60	3	108.91	8.71	9.49	3	105.94	90.41	119.96	7.87	8.34
PFDoA	3	95.07	10.68	10.16	3	112.35	8.87	9.97	3	105.17	6.79	7.14	3	104.20	86.28	123.36	8.33	8.68
PFTrDA	3	98.28	11.38	11.19	3	116.26	6.86	7.98	3	109.29	8.23	8.99	3	107.95	87.19	124.54	8.40	9.07
PFTeDA	3	96.83	9.98	9.67	3	113.97	6.56	7.47	3	108.71	7.55	8.21	3	106.50	87.20	122.51	8.24	8.78
PFBS	1	94.37	NA	NA	3	109.33	11.47	12.54	3	110.02	5.35	5.88	3	104.58	94.37	123.82	8.46	8.84
PFPeS	1	95.13	NA	NA	3	110.00	12.63	13.89	3	107.19	4.47	4.79	3	104.11	95.13	125.46	7.59	7.90
PFHxS	1	97.34	NA	NA	1	111.63	NA	NA	1	111.89	NA	NA	3	106.96	97.34	111.89	7.79	8.33
PFHpS	3	96.10	12.80	12.30	3	111.78	8.97	10.02	3	106.54	12.31	13.11	3	104.81	84.70	123.33	7.62	7.98
PFOS	1	99.16	NA	NA	1	115.94	NA	NA	1	111.34	NA	NA	3	108.81	99.16	115.94	7.97	8.67
PFNS	3	96.10	19.29	18.54	3	104.38	16.55	17.27	3	110.63	6.70	7.42	3	103.70	76.88	123.16	7.03	7.29
PFDS	3	85.16	12.11	10.31	3	99.32	3.92	3.89	3	99.02	6.90	6.83	3	94.50	74.12	106.91	8.56	8.09
PFDoS	3	73.34	23.94	17.56	3	84.02	18.08	15.19	3	86.52	19.14	16.56	3	81.30	59.08	101.71	8.61	7.00
4:2 FTS	3	99.00	2.56	2.53	3	115.30	5.35	6.17	3	109.25	10.27	11.22	3	107.85	96.77	122.34	7.64	8.24
6:2 FTS	2	103.35	51.88	53.61	3	131.55	4.18	5.49	3	110.38	9.46	10.44	3	115.09	65.44	141.26	12.76	14.68
8:2 FTS	3	103.49	2.57	2.66	3	124.78	3.85	4.81	3	117.64	4.48	5.27	3	115.30	100.45	127.91	9.40	10.83
PFOSA	3	108.11	26.75	28.92	3	115.86	7.20	8.34	3	127.23	4.31	5.48	3	117.07	88.51	141.32	8.22	9.62
N-MeFOSA	3	87.12	9.16	7.98	3	111.82	5.93	6.63	3	106.13	5.58	5.92	3	101.69	79.01	118.48	12.72	12.94
N-EtFOSA	3	84.55	8.75	7.39	3	109.81	3.37	3.70	3	105.07	5.07	5.33	3	99.81	80.03	113.43	13.45	13.43
N-MeFOSAA	3	138.60	33.72	46.74	3	165.05	20.02	33.04	3	168.59	19.03	32.08	3	157.41	105.71	204.78	10.41	16.39
N-EtFOSAA	3	109.28	7.62	8.33	3	137.79	2.81	3.88	3	138.27	13.19	18.24	3	128.45	100.81	159.22	12.93	16.60
N-MeFOSE	3	91.99	9.08	8.35	3	111.33	6.32	7.04	3	107.47	8.20	8.82	3	103.60	86.52	119.46	9.88	10.24
N-EtFOSE	3	95.43	7.45	7.11	3	118.03	4.96	5.85	3	112.90	7.99	9.02	3	108.79	90.90	124.21	10.89	11.84
HFPO-DA	3	96.88	12.98	12.58	3	115.14	4.54	5.23	3	103.29	3.06	3.16	3	105.10	83.30	120.55	8.81	9.26
ADONA	3	92.07	5.66	5.21	3	109.46	2.40	2.63	3	106.04	9.86	10.46	3	102.52	86.93	117.64	8.99	9.21
PFMPA	3	97.08	7.42	7.21	3	112.61	9.37	10.56	3	102.46	10.91	11.18	3	104.05	92.90	124.26	7.58	7.89
PFMBA	3	91.82	6.88	6.32	3	108.45	9.09	9.86	3	103.95	8.39	8.72	3	101.41	85.76	119.53	8.48	8.60
NFDHA	3	99.19	23.67	23.47	3	109.14	5.24	5.72	3	113.28	20.40	23.11	3	107.20	79.02	139.28	6.76	7.24
9Cl-PF3ONS	3	91.51	13.85	12.67	3	108.92	3.97	4.32	3	109.51	11.24	12.31	3	103.31	77.46	123.13	9.90	10.22
11Cl-PF3OUdS	3	80.90	18.33	14.83	3	97.64	7.12	6.95	3	99.98	9.20	9.20	3	92.84	63.79	109.19	11.21	10.41
PFEESA	3	96.13	4.10	3.94	3	105.75	3.88	4.10	3	105.22	9.55	10.04	3	102.37	93.21	116.74	5.28	5.41
3:3 FTCA	3	96.48	8.56	8.26	3	109.15	9.90	10.81	3	103.28	8.77	9.06	3	102.97	87.55	120.65	6.16	6.34
5:3 FTCA	3	91.43	11.26	10.29	3	104.17	11.20	11.67	3	97.72	9.25	9.04	3	97.77	80.99	115.19	6.51	6.37
7:3 FTCA	3	85.65	19.80	16.96	3	100.43	12.17	12.22	3	100.49	12.80	12.86	3	95.52	74.95	114.27	8.95	8.55

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations based on detected values (not flagged at "U" or "B". Can range from 0-7)

(2) Mean sample mean percent recoveries for detected values values from Appendix D Individual Sample Tables. If n = 0 from Appendix D “NA” is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , ‘NA’ is reported.

(5) n= the number from all spike categories with percent recovery values

(6) mean of the mean percent recoveries with numerical values. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), ‘NA’ was is reported.

Table E-3: All Wastewater Sample Matrix Recovery Table

Data from Tables D-4 through D-10

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	7	95.99	5.76	5.53	7	97.19	11.35	11.04	7	99.46	6.88	6.85	3	97.55	86.44	118.89	1.81	1.77
PFPeA	6	102.31	7.80	7.98	7	102.66	11.44	11.75	7	104.79	9.03	9.46	3	103.25	89.30	125.80	1.30	1.34
PFHxA	4	94.84	8.69	8.24	5	98.19	13.94	13.69	5	101.02	10.81	10.93	3	98.02	83.88	114.33	3.16	3.09
PFHpA	6	97.15	3.97	3.85	7	98.15	9.34	9.17	7	98.37	8.20	8.06	3	97.89	89.40	115.32	0.66	0.65
PFOA	3	98.07	8.98	8.80	5	97.28	14.51	14.12	7	98.90	7.07	6.99	3	98.08	83.56	119.23	0.83	0.81
PFNA	7	96.86	9.36	9.07	7	98.30	11.70	11.50	7	100.78	7.66	7.72	3	98.65	79.75	120.61	2.01	1.98
PFDA	7	97.57	10.16	9.91	7	103.39	12.18	12.59	7	103.04	5.62	5.79	3	101.34	78.41	122.52	3.22	3.26
PFUnA	7	97.19	6.93	6.74	7	98.55	10.22	10.07	7	101.61	6.34	6.45	3	99.11	86.75	118.55	2.28	2.26
PFDoA	7	100.05	10.25	10.25	7	99.94	8.07	8.06	7	103.13	8.11	8.36	3	101.04	87.95	117.68	1.79	1.81
PFTrDA	7	106.21	14.47	15.36	7	109.73	14.81	16.26	7	109.44	12.81	14.02	3	108.46	91.69	136.24	1.80	1.95
PFTeDA	7	99.14	10.03	9.94	7	100.49	11.54	11.60	7	102.75	8.68	8.92	3	100.79	88.67	122.38	1.81	1.83
PFBS	4	102.57	6.17	6.33	6	104.05	9.07	9.43	7	106.36	4.53	4.82	3	104.32	93.97	120.06	1.83	1.91
PFPeS	7	95.66	5.05	4.83	7	98.58	9.10	8.97	7	100.99	7.47	7.55	3	98.41	88.86	118.01	2.71	2.67
PFHxS	6	109.03	7.80	8.51	7	106.88	9.19	9.82	7	107.78	7.62	8.22	3	107.89	95.26	125.40	1.00	1.08
PFHpS	7	104.02	13.97	14.53	7	105.28	14.32	15.07	7	105.53	13.81	14.58	3	104.94	84.43	132.05	0.77	0.81
PFOS	3	100.29	6.19	6.21	3	94.47	3.92	3.71	4	103.50	2.80	2.90	3	99.42	91.35	107.45	4.60	4.58
PFNS	7	91.53	9.66	8.84	7	96.36	7.69	7.41	7	100.03	6.10	6.10	3	95.97	76.66	107.60	4.45	4.27
PFDS	7	75.62	13.04	9.86	7	78.92	15.04	11.87	7	79.55	16.45	13.08	3	78.03	57.66	96.04	2.71	2.11
PFDoS	6	63.68	22.67	14.44	7	56.49	34.56	19.52	7	55.89	38.89	21.73	3	58.69	24.85	82.95	7.39	4.34
4:2 FTS	7	98.00	7.18	7.04	7	101.21	9.05	9.16	7	102.71	6.19	6.35	3	100.64	87.32	117.95	2.39	2.41
6:2 FTS	6	115.58	7.61	8.80	3	121.89	16.36	19.94	6	107.99	19.47	21.02	3	115.15	88.81	149.19	6.04	6.96
8:2 FTS	6	114.99	7.29	8.38	7	117.09	9.54	11.17	7	119.63	7.85	9.39	3	117.24	105.02	139.39	1.98	2.32
PFOSA	7	105.66	13.79	14.57	7	106.97	12.27	13.13	7	110.60	10.52	11.63	3	107.74	89.35	134.69	2.38	2.56
N-MeFOSA	7	91.09	7.19	6.55	7	97.64	10.09	9.85	7	105.04	12.00	12.61	3	97.92	79.36	132.85	7.13	6.98
N-EtFOSA	7	90.92	7.44	6.77	7	99.36	17.51	17.40	7	103.26	8.54	8.82	3	97.85	80.51	135.08	6.45	6.31
N-MeFOSAA	7	157.25	49.03	77.10	7	185.75	50.15	93.16	7	187.62	59.05	110.78	3	176.88	85.62	411.90	9.62	17.02
N-EtFOSAA	7	132.34	48.25	63.86	7	147.76	36.07	53.29	7	158.24	33.34	52.75	3	146.11	54.83	260.82	8.91	13.02
N-MeFOSE	7	92.38	11.75	10.86	7	94.16	16.05	15.11	7	96.34	12.16	11.72	3	94.29	76.32	123.75	2.10	1.98
N-EtFOSE	7	98.74	12.99	12.82	7	102.44	15.66	16.05	7	103.05	10.72	11.05	3	101.41	85.23	135.86	2.30	2.33
HFPO-DA	7	98.27	6.72	6.60	7	97.96	10.88	10.66	7	99.82	7.29	7.28	3	98.68	84.47	116.41	1.01	1.00
ADONA	7	102.05	10.02	10.23	7	98.55	10.16	10.01	7	102.23	11.71	11.97	3	100.94	82.04	120.46	2.06	2.08
PFMPA	7	107.75	11.88	12.80	7	107.65	11.38	12.25	7	111.30	14.42	16.05	3	108.90	86.64	135.68	1.91	2.08
PFMBA	7	98.12	7.07	6.94	7	99.47	11.26	11.20	7	103.35	15.08	15.59	3	100.32	85.18	134.95	2.71	2.72
NFDHA	7	71.27	21.11	15.05	7	78.38	36.79	28.84	7	79.67	21.67	17.26	3	76.44	42.25	118.99	5.92	4.52
9Cl-PF3ONS	7	90.98	29.95	27.25	7	85.81	16.58	14.23	7	90.85	16.45	14.94	3	89.22	49.80	128.10	3.30	2.95
11Cl-PF3OUdS	7	75.55	36.64	27.68	7	74.08	26.06	19.30	7	75.24	25.38	19.09	3	74.96	30.92	116.46	1.03	0.78
PFEESA	7	101.70	8.84	8.99	7	101.82	9.73	9.90	7	104.90	6.86	7.19	3	102.81	85.34	116.25	1.77	1.82
3:3 FTCA	7	97.56	36.16	35.28	7	85.87	47.37	40.68	7	93.71	41.46	38.85	3	92.38	23.76	135.53	6.45	5.96
5:3 FTCA	7	113.43	29.55	33.52	7	107.55	24.99	26.87	7	110.13	25.52	28.10	3	110.37	68.65	163.17	2.67	2.95
7:3 FTCA	7	94.62	20.68	19.56	7	96.51	13.92	13.43	7	101.57	13.87	14.09	3	97.57	60.18	120.60	3.69	3.60

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations based on detected values (not flagged at "U" or "B". Can range from 0-7)

(2) Mean sample mean percent recoveries for detected values values from Appendix D Individual Sample Tables. If n = 0 from Appendix D “NA” is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , ‘NA’ is reported.

(5) n= the number from all spike categories with percent recovery values

(6) mean of the mean percent recoveries with numerical values. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), ‘NA’ was is reported.

Table E-4. All Landfill Leachate Sample Matrix Recovery Table

Data from Tables D14 - D16

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	1	102.77	NA	NA	2	104.83	5.66	5.93	2	107.38	0.65	0.70	3	104.99	100.64	109.03	2.20	2.31
PFPeA	0	NA	NA	NA	1	117.40	NA	NA	1	112.68	NA	NA	2	115.04	112.68	117.40	2.90	3.34
PFHxA	0	NA	NA	NA	0	NA	NA	NA	0	NA	NA	NA	0	NA	NA	NA	NA	NA
PFHpA	1	105.38	NA	NA	1	110.12	NA	NA	1	109.15	NA	NA	3	108.22	105.38	110.12	2.32	2.51
PFOA	1	104.30	NA	NA	1	107.14	NA	NA	1	107.86	NA	NA	3	106.43	104.30	107.86	1.77	1.88
PFNA	2	99.49	0.99	0.98	2	102.01	5.42	5.53	2	109.73	0.96	1.05	3	103.74	98.10	110.47	5.14	5.34
PFDA	2	108.63	1.00	1.09	2	112.81	0.56	0.63	2	111.43	1.27	1.42	3	110.96	107.86	113.26	1.92	2.13
PFUnA	3	98.61	3.59	3.54	3	108.31	4.01	4.35	3	112.04	4.71	5.28	3	106.32	94.76	115.21	6.52	6.93
PFDoA	2	103.35	3.76	3.89	3	105.80	6.53	6.91	3	119.81	7.66	9.17	3	109.65	97.83	129.86	8.10	8.88
PFTrDA	3	106.62	7.10	7.57	3	105.48	6.80	7.18	3	111.10	2.36	2.62	3	107.73	99.27	114.38	2.75	2.97
PFTeDA	3	114.22	7.62	8.70	3	112.32	2.62	2.94	3	110.21	2.54	2.80	3	112.25	104.22	120.07	1.79	2.00
PFBS	0	NA	NA	NA	0	NA	NA	NA	1	117.91	NA	NA	1	117.91	117.91	117.91	NA	NA
PFPeS	2	103.33	3.60	3.72	3	114.95	3.18	3.65	3	118.02	4.71	5.56	3	112.10	100.70	124.31	6.91	7.75
PFHxS	1	116.92	NA	NA	2	111.05	9.54	10.59	2	116.26	0.87	1.01	3	114.74	103.56	118.54	2.80	3.22
PFHpS	3	105.20	4.17	4.39	3	109.56	6.78	7.43	3	117.09	2.59	3.03	3	110.62	101.07	119.05	5.44	6.01
PFOS	1	107.10	NA	NA	1	116.85	NA	NA	2	120.95	2.82	3.41	3	114.97	107.10	123.36	6.19	7.11
PFNS	3	96.97	7.16	6.94	3	102.08	8.25	8.42	3	100.23	11.24	11.26	3	99.76	87.55	109.08	2.59	2.59
PFDS	3	79.07	13.42	10.61	3	78.72	25.77	20.29	3	80.30	17.97	14.43	3	79.36	55.96	94.89	1.05	0.83
PFDoS	3	58.08	30.88	17.93	3	51.22	52.24	26.75	3	55.38	44.40	24.59	3	54.89	22.91	77.60	6.30	3.46
4:2 FTS	3	108.46	0.72	0.78	3	108.81	1.10	1.20	3	110.28	1.55	1.71	3	109.19	107.75	112.09	0.89	0.97
6:2 FTS	2	108.78	4.41	4.80	2	105.98	5.51	5.84	3	107.74	2.93	3.15	3	107.50	101.85	112.18	1.32	1.42
8:2 FTS	3	120.76	4.07	4.91	3	118.00	4.34	5.12	3	127.03	3.97	5.05	3	121.93	114.29	132.49	3.79	4.62
PFOSA	2	140.27	38.36	53.81	2	112.15	8.14	9.13	3	113.20	5.22	5.90	3	121.87	102.22	178.31	13.08	15.94
N-MeFOSA	3	104.56	8.52	8.91	3	110.57	0.95	1.05	3	113.77	4.28	4.87	3	109.63	94.28	119.40	4.27	4.68
N-EtFOSA	3	102.33	9.80	10.03	3	109.73	4.77	5.23	3	113.21	3.27	3.70	3	108.43	91.92	116.52	5.13	5.56
N-MeFOSAA	2	153.95	61.14	94.13	2	135.95	36.46	49.56	2	131.09	22.48	29.47	3	140.33	87.39	220.51	8.58	12.05
N-EtFOSAA	2	121.69	39.49	48.05	2	123.00	13.76	16.93	2	125.99	6.97	8.78	3	123.56	87.72	155.67	1.78	2.20
N-MeFOSE	2	105.24	4.70	4.94	2	108.55	3.25	3.53	2	112.52	0.40	0.45	3	108.77	101.75	112.83	3.35	3.64
N-EtFOSE	2	107.36	2.61	2.81	2	113.66	3.38	3.84	2	115.91	0.31	0.36	3	112.31	105.37	116.38	3.95	4.43
HFPO-DA	3	110.28	4.76	5.25	3	112.37	4.70	5.28	3	108.80	0.44	0.48	3	110.48	106.90	118.41	1.62	1.79
ADONA	3	140.65	27.15	38.18	3	143.42	29.47	42.27	3	143.61	33.36	47.91	3	142.56	103.60	196.71	1.16	1.66
PFMPA	3	113.00	44.12	49.86	3	144.82	26.28	38.06	3	148.16	29.82	44.19	3	135.33	64.16	197.46	14.34	19.41
PFMBA	3	128.17	22.80	29.22	3	134.54	20.82	28.01	3	133.47	20.89	27.88	3	132.06	109.93	166.11	2.58	3.41
NFDHA	3	54.45	72.15	39.29	3	52.39	64.28	33.68	3	58.40	66.10	38.60	3	55.08	17.30	102.96	5.54	3.05
9Cl-PF3ONS	3	137.71	35.13	48.38	3	142.76	34.77	49.64	3	139.65	39.46	55.11	3	140.04	92.47	200.22	1.82	2.55
11Cl-PF3OUdS	3	107.84	27.25	29.39	3	110.82	17.43	19.32	3	109.76	25.96	28.50	3	109.47	82.93	141.74	1.38	1.51
PFEESA	3	143.49	32.84	47.13	3	168.44	47.02	79.20	3	175.21	49.61	86.93	3	162.38	105.31	274.32	10.29	16.71
3:3 FTCA	3	105.80	69.95	74.01	3	135.92	42.10	57.22	3	140.80	43.30	60.97	3	127.51	29.12	210.24	14.87	18.96
5:3 FTCA	2	158.69	43.69	69.33	2	168.35	42.77	72.00	2	167.01	43.99	73.47	3	164.68	109.67	219.25	3.18	5.23
7:3 FTCA	2	109.54	30.41	33.31	2	137.02	31.54	43.22	2	137.60	29.97	41.23	3	128.06	85.99	167.59	12.52	16.04

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations based on detected values (not flagged at "U" or "B". Can range from 0-7)

(2) Mean sample mean percent recoveries for detected values values from Appendix D Individual Sample Tables. If n = 0 from Appendix D “NA” is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , ‘NA’ is reported.

(5) n= the number from all spike categories with percent recovery values

(6) mean of the mean percent recoveries with numerical values. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), ‘NA’ was is reported.

Table E-5: Soil Sample Matrix Recovery Table

Data from Tables D-18 through D-23

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	7	106.26	1.60	1.70	7	106.60	2.27	2.42	7	105.63	0.86	0.91	3	106.16	101.56	108.65	0.46	0.49
PFPeA	7	107.08	5.33	5.71	7	104.95	5.75	6.04	7	109.70	1.09	1.20	3	107.25	97.74	115.34	2.22	2.38
PFHxA	7	99.83	3.12	3.12	7	101.28	3.91	3.96	7	101.13	1.84	1.86	3	100.75	95.71	106.25	0.79	0.80
PFHpA	7	100.90	3.38	3.42	7	101.06	3.09	3.12	7	102.30	3.55	3.63	3	101.42	96.88	107.24	0.75	0.76
PFOA	6	98.88	2.69	2.66	6	100.48	4.22	4.24	6	100.63	1.55	1.56	3	100.00	94.20	106.42	0.97	0.97
PFNA	7	102.90	3.65	3.76	7	103.94	4.96	5.15	7	103.64	1.76	1.83	3	103.50	98.27	110.54	0.52	0.54
PFDA	6	103.24	3.12	3.22	6	105.89	5.30	5.61	6	106.92	1.05	1.12	3	105.35	99.30	112.17	1.80	1.90
PFUnA	7	101.03	5.27	5.32	7	104.41	4.63	4.83	7	103.42	1.07	1.11	3	102.95	91.73	108.82	1.69	1.74
PFDoA	6	103.25	3.93	4.06	7	105.59	6.68	7.06	7	106.54	5.40	5.76	3	105.13	90.57	116.27	1.61	1.69
PFTrDA	7	111.91	3.45	3.87	7	114.71	1.51	1.73	7	111.14	4.20	4.67	3	112.59	105.58	119.32	1.67	1.88
PFTeDA	7	103.80	5.79	6.01	7	106.43	2.54	2.71	7	105.14	0.79	0.84	3	105.12	93.43	112.88	1.25	1.31
PFBS	7	104.18	3.02	3.14	7	106.39	3.16	3.36	7	106.44	1.96	2.09	3	105.67	99.21	109.65	1.22	1.29
PFPeS	7	103.61	1.79	1.85	7	103.39	3.33	3.44	7	104.10	3.67	3.82	3	103.70	98.11	110.76	0.35	0.37
PFHxS	7	107.30	1.93	2.07	7	110.53	2.82	3.12	7	110.14	2.47	2.72	3	109.32	104.73	114.88	1.61	1.76
PFHpS	7	97.71	1.40	1.37	7	99.74	3.95	3.94	7	99.82	1.98	1.98	3	99.09	94.00	103.28	1.21	1.20
PFOS	6	107.53	3.25	3.50	6	110.82	5.26	5.83	7	109.09	2.53	2.75	3	109.15	102.49	118.60	1.51	1.65
PFNS	7	101.50	9.23	9.37	7	99.83	4.78	4.77	7	101.09	7.20	7.28	3	100.81	88.02	111.46	0.87	0.87
PFDS	7	99.01	3.91	3.88	7	102.30	2.64	2.70	7	100.48	2.82	2.83	3	100.60	91.87	106.42	1.64	1.65
PFDoS	7	88.73	9.09	8.06	7	91.24	5.28	4.82	7	90.74	5.11	4.64	3	90.24	76.70	100.10	1.47	1.33
4:2 FTS	7	100.67	3.43	3.45	7	105.28	3.18	3.34	7	105.78	2.07	2.19	3	103.91	96.06	110.38	2.71	2.82
6:2 FTS	3	107.68	4.01	4.32	5	100.12	39.51	39.55	7	79.36	49.89	39.59	3	95.72	11.24	147.45	15.32	14.66
8:2 FTS	7	120.18	5.05	6.07	7	122.28	4.31	5.27	7	122.01	1.17	1.43	3	121.49	113.31	131.95	0.94	1.14
PFOSA	7	104.41	1.81	1.89	7	107.72	2.83	3.05	7	106.36	1.10	1.17	3	106.16	102.02	110.54	1.57	1.66
N-MeFOSA	7	108.96	3.41	3.72	7	109.36	1.75	1.91	7	108.35	1.63	1.77	3	108.89	102.26	113.86	0.47	0.51
N-EtFOSA	7	111.05	2.13	2.36	7	113.71	2.31	2.62	7	113.28	1.02	1.16	3	112.68	108.13	117.03	1.27	1.43
N-MeFOSAA	7	100.51	3.36	3.38	7	104.68	4.67	4.89	7	103.88	3.65	3.79	3	103.02	97.15	110.15	2.15	2.21
N-EtFOSAA	7	109.07	2.66	2.91	7	108.32	4.50	4.87	7	110.84	2.39	2.65	3	109.41	101.21	114.53	1.18	1.29
N-MeFOSE	7	105.40	1.56	1.65	7	109.35	2.29	2.51	7	108.52	0.78	0.84	3	107.76	104.07	112.51	1.94	2.09
N-EtFOSE	7	108.55	1.39	1.51	7	110.45	2.84	3.14	7	112.16	1.04	1.17	3	110.39	105.49	114.25	1.64	1.81
HFPO-DA	7	104.56	6.11	6.39	7	104.67	11.17	11.69	7	105.92	5.19	5.50	3	105.05	87.79	119.70	0.72	0.76
ADONA	7	106.41	9.23	9.82	7	106.44	8.34	8.88	7	99.31	8.55	8.49	3	104.05	91.91	123.47	3.95	4.11
PFMPA	7	113.46	2.88	3.26	7	110.57	4.44	4.91	7	101.56	2.21	2.24	3	108.53	98.34	116.95	5.72	6.21
PFMBA	7	117.07	3.54	4.15	7	113.53	4.55	5.17	7	100.74	5.02	5.05	3	110.44	90.68	122.27	7.78	8.59
NFDHA	7	95.39	20.38	19.44	7	91.40	14.58	13.32	7	104.15	12.21	12.72	3	96.98	70.63	125.78	6.73	6.52
9Cl-PF3ONS	7	114.11	8.59	9.81	7	115.90	8.57	9.94	7	104.89	9.33	9.79	3	111.64	96.26	130.43	5.29	5.91
11Cl-PF3OUdS	7	111.36	7.63	8.50	7	112.96	7.50	8.48	7	106.55	11.59	12.34	3	110.29	93.93	127.26	3.02	3.34
PFEESA	7	103.38	2.78	2.87	7	99.01	3.00	2.97	7	99.39	2.08	2.06	3	100.59	94.93	106.75	2.41	2.42
3:3 FTCA	7	108.56	2.81	3.05	7	106.52	4.76	5.08	7	97.82	4.44	4.34	3	104.30	92.31	112.86	5.47	5.70
5:3 FTCA	7	100.01	5.40	5.40	7	94.09	3.11	2.93	7	95.14	6.74	6.41	3	96.42	86.49	108.30	3.27	3.16
7:3 FTCA	7	100.20	4.08	4.09	7	97.76	2.68	2.62	7	99.98	5.88	5.88	3	99.32	90.65	108.69	1.36	1.35

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations based on detected values (not flagged at "U" or "B". Can range from 0-7)

(2) Mean sample mean percent recoveries for detected values values from Appendix D Individual Sample Tables. If n = 0 from Appendix D “NA” is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , ‘NA’ is reported.

(5) n= the number from all spike categories with percent recovery values

(6) mean of the mean percent recoveries with numerical values. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), ‘NA’ was is reported.

Table E-6. All Sediment Sample Matrix Recovery Table

Data from Tables D-24 through D-26

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	3	102.38	4.16	4.26	3	105.59	1.30	1.37	3	106.86	1.32	1.41	3	104.95	97.66	108.49	2.20	2.31
PFPeA	3	102.14	2.04	2.08	3	108.48	0.49	0.53	3	110.35	1.29	1.42	3	106.99	99.77	111.87	4.02	4.31
PFHxA	3	96.90	1.58	1.53	3	101.72	0.71	0.73	3	101.50	1.96	1.99	3	100.04	95.70	103.54	2.72	2.73
PFHpA	3	95.45	0.98	0.93	3	101.55	0.92	0.94	3	102.59	1.21	1.25	3	99.87	94.59	103.65	3.87	3.86
PFOA	3	98.70	4.37	4.31	3	102.38	1.88	1.92	3	103.03	3.59	3.70	3	101.37	95.06	106.25	2.30	2.34
PFNA	3	99.76	3.15	3.14	3	105.69	1.49	1.57	3	106.51	2.70	2.87	3	103.98	96.20	109.79	3.54	3.68
PFDA	3	102.81	4.34	4.46	3	104.36	3.30	3.44	3	107.20	1.23	1.32	3	104.79	99.54	108.54	2.13	2.23
PFUnA	3	98.62	1.94	1.92	3	103.41	2.01	2.08	3	107.49	1.18	1.27	3	103.17	97.27	108.96	4.31	4.44
PFDoA	3	103.69	7.57	7.85	3	106.38	3.51	3.73	3	108.62	3.60	3.91	3	106.23	97.71	112.58	2.32	2.47
PFTrDA	3	110.91	5.55	6.16	3	111.76	0.66	0.73	3	111.60	1.08	1.20	3	111.43	103.81	114.54	0.40	0.45
PFTeDA	3	101.23	3.18	3.22	3	107.67	0.56	0.60	3	105.93	0.64	0.68	3	104.94	97.55	108.32	3.17	3.33
PFBS	3	102.05	3.38	3.45	3	104.43	2.17	2.26	3	108.39	1.36	1.48	3	104.95	98.18	109.49	3.05	3.20
PFPeS	3	100.03	0.95	0.95	3	105.59	1.09	1.15	3	105.12	1.84	1.94	3	103.58	99.07	106.86	2.98	3.09
PFHxS	3	109.49	1.29	1.42	3	114.99	3.20	3.68	3	114.78	5.02	5.76	3	113.08	107.99	120.66	2.75	3.11
PFHpS	3	94.82	3.68	3.49	3	98.16	2.15	2.11	3	99.82	3.18	3.17	3	97.60	91.69	103.33	2.61	2.55
PFOS	2	105.38	0.19	0.20	3	110.68	0.89	0.99	3	112.73	1.91	2.15	3	109.60	105.24	115.21	3.46	3.79
PFNS	3	93.98	7.37	6.93	3	96.67	1.46	1.41	3	111.85	1.15	1.29	3	100.83	87.99	112.69	9.56	9.64
PFDS	3	95.77	2.22	2.12	3	99.55	1.01	1.01	3	104.72	5.14	5.38	3	100.01	94.39	110.62	4.49	4.49
PFDoS	3	91.45	1.61	1.48	3	94.65	1.93	1.83	3	94.54	5.39	5.09	3	93.55	90.12	100.42	1.94	1.81
4:2 FTS	3	100.05	3.06	3.06	3	103.14	2.06	2.13	3	105.10	2.24	2.35	3	102.77	96.74	106.56	2.48	2.54
6:2 FTS	3	90.58	13.64	12.36	3	105.78	18.31	19.37	3	103.41	2.51	2.59	3	99.92	76.33	126.38	8.18	8.18
8:2 FTS	3	114.33	3.51	4.02	3	121.46	3.98	4.83	3	124.33	4.37	5.43	3	120.04	110.16	128.13	4.29	5.15
PFOSA	3	102.40	3.59	3.68	3	107.89	1.53	1.65	3	108.81	1.13	1.23	3	106.36	98.23	110.21	3.26	3.46
N-MeFOSA	3	104.84	0.69	0.72	3	109.61	1.15	1.26	3	108.85	2.79	3.03	3	107.77	104.33	111.23	2.38	2.56
N-EtFOSA	3	107.12	3.58	3.84	3	113.52	1.92	2.17	3	110.54	3.15	3.48	3	110.39	102.71	116.03	2.90	3.21
N-MeFOSAA	3	93.72	3.81	3.57	3	99.30	4.83	4.80	3	105.40	1.72	1.82	3	99.47	90.89	107.50	5.88	5.85
N-EtFOSAA	3	106.48	7.24	7.71	3	107.42	5.92	6.36	3	107.08	1.19	1.27	3	106.99	99.90	114.96	0.44	0.47
N-MeFOSE	3	103.05	7.49	7.72	3	109.69	4.65	5.10	3	111.85	4.58	5.12	3	108.20	96.00	117.68	4.24	4.59
N-EtFOSE	3	105.67	3.65	3.86	3	111.39	0.71	0.79	3	113.36	0.62	0.70	3	110.14	101.32	114.17	3.63	4.00
HFPO-DA	3	99.30	9.36	9.30	3	117.22	7.61	8.92	3	103.31	3.08	3.18	3	106.61	90.26	127.30	8.82	9.40
ADONA	3	103.76	13.37	13.87	3	113.33	4.15	4.70	3	102.95	1.63	1.68	3	106.68	93.00	119.42	5.41	5.77
PFMPA	3	96.40	2.15	2.07	3	96.76	0.66	0.63	3	99.70	0.91	0.90	3	97.62	94.01	100.40	1.85	1.81
PFMBA	3	99.61	1.73	1.73	3	96.31	3.97	3.82	3	103.07	0.75	0.77	3	99.66	93.43	103.75	3.39	3.38
NFDHA	3	81.47	14.15	11.53	3	101.86	15.27	15.56	3	100.02	7.25	7.26	3	94.45	68.24	115.78	11.94	11.28
9Cl-PF3ONS	3	111.38	12.69	14.13	3	120.04	4.02	4.83	3	110.53	1.46	1.61	3	113.98	101.31	127.54	4.62	5.26
11Cl-PF3OUdS	3	114.01	13.10	14.93	3	122.86	5.25	6.45	3	112.50	2.36	2.65	3	116.46	102.35	130.84	4.81	5.60
PFEESA	3	94.98	5.57	5.29	3	99.09	3.13	3.10	3	103.13	0.76	0.78	3	99.06	91.46	103.84	4.11	4.07
3:3 FTCA	3	93.58	2.65	2.48	3	93.58	4.03	3.77	3	94.86	6.26	5.94	3	94.01	88.45	100.16	0.79	0.74
5:3 FTCA	3	96.37	15.82	15.25	3	93.31	11.59	10.81	3	94.03	13.12	12.33	3	94.57	78.79	106.04	1.69	1.60
7:3 FTCA	3	109.23	5.19	5.67	3	105.72	4.02	4.25	3	103.58	7.73	8.01	3	106.18	94.50	113.62	2.69	2.85

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations based on detected values (not flagged at "U" or "B". Can range from 0-7)

(2) Mean sample mean percent recoveries for detected values values from Appendix D Individual Sample Tables. If n = 0 from Appendix D “NA” is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , ‘NA’ is reported.

(5) n= the number from all spike categories with percent recovery values

(6) mean of the mean percent recoveries with numerical values. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), ‘NA’ was is reported.

Table E-7. All Biosolid Sample Matrix Recovery Table

Data from Tables D27 - D29

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	3	67.94	41.75	28.36	3	103.59	6.65	6.89	3	98.49	5.45	5.37	3	90.01	36.96	109.20	21.42	19.28
PFPeA	3	92.64	2.95	2.73	3	114.75	5.04	5.78	3	110.59	3.50	3.87	3	105.99	89.97	120.88	11.08	11.75
PFHxA	3	84.03	3.13	2.63	3	103.85	1.60	1.66	3	98.57	5.32	5.25	3	95.48	81.01	105.37	10.75	10.27
PFHpA	3	87.30	4.03	3.52	3	99.24	7.18	7.13	3	98.63	4.28	4.22	3	95.06	83.64	103.44	7.07	6.73
PFOA	3	83.24	2.04	1.70	3	101.63	3.41	3.46	3	99.36	2.88	2.86	3	94.74	81.40	105.60	10.58	10.02
PFNA	3	88.77	3.82	3.39	3	104.84	1.87	1.96	3	103.21	2.89	2.99	3	98.94	86.54	107.09	8.94	8.84
PFDA	3	82.18	12.06	9.91	3	103.98	5.28	5.49	3	102.70	6.72	6.90	3	96.29	70.74	109.69	12.70	12.23
PFUnA	3	89.01	2.71	2.41	3	104.56	7.10	7.43	3	103.12	1.26	1.30	3	98.90	87.30	110.11	8.69	8.59
PFDoA	3	89.86	13.28	11.94	3	122.84	24.14	29.65	3	116.71	7.40	8.64	3	109.80	78.21	156.18	15.98	17.54
PFTrDA	3	103.88	12.80	13.29	3	139.24	16.62	23.13	3	139.58	22.17	30.95	3	127.57	88.91	174.79	16.08	20.51
PFTeDA	3	85.76	6.40	5.49	3	104.48	0.29	0.31	3	106.54	0.98	1.05	3	98.93	81.34	107.42	11.58	11.45
PFBS	3	89.10	32.55	29.00	3	98.65	25.44	25.10	3	86.38	23.36	20.18	3	91.38	55.94	117.74	7.05	6.44
PFPeS	3	111.06	25.20	27.98	3	119.74	16.88	20.21	3	111.69	11.32	12.65	3	114.16	89.24	143.01	4.24	4.84
PFHxS	2	107.44	11.62	12.48	2	124.69	10.59	13.20	2	115.89	8.13	9.42	3	116.01	98.61	134.03	7.44	8.63
PFHpS	3	117.24	45.57	53.43	3	125.99	34.44	43.39	3	105.51	15.27	16.11	3	116.25	81.92	178.70	8.84	10.28
PFOS	1	96.23	NA	NA	1	109.40	NA	NA	2	143.41	34.83	49.95	3	116.34	96.23	178.73	20.92	24.34
PFNS	3	82.46	5.84	4.81	3	100.29	6.19	6.20	3	100.86	3.24	3.27	3	94.53	77.83	105.59	11.07	10.46
PFDS	3	81.72	35.31	28.85	3	97.45	21.66	21.10	3	84.76	14.02	11.88	3	87.98	56.78	115.12	9.48	8.34
PFDoS	3	43.24	26.46	11.44	3	41.99	36.71	15.41	3	36.63	26.70	9.78	3	40.62	29.11	59.34	8.65	3.51
4:2 FTS	3	88.61	3.82	3.38	3	104.28	4.27	4.46	3	103.15	1.00	1.03	3	98.68	84.77	109.06	8.86	8.74
6:2 FTS	2	74.25	42.00	31.18	3	86.97	26.13	22.72	3	89.96	24.11	21.69	3	83.73	52.20	111.45	9.96	8.34
8:2 FTS	3	108.32	7.53	8.15	3	131.67	1.69	2.22	3	124.25	1.68	2.09	3	121.42	102.08	134.24	9.83	11.93
PFOSA	3	90.64	4.44	4.03	3	107.77	3.75	4.04	3	106.04	1.88	1.99	3	101.48	86.03	112.23	9.30	9.43
N-MeFOSA	3	96.58	5.36	5.17	3	112.48	4.55	5.12	3	109.69	4.11	4.51	3	106.25	92.03	117.92	7.99	8.49
N-EtFOSA	3	99.24	5.45	5.41	3	117.76	3.92	4.61	3	112.64	3.11	3.50	3	109.88	94.72	121.50	8.70	9.56
N-MeFOSAA	2	88.04	2.34	2.06	2	108.42	2.26	2.45	3	109.01	6.54	7.12	3	101.82	86.58	117.02	11.73	11.94
N-EtFOSAA	2	93.56	7.72	7.22	3	112.25	2.86	3.22	3	109.34	4.69	5.12	3	105.05	88.45	115.26	9.57	10.06
N-MeFOSE	3	105.33	18.84	19.84	3	113.05	6.67	7.54	3	109.90	2.92	3.21	3	109.42	93.73	128.23	3.55	3.88
N-EtFOSE	3	94.52	13.74	12.99	3	117.24	17.59	20.62	3	115.86	14.55	16.86	3	109.21	79.56	137.44	11.67	12.74
HFPO-DA	3	87.56	6.01	5.26	3	122.86	13.59	16.70	3	113.85	20.42	23.24	3	108.09	81.76	141.72	16.97	18.34
ADONA	3	92.71	4.91	4.56	3	117.62	1.08	1.27	3	111.30	12.09	13.45	3	107.21	87.69	126.07	12.08	12.95
PFMPA	3	84.93	4.07	3.45	3	34.97	54.58	19.08	3	36.68	73.68	27.03	3	52.19	16.69	88.82	54.34	28.36
PFMBA	3	87.82	1.89	1.66	3	117.77	21.57	25.40	3	115.09	17.26	19.87	3	106.90	86.22	146.51	15.50	16.57
NFDHA	3	58.06	59.21	34.38	3	74.32	42.50	31.59	3	69.50	40.90	28.42	3	67.30	28.99	110.24	12.41	8.35
9Cl-PF3ONS	3	93.25	8.03	7.49	3	120.49	4.67	5.63	3	114.80	10.14	11.64	3	109.51	86.25	128.01	13.12	14.37
11Cl-PF3OUdS	3	74.21	16.79	12.46	3	108.51	13.77	14.94	3	102.15	5.25	5.37	3	94.96	61.55	125.54	19.21	18.25
PFEESA	3	91.89	2.81	2.58	3	106.41	5.32	5.66	3	104.18	11.47	11.95	3	100.83	89.03	117.98	7.76	7.82
3:3 FTCA	3	80.49	10.11	8.14	3	29.53	68.95	20.36	3	29.33	89.49	26.25	3	46.45	10.06	89.65	63.46	29.48
5:3 FTCA	3	95.31	22.73	21.67	3	116.97	14.22	16.63	3	103.50	13.46	13.93	3	105.26	82.44	129.98	10.39	10.94
7:3 FTCA	3	82.21	14.81	12.18	3	115.73	11.16	12.92	3	110.90	20.58	22.82	3	102.95	70.17	135.71	17.60	18.12

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations based on detected values (not flagged at "U" or "B". Can range from 0-7)

(2) Mean sample mean percent recoveries for detected values values from Appendix D Individual Sample Tables. If n = 0 from Appendix D “NA” is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , ‘NA’ is reported.

(5) n= the number from all spike categories with percent recovery values

(6) mean of the mean percent recoveries with numerical values. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), ‘NA’ was is reported.

Table E-8. All Tissue Sample Matrix Recovery Table

Data from Tables D30 - D32

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	3	108.10	1.04	1.13	3	106.63	0.45	0.48	3	100.49	0.84	0.84	3	105.07	99.59	109.29	3.84	4.03
PFPeA	3	112.19	4.96	5.56	3	113.60	1.38	1.57	3	106.31	1.43	1.52	3	110.70	104.80	117.77	3.49	3.87
PFHxA	3	103.96	5.59	5.81	3	104.22	3.43	3.58	3	95.25	3.05	2.90	3	101.14	92.12	110.37	5.05	5.10
PFHpA	3	112.71	5.30	5.98	3	107.02	1.61	1.72	3	105.00	5.98	6.28	3	108.24	97.85	116.37	3.69	4.00
PFOA	3	108.79	2.57	2.80	3	105.32	2.53	2.66	3	99.78	1.60	1.60	3	104.63	97.94	111.61	4.34	4.54
PFNA	3	109.57	3.28	3.60	3	107.36	4.08	4.39	3	100.74	3.37	3.39	3	105.89	98.76	113.28	4.34	4.60
PFDA	3	105.43	3.49	3.68	3	106.81	2.20	2.35	3	99.46	2.90	2.88	3	103.90	96.51	108.90	3.76	3.91
PFUnA	3	107.43	0.50	0.54	3	106.07	1.09	1.16	3	98.73	3.11	3.07	3	104.08	95.59	107.92	4.50	4.68
PFDoA	3	123.86	11.70	14.50	3	112.46	1.46	1.64	3	102.19	6.50	6.65	3	112.84	94.53	139.53	9.61	10.84
PFTrDA	3	128.85	15.79	20.35	3	120.45	12.17	14.66	3	106.50	5.54	5.90	3	118.60	99.83	146.96	9.52	11.29
PFTeDA	3	109.77	2.29	2.51	3	106.98	0.95	1.02	3	100.37	0.39	0.39	3	105.71	99.92	112.02	4.57	4.83
PFBS	3	110.64	1.81	2.00	3	108.45	0.91	0.99	3	102.66	0.59	0.60	3	107.25	102.06	112.44	3.84	4.12
PFPeS	3	97.58	9.30	9.08	3	98.59	1.82	1.79	3	92.87	5.42	5.04	3	96.35	87.06	106.02	3.17	3.05
PFHxS	3	125.70	20.02	25.17	3	121.32	10.99	13.34	3	111.87	9.79	10.95	3	119.63	100.43	150.76	5.91	7.07
PFHpS	3	102.97	3.47	3.58	3	102.34	2.69	2.75	3	95.64	1.88	1.80	3	100.32	94.23	106.84	4.05	4.06
PFOS	2	108.38	1.73	1.87	2	110.11	3.66	4.03	2	99.79	0.65	0.65	3	106.10	99.33	112.96	5.21	5.53
PFNS	3	91.86	23.91	21.96	3	95.57	16.74	16.00	3	86.05	12.92	11.12	3	91.16	74.07	116.79	5.26	4.80
PFDS	3	88.00	15.37	13.52	3	89.29	16.26	14.52	3	81.59	8.86	7.23	3	86.29	72.44	97.81	4.77	4.12
PFDoS	3	73.08	40.91	29.89	3	63.32	34.34	21.75	3	51.97	52.18	27.12	3	62.79	33.41	94.40	16.83	10.56
4:2 FTS	3	109.72	1.27	1.40	3	104.17	1.66	1.73	3	99.44	2.04	2.03	3	104.44	97.10	110.96	4.92	5.14
6:2 FTS	2	91.39	41.20	37.65	2	95.20	26.19	24.94	3	68.28	66.86	45.65	3	84.96	17.62	118.02	17.15	14.57
8:2 FTS	3	123.59	1.95	2.41	3	127.39	2.39	3.04	3	117.41	2.77	3.25	3	122.80	114.11	130.88	4.10	5.04
PFOSA	3	112.08	1.23	1.37	3	110.88	1.01	1.12	3	103.92	0.76	0.79	3	108.96	103.39	113.28	4.04	4.40
N-MeFOSA	3	118.70	6.59	7.82	3	109.77	14.23	15.62	3	117.10	9.65	11.30	3	115.19	98.43	130.07	4.13	4.76
N-EtFOSA	3	122.20	11.09	13.55	3	126.30	15.92	20.11	3	108.56	10.82	11.74	3	119.02	95.03	148.61	7.80	9.29
N-MeFOSAA	3	110.37	5.11	5.64	3	111.07	3.55	3.94	3	101.85	3.18	3.24	3	107.77	99.25	116.60	4.76	5.13
N-EtFOSAA	3	118.60	5.38	6.37	3	113.46	3.28	3.72	3	109.50	6.42	7.03	3	113.85	104.05	125.92	4.01	4.56
N-MeFOSE	3	152.11	17.63	26.81	3	149.22	21.58	32.21	3	120.37	11.38	13.70	3	140.57	104.63	186.40	12.48	17.55
N-EtFOSE	3	134.58	41.00	55.17	3	216.70	50.87	110.24	3	136.82	50.47	69.05	3	162.70	89.20	342.83	28.75	46.78
HFPO-DA	3	107.93	14.85	16.02	3	106.65	2.96	3.16	3	95.93	5.76	5.53	3	103.51	89.50	118.48	6.37	6.59
ADONA	3	134.47	12.73	17.12	3	116.00	6.37	7.39	3	105.40	2.42	2.55	3	118.62	102.47	151.24	12.40	14.71
PFMPA	3	120.08	16.96	20.37	3	119.32	16.07	19.18	3	112.46	16.47	18.52	3	117.29	97.97	143.51	3.58	4.20
PFMBA	3	108.90	7.79	8.48	3	110.79	4.07	4.51	3	103.36	0.48	0.49	3	107.68	99.78	116.56	3.59	3.86
NFDHA	3	86.83	24.44	21.22	3	65.07	19.52	12.70	3	66.13	20.73	13.71	3	72.67	50.31	111.28	16.89	12.27
9Cl-PF3ONS	3	142.37	12.49	17.79	3	128.71	15.63	20.12	3	117.66	15.60	18.36	3	129.58	101.85	160.19	9.55	12.38
11Cl-PF3OUdS	3	136.96	24.39	33.40	3	124.41	30.07	37.41	3	115.07	25.66	29.53	3	125.48	83.01	158.53	8.75	10.99
PFEESA	3	122.87	12.32	15.14	3	117.29	14.51	17.02	3	107.93	10.90	11.76	3	116.03	94.47	136.93	6.50	7.55
3:3 FTCA	3	91.61	20.68	18.95	3	93.66	26.36	24.69	3	91.19	11.99	10.94	3	92.16	68.70	118.06	1.43	1.32
5:3 FTCA	3	182.95	25.36	46.41	3	176.82	20.34	35.97	3	150.83	20.40	30.77	3	170.20	129.70	233.54	10.02	17.06
7:3 FTCA	3	195.59	39.96	78.16	3	208.52	36.76	76.64	3	179.18	38.37	68.76	3	194.43	114.19	276.83	7.56	14.70

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations based on detected values (not flagged at "U" or "B". Can range from 0-7)

(2) Mean sample mean percent recoveries for detected values values from Appendix D Individual Sample Tables. If n = 0 from Appendix D “NA” is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , ‘NA’ is reported.

(5) n= the number from all spike categories with percent recovery values

(6) mean of the mean percent recoveries with numerical values. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples’ percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), ‘NA’ was is reported.

Table E-9. Combined Results for All Aqueous Media Matrix Spike Recovery

Data from Tables E1 - E4

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				High 2 Spike Recovery (groundwater only)				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	4	98.92	3.31	3.27	4	106.59	6.71	7.15	4	104.88	3.77	3.95	1	99.67	NA	NA	4	102.51	86.44	118.89	3.70	3.80
PFPeA	4	103.53	1.67	1.73	4	113.43	6.41	7.27	4	111.54	4.11	4.58	1	104.95	NA	NA	4	108.36	89.30	130.04	4.48	4.86
PFHxA	4	94.11	1.10	1.04	4	100.74	3.57	3.60	4	102.98	2.69	2.77	1	98.68	NA	NA	4	99.13	83.88	114.37	3.81	3.78
PFHpA	4	97.03	8.67	8.41	4	107.63	6.37	6.85	4	104.10	4.91	5.11	1	98.99	NA	NA	4	101.94	81.06	125.56	4.73	4.82
PFOA	4	96.79	8.50	8.22	4	102.43	4.83	4.94	4	103.11	4.37	4.50	1	96.56	NA	NA	4	99.72	83.56	119.23	3.54	3.53
PFNA	4	98.24	1.12	1.10	4	105.14	5.91	6.22	4	106.98	3.89	4.17	1	98.96	NA	NA	4	102.33	79.75	123.85	4.28	4.38
PFDA	4	101.52	5.55	5.63	4	112.12	5.42	6.07	4	110.12	4.57	5.03	1	104.45	NA	NA	4	107.05	78.41	124.71	4.59	4.92
PFUnA	4	98.04	1.52	1.49	4	108.01	6.13	6.62	4	108.61	4.50	4.88	1	99.91	NA	NA	4	103.64	86.75	119.96	5.26	5.45
PFDoA	4	98.43	4.08	4.01	4	107.93	5.88	6.34	4	109.60	6.79	7.45	1	99.68	NA	NA	4	103.91	82.26	129.86	5.46	5.67
PFTrDA	4	102.85	4.08	4.20	4	112.19	4.98	5.59	4	111.12	2.25	2.50	1	93.57	NA	NA	4	104.93	87.19	136.24	8.24	8.65
PFTeDA	4	102.52	7.71	7.91	4	110.57	6.19	6.84	4	108.62	3.93	4.27	1	98.28	NA	NA	4	105.00	87.20	122.51	5.37	5.64
PFBS	4	99.36	4.41	4.38	4	108.89	4.26	4.63	4	111.57	4.33	4.83	1	104.02	NA	NA	4	105.96	93.97	123.82	5.09	5.40
PFPeS	4	97.06	4.35	4.22	4	111.22	8.66	9.63	4	109.87	6.73	7.40	1	93.01	NA	NA	4	102.79	88.86	125.46	8.88	9.12
PFHxS	4	107.76	9.14	9.85	4	109.85	2.36	2.59	4	111.98	3.79	4.24	1	106.72	NA	NA	4	109.08	95.26	125.40	2.14	2.33
PFHpS	4	99.32	6.40	6.36	4	111.28	4.95	5.51	4	111.58	5.75	6.42	1	92.27	NA	NA	4	103.61	84.43	132.05	9.14	9.47
PFOS	4	102.18	4.20	4.30	4	109.09	11.61	12.67	4	111.93	7.81	8.74	1	109.76	NA	NA	4	108.24	91.35	123.36	3.90	4.22
PFNS	4	98.30	7.39	7.26	4	104.99	8.36	8.78	4	107.83	9.05	9.75	1	99.50	NA	NA	4	102.66	76.66	130.47	4.40	4.52
PFDS	4	83.37	9.48	7.90	4	90.79	15.54	14.11	4	90.49	13.61	12.32	1	93.36	NA	NA	4	89.50	55.96	115.08	4.79	4.29
PFDoS	4	70.96	18.92	13.43	4	72.72	31.27	22.74	4	74.41	30.04	22.35	1	86.89	NA	NA	4	76.25	22.91	105.99	9.49	7.24
4:2 FTS	4	101.06	4.90	4.95	4	109.44	5.57	6.10	4	108.79	3.99	4.34	1	98.66	NA	NA	4	104.49	87.32	122.34	5.21	5.44
6:2 FTS	4	108.94	4.63	5.04	4	121.18	8.99	10.90	4	110.82	3.98	4.41	1	99.64	NA	NA	4	110.15	65.44	149.19	8.02	8.84
8:2 FTS	4	112.04	6.67	7.48	4	122.02	4.40	5.37	4	122.08	3.47	4.24	1	109.19	NA	NA	4	116.33	100.45	139.39	5.76	6.70
PFOSA	4	118.01	16.37	19.31	4	113.80	4.93	5.62	4	120.61	8.50	10.25	1	102.02	NA	NA	4	113.61	88.51	178.31	7.24	8.22
N-MeFOSA	4	92.71	8.72	8.08	4	107.16	6.05	6.48	4	108.89	3.72	4.05	1	98.07	NA	NA	4	101.71	73.25	132.85	7.52	7.65
N-EtFOSA	4	90.97	8.84	8.05	4	105.97	4.67	4.95	4	107.30	4.04	4.34	1	103.16	NA	NA	4	101.85	73.76	135.08	7.32	7.46
N-MeFOSAA	4	136.99	19.81	27.14	4	154.10	16.96	26.14	4	155.06	17.89	27.73	1	95.77	NA	NA	4	135.48	85.62	411.90	20.48	27.74
N-EtFOSAA	4	114.65	13.94	15.98	4	133.02	9.00	11.98	4	136.20	11.89	16.20	1	96.60	NA	NA	4	120.12	54.83	260.82	15.26	18.33
N-MeFOSE	4	96.51	6.38	6.16	4	106.76	8.05	8.60	4	107.09	7.02	7.52	1	105.31	NA	NA	4	103.92	76.32	123.75	4.81	5.00
N-EtFOSE	4	99.82	5.22	5.21	4	112.94	6.44	7.27	4	111.89	5.40	6.05	1	107.79	NA	NA	4	108.11	85.23	135.86	5.51	5.95
HFPO-DA	4	101.23	6.05	6.13	4	110.94	8.10	8.99	4	106.59	6.02	6.41	1	102.94	NA	NA	4	105.42	83.30	125.39	4.08	4.30
ADONA	4	107.15	21.23	22.75	4	115.60	16.75	19.36	4	116.80	16.01	18.70	1	84.20	NA	NA	4	105.94	82.04	196.71	14.27	15.11
PFMPA	4	105.08	6.51	6.84	4	120.06	14.00	16.80	4	117.77	17.50	20.61	1	98.33	NA	NA	4	110.31	64.16	197.46	9.38	10.35
PFMBA	4	104.47	15.48	16.17	4	113.62	13.13	14.91	4	112.04	12.85	14.40	1	99.39	NA	NA	4	107.38	84.43	166.11	6.20	6.66
NFDHA	4	80.80	27.01	21.82	4	86.71	30.94	26.83	4	89.22	28.11	25.08	1	107.22	NA	NA	4	90.99	17.30	139.28	12.51	11.38
9Cl-PF3ONS	4	104.16	21.60	22.50	4	112.66	20.76	23.39	4	114.44	17.67	20.23	1	89.55	NA	NA	4	105.20	49.80	200.22	10.80	11.36
11Cl-PF3OUdS	4	88.58	15.99	14.16	4	96.76	16.59	16.05	4	98.02	16.06	15.74	1	88.09	NA	NA	4	92.86	30.92	141.74	5.66	5.26
PFEESA	4	109.99	20.41	22.45	4	121.81	25.71	31.32	4	124.20	27.49	34.14	1	100.17	NA	NA	4	114.04	85.34	274.32	9.77	11.14
3:3 FTCA	4	99.76	4.19	4.18	4	111.54	18.47	20.60	4	111.72	18.26	20.40	1	95.19	NA	NA	4	104.55	23.76	210.24	8.02	8.38
5:3 FTCA	4	115.43	26.23	30.28	4	122.79	24.84	30.50	4	120.87	25.86	31.26	1	88.59	NA	NA	4	111.92	68.65	219.25	14.17	15.86
7:3 FTCA	4	97.26	10.22	9.94	4	111.83	16.34	18.27	4	112.19	15.48	17.37	1	97.59	NA	NA	4	104.72	60.18	167.59	8.05	8.42

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations from Tables E1 - E4.

(2) Mean sample mean percent recoveries for detected values values from Tables E1 - E4. If n = 0, "NA" is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , 'NA' is reported.

(5) n= the number from all spike categories with reported percent recovery values

(6) mean of the mean percent recoveries. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples' percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples' percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), 'NA' was is reported.

Table E-10. Combined Results for All Solid Media Matrix Spike Recovery

Data from Tables E5 - E7

Analyte	Low Spike Recovery				Medium Spike Recovery				High Spike Recovery				ALL SPIKE RECOVERIES					
	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (1)	% Mean Recovery (2)	RSD (3)	SD (4)	n = (5)	% Mean Recovery (6)	Lowest % Recovery (7)	Highest % Recovery (8)	RSD (9)	SD (4)
PFBA	3	92.19	22.88	21.09	3	105.26	1.46	1.53	3	103.66	4.36	4.52	3	100.37	36.96	109.20	7.10	7.13
PFPeA	3	100.62	7.29	7.34	3	109.39	4.53	4.96	3	110.21	0.42	0.46	3	106.74	89.97	120.88	4.98	5.32
PFHxA	3	93.58	8.98	8.41	3	102.29	1.35	1.38	3	100.40	1.59	1.60	3	98.76	81.01	106.25	4.64	4.58
PFHpA	3	94.55	7.24	6.85	3	100.62	1.21	1.21	3	101.17	2.18	2.21	3	98.78	83.64	107.24	3.72	3.67
PFOA	3	93.61	9.59	8.98	3	101.50	0.94	0.95	3	101.01	1.85	1.86	3	98.70	81.40	106.42	4.48	4.42
PFNA	3	97.14	7.64	7.42	3	104.82	0.83	0.87	3	104.45	1.72	1.79	3	102.14	86.54	110.54	4.24	4.33
PFDA	3	96.08	12.53	12.04	3	104.74	0.97	1.01	3	105.61	2.39	2.52	3	102.14	70.74	112.17	5.16	5.27
PFUnA	3	96.22	6.61	6.36	3	104.13	0.60	0.62	3	104.68	2.33	2.44	3	101.67	87.30	110.11	4.66	4.73
PFDoA	3	98.94	7.95	7.86	3	111.60	8.73	9.74	3	110.62	4.85	5.37	3	107.05	78.21	156.18	6.58	7.05
PFTrDA	3	108.90	4.02	4.38	3	121.90	12.37	15.08	3	120.77	13.49	16.29	3	117.19	88.91	174.79	6.14	7.20
PFTeDA	3	96.93	10.07	9.76	3	106.19	1.51	1.61	3	105.87	0.67	0.70	3	103.00	81.34	112.88	5.11	5.26
PFBS	3	98.44	8.29	8.16	3	103.16	3.90	4.02	3	100.40	12.14	12.18	3	100.67	55.94	117.74	2.35	2.37
PFPeS	3	104.90	5.36	5.63	3	109.57	8.10	8.87	3	106.97	3.85	4.12	3	107.15	89.24	143.01	2.19	2.34
PFHxS	3	108.08	1.13	1.23	3	116.73	6.20	7.24	3	113.60	2.68	3.05	3	112.80	98.61	134.03	3.89	4.38
PFHpS	3	103.26	11.81	12.20	3	107.96	14.48	15.63	3	101.72	3.23	3.28	3	104.31	81.92	178.70	3.12	3.25
PFOS	3	103.05	5.82	6.00	3	110.30	0.71	0.79	3	121.74	15.48	18.85	3	111.70	96.23	178.73	8.44	9.43
PFNS	3	92.64	10.35	9.59	3	98.93	1.99	1.97	3	104.60	6.00	6.28	3	98.72	77.83	112.69	6.06	5.98
PFDS	3	92.17	9.97	9.19	3	99.77	2.44	2.43	3	96.65	10.88	10.51	3	96.20	56.78	115.12	3.97	3.82
PFDoS	3	74.48	36.37	27.08	3	75.96	38.79	29.47	3	73.97	43.79	32.39	3	74.80	29.11	100.42	1.38	1.03
4:2 FTS	3	96.44	7.04	6.79	3	104.24	1.03	1.07	3	104.68	1.30	1.36	3	101.79	84.77	110.38	4.55	4.63
6:2 FTS	3	90.84	18.40	16.71	3	97.62	9.88	9.65	3	90.91	13.26	12.05	3	93.12	11.24	147.45	4.18	3.90
8:2 FTS	3	114.28	5.19	5.93	3	125.14	4.54	5.68	3	123.53	1.06	1.32	3	120.98	102.08	134.24	4.85	5.86
PFOSA	3	99.15	7.50	7.44	3	107.79	0.08	0.09	3	107.07	1.41	1.51	3	104.67	86.03	112.23	4.58	4.80
N-MeFOSA	3	103.46	6.09	6.31	3	110.48	1.57	1.73	3	108.97	0.62	0.68	3	107.64	92.03	117.92	3.43	3.69
N-EtFOSA	3	105.80	5.68	6.01	3	115.00	2.08	2.39	3	112.16	1.28	1.44	3	110.99	94.72	121.50	4.24	4.71
N-MeFOSAA	3	94.09	6.64	6.24	3	104.13	4.40	4.58	3	106.10	2.48	2.63	3	101.44	86.58	117.02	6.35	6.44
N-EtFOSAA	3	103.04	8.06	8.31	3	109.33	2.35	2.57	3	109.09	1.73	1.89	3	107.15	88.45	115.26	3.33	3.57
N-MeFOSE	3	104.59	1.27	1.33	3	110.70	1.84	2.04	3	110.09	1.52	1.67	3	108.46	93.73	128.23	3.10	3.36
N-EtFOSE	3	102.91	7.20	7.41	3	113.03	3.25	3.68	3	113.79	1.66	1.89	3	109.91	79.56	137.44	5.53	6.08
HFPO-DA	3	97.14	8.96	8.70	3	114.92	8.10	9.31	3	107.70	5.10	5.49	3	106.58	81.76	141.72	8.39	8.94
ADONA	3	100.96	7.20	7.27	3	112.46	5.02	5.64	3	104.52	5.88	6.14	3	105.98	87.69	126.07	5.56	5.89
PFMPA	3	98.26	14.61	14.36	3	80.77	49.85	40.26	3	79.31	46.56	36.93	3	86.11	16.69	116.95	12.25	10.55
PFMBA	3	101.50	14.49	14.71	3	109.20	10.41	11.37	3	106.30	7.25	7.70	3	105.67	86.22	146.51	3.68	3.89
NFDHA	3	78.31	24.09	18.86	3	89.19	15.58	13.90	3	91.23	20.75	18.93	3	86.24	28.99	125.78	8.05	6.95
9Cl-PF3ONS	3	106.25	10.67	11.34	3	118.81	2.13	2.53	3	110.07	4.52	4.97	3	111.71	86.25	130.43	5.76	6.44
11Cl-PF3OUdS	3	99.86	22.28	22.25	3	114.78	6.40	7.35	3	107.07	4.85	5.20	3	107.24	61.55	130.84	6.96	7.46
PFEESA	3	96.75	6.15	5.95	3	101.50	4.19	4.25	3	102.23	2.46	2.52	3	100.16	89.03	117.98	2.97	2.98
3:3 FTCA	3	94.21	14.91	14.05	3	76.54	53.86	41.23	3	74.01	52.32	38.72	3	81.59	10.06	112.86	13.49	11.01
5:3 FTCA	3	97.23	2.54	2.47	3	101.46	13.24	13.44	3	97.56	5.31	5.18	3	98.75	78.79	129.98	2.38	2.35
7:3 FTCA	3	97.21	14.15	13.76	3	106.41	8.46	9.00	3	104.82	5.31	5.56	3	102.81	70.17	135.71	4.78	4.91

Notes:

NA = not applicable

(1) n = the number of samples for a matrix, for spike concentrations from Tables E5 - E8.

(2) Mean sample mean percent recoveries for detected values values from Tables E5 - E8. If n = 0, "NA" is reported.

(3) Relative Standard Devisation (RSD) of the mean of sample mean percent recoveries. RSD is the standard deviation divided by the mean, times 100.

(4) Standard Deviation (SD) of the mean of sample mean percent recoveries. If the number of samples is < 2 , 'NA' is reported.

(5) n= the number from all spike categories with reported percent recovery values

(6) mean of the mean percent recoveries. If there are no numerical values, 'NA' is reported.

(7) The lowest percent recovery of the mean of the three subsamples' percent recoveries for the matrix from Appendix D

(8) The highest t percent recovery of the mean of the three subsamples' percent recoveries for the matrix from Appendix D

(9) Relative Standard Deviation (RSD) of the mean percent recoveries for all groundwater sample matrices and spike concentrations. If the number of samples is <2), 'NA' was is reported.

Appendix F

Extracted Internal Standards (EIS) Recovery

APPENDIX F. EXTRACTED INTERNAL STANDARD (EIS) SPIKE RECOVERY TABLES

This appendix presents 12 summary tables by media of the EIS spike recoveries. EIS spike recovery values indicate the extraction efficiency of the sample preparation and detection method for PFAS compounds. EIS summaries were generated for each of the eight environmental media: groundwater, wastewater, surface water, landfill leachate, soils, sediment, biosolids and tissue. For those individual media tables, the mean sample concentration, mean of the sample percent recoveries, and percent RSD for 24 isotopically labeled analog PFAS analytes as the EIS compounds are presented. The calculated mean of the sample mean percent recoveries, percent RSD, and SD of the sample mean percent recoveries for an analyte, for the matrix is also included, as well as the lowest and highest sample mean percent recovery for the matrix.

Summary tables by aqueous, solid, and tissue media are also presented. The tables show the mean of the matrix mean concentrations; mean of the matrix mean percent recoveries; and percent RSD, SD, and 2×SD of the matrix mean percent recoveries for the 24 EIS compounds. , for all aqueous matrices, all solid matrices (including biosolid, sediment, and soil, but excluding tissue), all solid matrices (including biosolid, sediment, soil, and tissue), and all media. IDA identified the lowest and highest sample mean percent recovery for the media type.

Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021). Briefly, the tables include the following information.

Heading: Sample ID

- Compound—Always 24 EIS compounds.
- Mean Concentration—Mean spike concentration of the EDD entries for a compound from a sample of a matrix in µg/kg (solid matrices) or ng/L (aqueous matrices).
- Number EIS Spikes (n=)—Number of EDD entries for the compound from a sample of a matrix. Can be 12 or 16.
- Mean Percent Recovery—Mean of the percent recovery entries in the EDD for a compound from a sample of a matrix.
- RSD—Percent RSD of the mean percent recoveries reported in the EDD. If the number of samples is less than two, then NA was used.

Heading: ALL EIS SPIKE RECOVERIES

- Mean Concentration—Mean of the sample mean spike concentrations from columns Mean Concentration.
- Number EIS Spikes (n=)—Number of samples for a matrix, for the compound. Always three.
- Mean Percent Recovery—Mean of the sample mean percent recoveries from columns Mean Percent Recovery.
- RSD—Percent RSD of the sample mean percent recoveries reported in the EDD. If the number of samples is less than two, then NA was used.

Table F-1 Groundwater Sample EIS Spike Recovery

Compound	GW CO				GW WL378				GW Wurtsmith AFB				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	76.4	16	88.93	8.08	78	16	95.73	2.8	79.2	16	97.21	2.66	77.9	3	93.96	4.7
Perfluoro-n-[13C5]pentanoic acid	38.2	16	94.29	2.33	39	16	94.61	4.46	39.6	16	96.24	4.21	38.9	3	95.05	1.1
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	19.1	16	93.82	4.2	19.5	17	91.56	3.55	19.8	19	93.44	3.08	19.5	3	92.94	1.3
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	19.1	16	92.88	5.64	19.5	16	91.98	4.51	19.8	16	92.88	3.85	19.5	3	92.58	0.56
Perfluoro-n-[13C8]octanoic acid	19.3	16	92.77	4.39	19.7	16	92.89	4.56	20	16	94.34	4.73	19.7	3	93.33	0.93
Perfluoro-n-[13C9]nonanoic acid	9.54	16	92.08	4.6	9.75	16	92.7	2.73	9.91	16	95.19	2.85	9.73	3	93.32	1.76
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	9.54	16	90.07	5.68	9.75	16	89.66	4.55	9.91	16	92.71	4.42	9.73	3	90.81	1.82
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	9.54	16	88.77	7.19	9.75	16	90.44	4.89	9.91	16	94.49	2.79	9.73	3	91.23	3.22
Perfluoro-n-[1,2-13C2]dodecanoic acid	9.54	16	80.44	10.36	9.75	16	81.43	7.95	9.91	16	86.74	5.58	9.73	3	82.87	4.09
Perfluoro-n-[1,2-13C2]tetradecanoic acid	9.54	16	81.59	12.05	9.75	16	82.84	8.61	9.91	16	83.94	9.2	9.73	3	82.79	1.42
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	19.1	16	99.66	3.9	19.5	16	96.86	4.32	19.8	16	99.38	5.25	19.5	3	98.63	1.57
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	19.3	16	92.72	3.71	19.7	16	92.1	3.22	20	16	94.62	3.16	19.7	3	93.15	1.41
Perfluoro-1-[13C8]octanesulfonic acid	19.3	16	94.88	4.39	19.7	16	94.84	5.2	19.9	18	96.04	4.19	19.6	3	95.25	0.72
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	38.4	16	106.74	9.86	39.2	16	104.95	10.89	39.8	16	110.37	25.69	39.1	3	107.35	2.57
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	38.2	16	108.62	7.26	39	16	75.83	31.27	39.6	16	102.78	32.75	38.9	3	95.74	18.27
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	38.2	16	102.27	8.73	39	16	76.86	29.38	39.6	16	100.54	31.39	38.9	3	93.22	15.23
Perfluoro-1-[13C8]octanesulfonamide	19.1	16	91.78	6.05	19.5	16	91.63	5.37	19.8	16	92.62	6.64	19.5	3	92.01	0.58
N-methyl-d3-perfluoro-1-octanesulfonamide	19.1	16	74.05	7.94	19.5	16	69.16	9.03	19.8	16	65.78	19.85	19.5	3	69.66	5.97
N-ethyl-d5-perfluoro-1-octanesulfonamide	19.1	16	70.18	11.5	19.5	16	67.17	10.72	19.8	16	67.34	17.93	19.5	3	68.23	2.48
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	38.2	16	88.36	6.43	39	16	82.19	9.75	39.6	16	102.02	7.1	38.9	3	90.86	11.17
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	38.2	16	86.99	7.58	39	16	80.51	13.69	39.6	16	103.85	6.65	38.9	3	90.45	13.32
N-methyl-d7-perfluorooctanesulfonamidoethanol	191	16	74.49	7.79	195	16	74.82	7.09	198	16	77.01	6.25	195	3	75.44	1.81
N-ethyl-d9-perfluorooctane sulfonamidoethanol	191	16	70.51	10.73	195	16	68.74	8.98	198	16	72.61	6.3	195	3	70.62	2.74
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	76.4	16	104.44	5.56	78	16	100.76	11.14	79.2	16	103	8.12	77.9	3	102.73	1.8

py_Log Date 8/11/2021 12:11:50 PM

Notes:
1. RSD = Relative Standard Deviation

Table F-2 Surface Water EIS Spike Recovery

Compound	SW COP-SW01 to 11				SW Marine Surface water				SW RP-SW01 to 11				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	77	12	95.72	3.72	73.1	12	66.66	3.59	80.1	12	93.65	2.19	76.7	3	85.34	19
Perfluoro-n-[13C5]pentanoic acid	38.5	12	96.68	3.99	36.5	12	96.44	2.67	40	13	94.39	6.4	38.3	3	95.84	1.31
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	19.2	12	92.75	4.29	18.3	12	93.79	4.31	20	13	91.22	4.1	19.2	3	92.59	1.4
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	19.2	12	90.09	5.32	18.3	12	90.06	5.21	20	12	91.13	5.16	19.2	3	90.43	0.68
Perfluoro-n-[13C8]octanoic acid	19.4	12	92.23	4.55	18.5	12	92.73	4.2	20.2	12	92.13	4.82	19.4	3	92.37	0.35
Perfluoro-n-[13C9]nonanoic acid	9.62	12	92.02	4.12	9.13	12	90.63	2.56	10	12	90.08	4.14	9.59	3	90.91	1.1
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	9.62	12	90.36	5.02	9.13	12	86.35	4.65	10	12	88.76	5.67	9.59	3	88.49	2.28
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	9.62	12	91.18	5.11	9.13	12	84	5.08	10	12	88.87	7.16	9.59	3	88.02	4.17
Perfluoro-n-[1,2-13C2]dodecanoic acid	9.62	12	79.82	5.61	9.13	12	75.15	6.38	10	12	76.98	10.98	9.59	3	77.31	3.04
Perfluoro-n-[1,2-13C2]tetradecanoic acid	9.62	12	56.15	7.64	9.13	12	72.92	6.33	10	12	75.76	7.95	9.59	3	68.28	15.52
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	19.2	12	95.32	3.76	18.3	12	96.21	2.87	20	12	95.69	4.83	19.2	3	95.74	0.46
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	19.4	12	91.62	4.19	18.5	12	92.73	2.88	20.2	12	90.65	4.5	19.4	3	91.67	1.14
Perfluoro-1-[13C8]octanesulfonic acid	19.4	12	93.95	4.03	18.5	12	90.83	4.51	20.4	13	93.11	3.66	19.4	3	92.63	1.75
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	38.6	12	119.75	7.52	36.7	12	108.32	4.42	40.2	12	108.82	12.68	38.5	3	112.3	5.75
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	38.5	12	92.41	5.26	36.5	12	112.5	5.31	40	12	88.88	19.1	38.4	3	97.93	13.01
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	38.5	12	87.61	5.06	36.5	12	97.62	2.65	40	12	87.17	19.79	38.4	3	90.8	6.51
Perfluoro-1-[13C8]octanesulfonamide	19.2	12	87.01	3.7	18.3	12	81.39	5.38	20	12	80.23	14.14	19.2	3	82.88	4.37
N-methyl-d3-perfluoro-1-octanesulfonamide	19.2	12	69.12	6.49	18.3	12	65.74	5.27	20	12	60.14	11.14	19.2	3	65	6.98
N-ethyl-d5-perfluoro-1-octanesulfonamide	19.2	12	66.03	7.96	18.3	12	63.18	5.24	20	12	60.62	9.53	19.2	3	63.28	4.28
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	38.5	12	86.68	5.13	36.5	12	80.85	4.59	40	12	80.12	16.2	38.4	3	82.55	4.35
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	38.5	12	87.85	4.85	36.5	12	78.23	2.68	40	12	78.52	18.79	38.4	3	81.54	6.71
N-methyl-d7-perfluorooctanesulfonamidoethanol	192	12	65.28	8.45	183	12	68	9.21	200	12	64.74	13.3	192	3	66.01	2.65
N-ethyl-d9-perfluorooctane sulfonamidoethanol	192	12	62.32	10.34	183	12	62.74	11.84	200	12	60.5	9.28	192	3	61.86	1.93
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	77	12	97.5	7.72	73.1	12	103.09	5.82	80.1	12	99.17	5.19	76.7	3	99.92	2.87

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-3 Wastewater Sample EIS Spike Recovery

Compound	WW #10				WW #3				WW #5			
	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	77.9	12	94.78	7.00	78.3	12	93.79	4.07	81.2	12	95.86	7.53
Perfluoro-n-[13C5]pentanoic acid	39.0	12	94.86	3.38	39.2	12	88.72	5.54	40.6	12	93.48	6.46
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	19.5	12	95.13	5.46	19.5	13	90.08	5.40	20.3	12	92.01	8
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	19.5	12	94.73	7.46	19.6	12	76.61	12.36	20.3	12	89.43	6.74
Perfluoro-n-[13C8]octanoic acid	19.7	12	95.05	5.24	19.8	12	87.27	7.88	20.5	12	93.43	7.36
Perfluoro-n-[13C9]nonanoic acid	9.74	12	93.91	5.02	9.79	12	82.37	10.62	10.1	12	93.79	7.56
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	9.74	12	90.98	4.52	9.79	12	70.56	18.37	10.1	12	91.08	7.53
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	9.74	12	88.88	4.82	9.79	12	56.25	24.58	10.1	12	90.15	8.32
Perfluoro-n-[1,2-13C2]dodecanoic acid	9.74	12	69.71	7.58	9.72	16	33.68	34.68	10.1	12	80.14	6.98
Perfluoro-n-[1,2-13C2]tetradecanoic acid	9.74	12	40.93	10.88	9.79	12	16.96	75.06	10.1	12	61.8	8.4
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	19.5	12	97.35	5.93	19.6	12	88.65	10.55	20.3	12	97.53	7.57
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	19.7	12	94.46	5.63	19.8	12	79.44	16.85	20.5	12	93.59	8.39
Perfluoro-1-[13C8]octanesulfonic acid	19.7	12	95.38	5.47	19.7	15	66.75	24.3	20.5	12	94.61	8.46
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	39.2	12	186.33	8.51	39.4	12	111.29	11.05	40.8	12	135.42	11.88
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	39.0	12	98.01	10.43	39.2	12	64.02	9.21	40.6	12	80.52	10.06
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	39.0	12	97.31	9.75	39.2	12	65.08	20.45	40.6	12	68.2	10.76
Perfluoro-1-[13C8]octanesulfonamide	19.5	12	83.02	8.63	19.6	12	63.88	30.79	20.3	12	58.8	36.48
N-methyl-d3-perfluoro-1-octanesulfonamide	19.5	12	61.63	6.95	19.6	12	16.57	112.65	20.3	12	49.78	31.8
N-ethyl-d5-perfluoro-1-octanesulfonamide	19.5	12	58.93	8.41	19.6	11	13.99	120.3	20.3	12	50.52	26.47
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	39.0	12	113.46	7.53	39.2	12	58.07	29.92	40.6	12	86.28	7.85
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	39.0	12	105.67	9.05	39.2	12	55.56	38.04	40.6	12	78.73	7.07
N-methyl-d7-perfluorooctanesulfonamidoethanol	195.0	12	63.43	7.71	196	12	27.15	55.33	203	12	50.38	35.57
N-ethyl-d9-perfluorooctane sulfonamidoethanol	195.0	12	57.98	4.74	196	12	19.88	70.86	203	12	50.95	26.32
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	77.9	12	102.97	8.37	78.4	13	95.53	4.74	81.2	12	100.19	10.17

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-3 Wastewater Sample EIS Spike Recovery

Compound	WW 3				WW #7				WW #8			
	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	78.2	12	87.67	4.73	79.8	12	87.36	3.22	78.9	12	89.88	15.08
Perfluoro-n-[13C5]pentanoic acid	39.1	12	92.53	6.72	39.9	12	103.4	4.95	39.5	12	89.83	15.69
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	19.5	12	94.02	6.58	20	21	88.84	6.08	19.7	12	88.54	14.22
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	19.5	12	93.88	3.82	20	12	78.73	4.07	19.7	12	88.02	13.47
Perfluoro-n-[13C8]octanoic acid	19.7	12	93.24	5.07	20.2	12	88.27	5.03	19.9	12	88.64	13.2
Perfluoro-n-[13C9]nonanoic acid	9.77	12	91.58	5.24	9.97	12	87.85	4.24	9.85	12	87.49	11.4
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	9.77	12	89.01	5.39	9.97	12	85.49	6.6	9.85	12	87.3	12.61
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	9.77	12	90.27	9.24	9.97	12	93.12	10.98	9.85	12	87.88	11.66
Perfluoro-n-[1,2-13C2]dodecanoic acid	9.77	12	73.74	12.19	9.97	20	71.45	17.56	9.85	12	77.87	9.58
Perfluoro-n-[1,2-13C2]tetradecanoic acid	9.77	12	50.21	8.74	9.99	21	153.44	38.43	9.85	12	77.69	6.86
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	19.5	12	96.69	3.97	20	12	72.28	5.69	19.7	12	93.32	8.64
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	19.7	12	92.07	5.31	20.2	12	88.88	4.85	19.9	12	89.04	7.78
Perfluoro-1-[13C8]octanesulfonic acid	19.7	13	91.78	6.74	20.2	17	80.35	7.35	19.9	12	90.48	7.35
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	39.3	12	174.25	7.34	40.1	12	166.5	4.79	39.6	12	105.8	15.45
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	39.1	12	92.14	9.85	39.9	12	119.58	6.51	39.5	12	102.45	15.68
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	39.1	12	81.87	13.81	39.9	12	92.91	5.35	39.5	12	96.17	13.24
Perfluoro-1-[13C8]octanesulfonamide	19.5	12	75.05	21.48	20	12	26.68	21.85	19.7	12	82.02	13.55
N-methyl-d3-perfluoro-1-octanesulfonamide	19.5	12	61.71	13.56	20	12	13.51	21.38	19.7	12	66.07	7.05
N-ethyl-d5-perfluoro-1-octanesulfonamide	19.5	12	57.92	10.39	20	12	11.93	19.88	19.7	12	67.31	5.52
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	39.1	12	93.48	9.46	39.9	12	21.46	37.39	39.5	12	90.54	11.73
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	39.1	12	105.33	10.69	39.9	12	11.68	32.91	39.5	12	86.58	12.05
N-methyl-d7-perfluorooctanesulfonamidoethanol	195	12	66.74	18.28	200	12	10.99	22.27	197	12	68.55	8.93
N-ethyl-d9-perfluorooctane sulfonamidoethanol	195	12	56.9	16.12	200	12	7.99	25.41	197	12	63.83	5.45
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	78.2	12	99.2	6.47	80	21	96.84	5.55	78.9	12	91.83	18.11

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-3 Wastewater Sample EIS Spike Recovery

Compound	WW #9				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	78.3	12	44.93	8.03	78.9	7	84.9	21.14
Perfluoro-n-[13C5]pentanoic acid	39.1	12	66.8	5.74	39.5	7	89.95	12.53
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	19.6	12	92.85	6.95	19.7	7	91.64	2.79
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	19.6	12	91.15	7.06	19.7	7	87.51	8.16
Perfluoro-n-[13C8]octanoic acid	19.8	12	94.78	6.34	19.9	7	91.53	3.64
Perfluoro-n-[13C9]nonanoic acid	9.78	12	93.39	7.72	9.87	7	90.05	4.82
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	9.78	12	92.23	7.62	9.87	7	86.66	8.63
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	9.78	12	89.54	9.41	9.87	7	85.16	15.09
Perfluoro-n-[1,2-13C2]dodecanoic acid	9.78	12	72.9	11.05	9.86	7	68.5	23.03
Perfluoro-n-[1,2-13C2]tetradecanoic acid	9.78	12	38.35	8.15	9.87	7	62.77	70.57
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	19.6	12	88.59	6.99	19.7	7	90.63	9.88
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	19.8	12	93.69	6.06	19.9	7	90.17	5.8
Perfluoro-1-[13C8]octanesulfonic acid	19.8	12	95.84	6.44	19.9	7	87.88	12.2
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	39.3	12	156.42	5.46	39.7	7	148	21.11
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	39.1	12	118	8.46	39.5	7	96.39	20.58
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	39.1	12	96.87	10.21	39.5	7	85.49	16.32
Perfluoro-1-[13C8]octanesulfonamide	19.6	12	82.33	13.83	19.7	7	67.4	30.19
N-methyl-d3-perfluoro-1-octanesulfonamide	19.6	12	60.89	12.08	19.7	7	47.17	47.74
N-ethyl-d5-perfluoro-1-octanesulfonamide	19.6	12	61.06	10.46	19.7	7	45.95	50.22
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	39.1	12	107.69	10.15	39.5	7	81.57	39.12
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	39.1	12	103.8	12.13	39.5	7	78.19	44.16
N-methyl-d7-perfluorooctanesulfonamidoethanol	196	12	52.88	17.59	197	7	48.59	44.8
N-ethyl-d9-perfluorooctane sulfonamidoethanol	196	12	51.12	12.35	197	7	44.09	48.4
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	78.3	12	104.93	11.53	79	7	98.78	4.53

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-4 Landfill Leachate Sample EIS Spike Recovery

Compound	LCH C & D Landfill Leachate				LCH MSW Incineration Ash Landfill Leachate				LCH MSW Landfill Leachate				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	346	12	86.52	8.47	319	12	94.66	1.68	333	12	8.99	54.14	333	3	63.39	74.59
Perfluoro-n-[13C5]pentanoic acid	173.0	12	78.39	12.36	160	12	94.98	7.66	166	12	38.88	38.95	166	3	70.75	40.74
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	86.6	12	96.9	10.75	79.7	15	93.03	3.96	83.3	12	72.87	7.22	83.2	3	87.6	14.73
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	86.6	12	93.5	9.04	79.8	12	92.38	4.82	83.3	12	81.27	4.79	83.2	3	89.05	7.59
Perfluoro-n-[13C8]octanoic acid	87.4	12	95.05	7.31	80.6	12	90.8	4.17	84.1	12	90.7	5.65	84	3	92.18	2.69
Perfluoro-n-[13C9]nonanoic acid	43.3	12	94.52	10.66	39.9	12	88.54	3.52	41.6	12	88.68	5.48	41.6	3	90.58	3.77
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	43.3	12	89.44	10.46	39.9	12	81.03	5.33	41.6	12	81.42	7.38	41.6	3	83.97	5.65
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	43.3	12	82.48	8.92	39.9	12	80.52	7.05	41.6	12	71.74	8	41.6	3	78.25	7.31
Perfluoro-n-[1,2-13C2]dodecanoic acid	43.3	12	64.38	11.3	39.9	12	69.58	7.28	41.6	12	47.23	11.69	41.6	3	60.39	19.36
Perfluoro-n-[1,2-13C2]tetradecanoic acid	43.3	12	47.01	12.86	39.9	12	57.62	7.03	41.6	12	22.38	23.53	41.6	3	42.33	42.71
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	86.6	12	95.91	10.22	79.8	12	97.86	4.29	83.3	12	73.88	9.65	83.2	3	89.22	14.92
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	87.4	12	94.78	7.59	80.6	12	91.82	2.79	84.1	12	93.08	6.42	84	3	93.22	1.6
Perfluoro-1-[13C8]octanesulfonic acid	87.4	12	95.74	8.24	80.6	12	86.33	5.44	84.1	12	86.54	6.67	84	3	89.54	6
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	174	12	199	8.13	161	12	107.92	6.24	167	12	80.57	24.52	167	3	129.16	48.01
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	173.0	12	111.9	9.95	160	12	98.85	8.2	166	12	182.5	12.9	166	3	131.08	34.33
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	173.0	12	80.15	9.2	160	12	92.81	6.5	166	12	139.33	15.52	166	3	104.1	29.94
Perfluoro-1-[13C8]octanesulfonamide	86.6	12	73.21	31.64	79.8	12	68.01	9.02	83.3	12	63.09	38.31	83.2	3	68.1	7.43
N-methyl-d3-perfluoro-1-octanesulfonamide	86.6	12	59.66	19.35	79.8	12	63.64	8.03	83.3	12	49.07	31.68	83.2	3	57.46	13.1
N-ethyl-d5-perfluoro-1-octanesulfonamide	86.6	12	56.95	14.57	79.8	12	64.37	8.51	83.3	12	47.56	30.13	83.2	3	56.29	14.96
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	173.0	12	96.77	7.16	160	12	82.72	2.97	166	12	104.65	7.91	166	3	94.71	11.73
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	173.0	12	93.67	8.84	160	12	81.15	3.11	166	12	104.04	8.72	166	3	92.96	12.33
N-methyl-d7-perfluorooctanesulfonamidoethanol	866.0	12	55.58	28.73	798	12	68.59	4.98	833	12	48.73	37.02	832	3	57.63	17.51
N-ethyl-d9-perfluorooctane sulfonamidoethanol	866.0	12	57.23	24.05	798	12	62.67	5.35	833	12	41.92	33.89	832	3	53.94	19.95
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	346	12	112.5	10.84	319	12	99.56	8.58	333	13	96.48	19.08	333	3	102.85	8.26

py_Log Date 8/11/2021 12:11:50 PM

Notes:
1. RSD = Relative Standard Deviation

Table F-5 Soil Sample EIS Spike Recovery

Compound	Soil 2014-107				Soil 2016-106				Soil 2017-111			
	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	8.04	12	98.4	2.78	8.28	12	98.62	2.34	8.2	12	99.24	3.2
Perfluoro-n-[13C5]pentanoic acid	4.0	12	91.38	6.77	4.14	12	91.03	5.59	4.1	12	88.31	6.77
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	2.01	12	95.74	3.45	2.07	12	96.08	5.41	2.05	12	97.27	4.01
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	2.01	12	92.18	4.73	2.07	12	91.78	6.48	2.05	12	94.79	6.19
Perfluoro-n-[13C8]octanoic acid	2.03	12	94.28	4.01	2.09	12	94.56	4.25	2.07	12	97.25	2.8
Perfluoro-n-[13C9]nonanoic acid	1.01	12	95.16	2.5	1.04	12	94.52	2.53	1.03	12	97.76	3.35
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	1.01	12	95.38	5.33	1.04	12	96.39	4.56	1.03	12	95.63	3.98
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	1.01	12	100.99	6.44	1.04	12	101.82	6.97	1.03	12	100.29	4.27
Perfluoro-n-[1,2-13C2]dodecanoic acid	1.01	12	90.76	7.17	1.04	12	92	9.15	1.03	12	90.72	7.16
Perfluoro-n-[1,2-13C2]tetradecanoic acid	1.01	12	76.37	7.73	1.04	12	89.4	4.2	1.03	12	76.08	6.64
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	2.01	12	99.62	3.58	2.07	12	98.15	3.29	2.05	12	100.65	3.27
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	2.03	12	94.86	2.87	2.09	12	93.8	3.87	2.07	12	96.28	1.78
Perfluoro-1-[13C8]octanesulfonic acid	2.03	12	98.65	2.52	2.09	12	99.11	3.41	2.07	12	101.04	3.19
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	4.04	12	134.08	7.86	4.16	12	132.92	6.18	4.12	12	134.5	8.47
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	4.0	12	91.58	6.75	4.14	12	91.63	4.15	4.1	12	95.83	5.63
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	4.0	12	104.09	9.26	4.14	12	111.55	9.72	4.1	12	119.94	14.39
Perfluoro-1-[13C8]octanesulfonamide	2.01	12	104.83	4.6	2.07	12	108.33	4.73	2.05	12	110.06	8.35
N-methyl-d3-perfluoro-1-octanesulfonamide	2.01	12	48.88	21.07	2.07	12	47.71	19.53	2.05	12	58.16	12.62
N-ethyl-d5-perfluoro-1-octanesulfonamide	2.01	12	37.28	22.05	2.07	12	37.92	20.44	2.05	12	44.57	13.43
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	4.0	12	112.33	6.1	4.14	12	115.5	3.83	4.1	12	123	6.38
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	4.0	12	126.92	7	4.14	12	125.67	5.67	4.1	12	136.08	3.93
N-methyl-d7-perfluorooctanesulfonamidoethanol	20.1	12	70.16	12.33	20.7	12	73.42	7.58	20.5	12	74.92	5.27
N-ethyl-d9-perfluorooctane sulfonamidoethanol	20.1	12	58.4	14.82	20.7	12	63.54	10.94	20.5	12	62.57	8.76
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	8.04	12	102.08	7.49	8.28	12	97.3	15.18	8.2	12	99.27	12.94

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-5 Soil Sample EIS Spike Recovery

Compound	Soil 2018-105				Soil 2019-107				Soil 2019-110			
	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	8.08	12	99.05	1.93	8.19	12	100.56	3.65	8.03	12	98.43	15.38
Perfluoro-n-[13C5]pentanoic acid	4.04	12	89.82	8.24	4.1	12	88.83	8.43	4.02	12	95.22	8.91
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	2.02	12	96.41	3.03	2.05	12	94.51	4.76	2.01	12	98.58	5
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	2.02	12	91.18	7.25	2.05	12	93.78	5.8	2.01	12	95.48	5.67
Perfluoro-n-[13C8]octanoic acid	2.04	12	94.82	2.36	2.07	12	97.28	3.76	2.03	12	100.81	5.31
Perfluoro-n-[13C9]nonanoic acid	1.01	12	96.36	4.42	1.02	12	97.08	5.01	1.0	12	100.07	5.06
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	1.01	12	95.69	5.36	1.02	12	98.36	4.83	1.0	12	98.32	5.86
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	1.01	12	102.22	6.6	1.02	12	106.06	4.41	1.0	12	105.77	7.04
Perfluoro-n-[1,2-13C2]dodecanoic acid	1.01	12	96.33	5.49	1.02	12	95.37	6.58	1.0	12	94.34	6.36
Perfluoro-n-[1,2-13C2]tetradecanoic acid	1.01	12	93.09	4.9	1.02	12	91.92	6.42	1.0	12	83.06	9.62
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	2.02	12	101.44	3.18	2.05	12	102.58	5.9	2.01	12	103.41	6.21
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	2.04	12	96.76	2.62	2.07	12	98.3	5.08	2.03	12	99.5	6.1
Perfluoro-1-[13C8]octanesulfonic acid	2.04	12	101.85	2.88	2.07	12	101.4	4.39	2.03	12	104.09	5.61
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	4.06	12	130.42	6.32	4.12	12	127.25	6.42	4.04	12	144.83	7.04
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	4.04	12	107.42	5.62	4.1	12	103.66	6.59	4.02	12	99.99	7.11
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	4.04	12	104.83	10.61	4.1	12	107.94	8.32	4.02	12	119.53	14.22
Perfluoro-1-[13C8]octanesulfonamide	2.02	12	101.89	5.53	2.05	12	102.68	6.58	2.01	12	107.85	7.44
N-methyl-d3-perfluoro-1-octanesulfonamide	2.02	12	44.64	16.54	2.05	12	50.03	16.75	2.01	12	56.27	21.82
N-ethyl-d5-perfluoro-1-octanesulfonamide	2.02	12	37.52	17.01	2.05	12	41.05	17.42	2.01	12	43.05	24.09
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	4.04	12	98.35	5.42	4.1	12	104.61	7.89	4.02	12	117	7.01
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	4.04	12	102.03	7.63	4.1	12	111.92	8.44	4.02	12	129.67	6.4
N-methyl-d7-perfluorooctanesulfonamidoethanol	20.2	12	78.01	7.78	20.5	12	77.77	8.36	20.1	12	73.36	11.34
N-ethyl-d9-perfluorooctane sulfonamidoethanol	20.2	12	71.4	9.67	20.5	12	70.12	9.35	20.1	12	65.33	11.97
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	8.08	12	104.14	8.52	8.19	12	94.65	10.37	8.03	12	106.17	4.9

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-5 Soil Sample EIS Spike Recovery

Compound	Soil 2018-116				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	8.33	12	99.35	2.34	8.17	7	99.09	0.76
Perfluoro-n-[13C5]pentanoic acid	4.17	12	91.34	9.57	4.08	7	90.85	2.51
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	2.08	12	95.04	5.05	2.04	7	96.23	1.42
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	2.08	12	89.11	7.93	2.04	7	92.62	2.39
Perfluoro-n-[13C8]octanoic acid	2.1	12	95.51	2.28	2.06	7	96.36	2.4
Perfluoro-n-[13C9]nonanoic acid	1.04	12	95.67	3.75	1.02	7	96.66	1.93
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	1.04	12	95.73	3.92	1.02	7	96.5	1.34
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	1.04	12	104.52	4.99	1.02	7	103.09	2.26
Perfluoro-n-[1,2-13C2]dodecanoic acid	1.04	12	94.51	6.46	1.02	7	93.43	2.42
Perfluoro-n-[1,2-13C2]tetradecanoic acid	1.04	12	93.24	7.01	1.02	7	86.17	8.85
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	2.08	12	101.03	3.53	2.04	7	100.98	1.74
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	2.1	12	96.67	2.38	2.06	7	96.6	2
Perfluoro-1-[13C8]octanesulfonic acid	2.1	12	100.22	3.04	2.06	7	100.91	1.81
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	4.19	12	124.33	4.67	4.1	7	132.62	4.94
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	4.17	12	106.7	5.26	4.08	7	99.55	6.74
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	4.17	12	106.02	6.77	4.08	7	110.56	6.09
Perfluoro-1-[13C8]octanesulfonamide	2.08	12	96.4	5.44	2.04	7	104.58	4.5
N-methyl-d3-perfluoro-1-octanesulfonamide	2.08	12	52.51	10.05	2.04	7	51.17	9.37
N-ethyl-d5-perfluoro-1-octanesulfonamide	2.08	12	42.72	10.39	2.04	7	40.59	7.4
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	4.17	12	93.41	6.6	4.08	7	109.17	9.82
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	4.17	12	97.72	6.98	4.08	7	118.57	12.43
N-methyl-d7-perfluorooctanesulfonamidoethanol	20.8	12	77.13	5.85	20.4	7	74.97	3.85
N-ethyl-d9-perfluorooctane sulfonamidoethanol	20.8	12	70.4	7.34	20.4	7	65.97	7.36
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	8.33	12	98.39	11.73	8.17	7	100.29	4.03

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-6 Sediment Sample EIS Spike Recovery

Compound	Marine sediment				Sediment #3				Sediment #2				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	8	12	101.9	4.40	8.25	12	99.23	2.69	8.19	12	99.86	1.99	8.15	3	100.33	1.39
Perfluoro-n-[13C5]pentanoic acid	4.0	12	96.46	7.9	4.12	12	94.34	6.05	4.09	12	95.00	7.98	4.07	3	95.27	1.14
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	2	12	99.03	4.67	2.06	12	96.58	2.81	2.04	12	96.23	3.32	2.04	3	97.28	1.57
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	2	12	94.8	4	2.06	12	91.63	4.86	2.04	12	91.01	3.15	2.04	3	92.48	2.2
Perfluoro-n-[13C8]octanoic acid	2.02	12	98.45	4.63	2.08	12	96.89	3.97	2.07	12	96.28	3.39	2.06	3	97.21	1.15
Perfluoro-n-[13C9]nonanoic acid	0.999	12	97.69	4.26	1.03	12	97.13	3.49	1.02	12	95.99	2.85	1.02	3	96.94	0.89
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	0.999	12	98.23	5.89	1.03	12	96.02	3.13	1.02	12	96.13	5.68	1.02	3	96.8	1.29
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	0.999	12	104.95	6.71	1.03	12	103.24	5.69	1.02	12	105.75	1.62	1.02	3	104.65	1.22
Perfluoro-n-[1,2-13C2]dodecanoic acid	0.999	12	94.27	7.85	1.03	12	93.05	7.67	1.02	12	94.02	9.46	1.02	3	93.78	0.69
Perfluoro-n-[1,2-13C2]tetradecanoic acid	0.999	12	94.09	7.6	1.03	12	91.79	7.61	1.02	12	90.23	4.33	1.02	3	92.04	2.11
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	2	12	102.04	5.67	2.06	12	100.46	3.72	2.04	12	100.56	2.81	2.04	3	101.02	0.88
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	2.02	12	98.6	4.37	2.08	12	97.66	3.18	2.07	12	96.93	2.31	2.06	3	97.73	0.86
Perfluoro-1-[13C8]octanesulfonic acid	2.02	12	101.06	5.18	2.08	12	102.96	4.37	2.07	12	101.25	3.24	2.06	3	101.76	1.03
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	4.02	12	127.33	8.01	4.14	12	127.42	5.98	4.11	12	120.42	6.38	4.09	3	125.06	3.21
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	4.0	12	110.23	6.56	4.12	12	110.5	6.14	4.09	12	96.13	3.38	4.07	3	105.62	7.78
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	4.0	12	105.24	11.77	4.12	12	104.65	7.57	4.09	12	105.16	8.64	4.07	3	105.02	0.3
Perfluoro-1-[13C8]octanesulfonamide	2	12	103.09	7.19	2.06	12	105.02	6.75	2.04	12	110.77	5.62	2.04	3	106.29	3.76
N-methyl-d3-perfluoro-1-octanesulfonamide	2	12	70.97	9.85	2.06	12	63.38	10.53	2.04	12	55.74	13.46	2.04	3	63.36	12.01
N-ethyl-d5-perfluoro-1-octanesulfonamide	2	12	60.13	10.65	2.06	12	54.2	10.62	2.04	12	43.37	14.97	2.04	3	52.57	16.17
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	4.0	12	97.52	6.42	4.12	12	94.04	6.32	4.09	12	110.33	3.57	4.07	3	100.63	8.53
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	4.0	12	96.69	6.56	4.12	12	100.95	7.2	4.09	12	116.08	4.8	4.07	3	104.57	9.75
N-methyl-d7-perfluorooctanesulfonamidoethanol	20.0	12	92.77	7.93	20.6	12	93.19	6.86	20.4	12	77.75	8.6	20.4	3	87.9	10.01
N-ethyl-d9-perfluorooctane sulfonamidoethanol	20.0	12	84.64	8.71	20.6	12	83.92	8.47	20.4	12	71.19	9.83	20.4	3	79.92	9.47
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	8	12	102.18	7.25	8.25	12	98.88	10.2	8.19	12	97.76	10.88	8.15	3	99.61	2.31

py_Log Date 8/11/2021 12:11:50 PM

Notes:
1. RSD = Relative Standard Deviation

Table F-7 Biosolid Sample EIS Spike Recovery

Compound	Biosolid #1				Biosolid #2				Biosolid #3				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	78.3	12	35.08	107.40	80.8	12	50.48	53.32	85.6	12	27.03	159.29	81.5	3	37.53	31.75
Perfluoro-n-[13C5]pentanoic acid	39.1	12	87.34	15.29	40.4	12	97.87	6.35	42.8	12	52.20	55.33	40.8	3	79.14	30.22
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	19.6	12	98.26	7.55	20.2	12	97.21	5.32	21.4	13	89.78	8.44	20.4	3	95.08	4.86
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	19.6	12	97.00	4.34	20.2	12	94.27	7.3	21.4	12	89.55	4.67	20.4	3	93.61	4.03
Perfluoro-n-[13C8]octanoic acid	19.8	12	98.34	5.11	20.4	12	96.97	6.12	21.6	12	96.87	3.74	20.6	3	97.39	0.84
Perfluoro-n-[13C9]nonanoic acid	9.78	12	96.52	5.30	10.1	12	96.5	5.39	10.7	12	95.93	3.47	10.2	3	96.31	0.35
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	9.78	12	97.32	5.27	10.1	12	97.43	6.18	10.7	12	96.11	4.04	10.2	3	96.95	0.76
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	9.78	12	79.28	7.05	10.1	12	102.37	5.96	10.7	12	73.38	6.05	10.2	3	85.01	18.02
Perfluoro-n-[1,2-13C2]dodecanoic acid	9.78	12	42.76	20.34	10.1	12	89.57	10.71	10.7	12	37.19	21.62	10.2	3	56.51	50.91
Perfluoro-n-[1,2-13C2]tetradecanoic acid	9.78	12	27.13	16.89	10.1	12	56.62	8.84	10.7	12	33.00	23.43	10.2	3	38.92	40.10
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	19.6	12	102.08	8.90	20.2	12	102.38	5.80	21.4	12	107.08	6.89	20.4	3	103.84	2.70
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	19.8	12	99.58	5.77	20.4	12	99.01	4.49	21.6	12	102.33	3.48	20.6	3	100.31	1.77
Perfluoro-1-[13C8]octanesulfonic acid	19.8	12	100.69	7.60	20.4	12	102.58	4.47	21.6	12	92.85	12.45	20.6	3	98.71	5.23
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	39.3	12	195.92	16.51	40.6	12	142.25	7.45	43	12	138.95	22.34	41	3	159.04	20.11
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	39.1	12	111.32	10.28	40.4	12	113.58	5.24	42.8	12	104.87	7.25	40.8	3	109.92	4.11
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	39.1	12	153.33	9.78	40.4	12	126.58	8.00	42.8	12	118.33	6.79	40.8	3	132.75	13.78
Perfluoro-1-[13C8]octanesulfonamide	19.6	12	97.21	22.1	20.2	12	100.78	10.26	21.4	12	95.52	12.83	20.4	3	97.84	2.74
N-methyl-d3-perfluoro-1-octanesulfonamide	19.6	12	52.19	29.35	20.2	12	71.30	19.92	21.4	12	44.19	11.06	20.4	3	55.89	24.92
N-ethyl-d5-perfluoro-1-octanesulfonamide	19.6	12	36.04	29.29	20.2	12	56.80	20.96	21.4	12	30.87	9.82	20.4	3	41.24	33.28
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	39.1	12	107.83	15.64	40.4	12	125.75	10.36	42.8	12	76.55	13.43	40.8	3	103.38	24.09
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	39.1	12	108.32	18.76	40.4	12	133.5	12.52	42.8	12	80.00	10.78	40.8	3	107.27	24.95
N-methyl-d7-perfluorooctanesulfonamidoethanol	196.0	12	41.36	15.37	202	12	74.92	8.32	214	12	20.71	27.75	204	3	45.66	59.92
N-ethyl-d9-perfluorooctane sulfonamidoethanol	196.0	12	24.27	16.55	202	12	53.61	10.9	214	12	27.00	28.07	204	3	34.96	46.36
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	78.3	12	97.52	8.05	80.8	12	98.87	10.05	85.6	12	89.36	8.26	81.5	3	95.25	5.40

py_Log Date 8/11/2021 12:11:50 PM

Notes:
1. RSD = Relative Standard Deviation

Table F-8 Tissue Sample EIS Spike Recovery

Compound	Clam Tissue				Fish Tissue # 2				Fish Tissue #1				ALL EIS SPIKE RECOVERIES			
	Mean Concentration Spike (µg/kg wet weight)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg wet weight)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg wet weight)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]	Mean Concentration Spike (µg/kg wet weight)	Number EIS Spikes (n =)	Mean Percent Recovery	RSD [1]
Perfluoro-n-[13C4]butanoic acid	20.1	12	98.52	2.44	20.1	12	93.36	14.03	20.1	12	84.01	23.09	20.1	3	91.96	8.00
Perfluoro-n-[13C5]pentanoic acid	10.0	12	93.84	6.09	10	12	107.34	19.46	10.1	12	86.44	14.08	10	3	95.88	11.05
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	5.01	12	94.88	3.65	5.02	12	92.31	15.70	5.02	12	94.94	6.77	5.02	3	94.04	1.60
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	5.01	12	93.01	2.09	5.02	12	82.66	19.48	5.02	12	79.69	6.35	5.02	3	85.12	8.21
Perfluoro-n-[13C8]octanoic acid	5.06	12	94.72	3.45	5.07	12	90.46	14.9	5.07	12	95.25	4.72	5.07	3	93.48	2.81
Perfluoro-n-[13C9]nonanoic acid	2.51	12	92.12	4.07	2.51	12	90.30	15.68	2.51	12	98.02	3.67	2.51	3	93.48	4.32
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	2.51	12	83.28	6.06	2.51	12	90.68	15.39	2.51	12	97.19	4.18	2.51	3	90.39	7.70
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	2.51	12	71.19	10.00	2.51	12	88.58	13.08	2.51	12	90.94	4.83	2.51	3	83.57	12.9
Perfluoro-n-[1,2-13C2]dodecanoic acid	2.51	12	53.52	14.64	2.51	12	70.06	18.56	2.51	12	95.83	8.81	2.51	3	73.14	29.15
Perfluoro-n-[1,2-13C2]tetradecanoic acid	2.51	12	31.36	21.81	2.51	12	38.36	75.43	2.51	12	101.68	13.3	2.51	3	57.13	67.81
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	5.01	12	97.87	3.29	5.02	12	88.83	19.85	5.02	12	90.92	5.84	5.02	3	92.54	5.12
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	5.06	12	98.39	2.18	5.07	12	98.64	20.1	5.07	12	98.46	4.93	5.07	3	98.5	0.13
Perfluoro-1-[13C8]octanesulfonic acid	5.06	12	96.11	3.99	5.07	12	91.90	15.79	5.07	12	103.37	2.63	5.07	3	97.12	5.97
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	10.1	12	192.33	8.87	10.1	12	214.83	17.31	10.1	12	194.37	17.99	10.1	3	200.51	6.21
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	10.0	12	145.25	13.18	10	12	149.49	20.10	10.1	12	229.75	5.98	10	3	174.83	27.23
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	10.0	12	164.92	27.00	10	12	136.17	25.55	10.1	12	220.25	9.59	10	3	173.78	24.59
Perfluoro-1-[13C8]octanesulfonamide	5.01	12	95.52	6.15	5.02	12	87.44	15.31	5.02	12	92.63	6.90	5.02	3	91.87	4.46
N-methyl-d3-perfluoro-1-octanesulfonamide	5.01	12	37.65	18.52	5.02	12	26.80	33.20	5.02	12	8.08	16.75	5.02	3	24.18	61.86
N-ethyl-d5-perfluoro-1-octanesulfonamide	5.01	12	30.11	27.83	5.02	12	21.64	36.58	5.02	12	7.51	18.70	5.02	3	19.75	57.81
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	10.0	12	125.83	10.50	10	12	138.53	15.91	10.1	12	106.41	7.62	10	3	123.59	13.09
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	10.0	12	141.25	11.50	10	12	151.09	14.23	10.1	12	78.52	8.08	10	3	123.62	31.84
N-methyl-d7-perfluorooctanesulfonamidoethanol	50.1	12	30.27	44.94	50.2	12	12.32	68.11	50.2	12	5.20	23.74	50.2	3	15.93	81.09
N-ethyl-d9-perfluorooctane sulfonamidoethanol	50.1	12	29.23	31.33	50.2	12	12.55	58.71	50.2	12	0.37	63.60	50.2	3	14.05	103.11
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	20.1	12	92.52	11.80	20.1	12	96.31	17.44	20.1	12	94.49	7.44	20.1	3	96.31	5.13

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-9 All Aqueous Sample EIS Spike Recovery

Compound	Mean Concentration Spike (ng/L)	Number EIS Spikes (n =)	Mean Percent Recovery	Highest % Recovery	Lowest % Recovery	RSD [1]	SD [2]	2x SD [3]
Perfluoro-n-[13C4]butanoic acid	142	4	81.9	97.21	8.99	15.9	13.02	26.05
Perfluoro-n-[13C5]pentanoic acid	70.8	4	87.89	103.4	38.88	13.34	11.73	23.45
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	35.4	4	91.19	96.9	72.87	2.7	2.46	4.92
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	35.4	4	89.89	94.73	76.61	2.39	2.15	4.3
Perfluoro-n-[13C8]octanoic acid	35.8	4	92.35	95.05	87.27	0.81	0.75	1.49
Perfluoro-n-[13C9]nonanoic acid	17.7	4	91.22	95.19	82.37	1.59	1.45	2.89
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	17.7	4	87.48	92.71	70.56	3.31	2.9	5.79
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	17.7	4	85.66	94.49	56.25	6.46	5.53	11.06
Perfluoro-n-[1,2-13C2]dodecanoic acid	17.7	4	72.27	86.74	33.68	13.68	9.88	19.77
Perfluoro-n-[1,2-13C2]tetradecanoic acid	17.7	4	64.04	153.44	16.96	26.17	16.76	33.51
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	35.4	4	93.55	99.66	72.28	4.7	4.39	8.79
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	35.8	4	92.05	94.78	79.44	1.57	1.45	2.89
Perfluoro-1-[13C8]octanesulfonic acid	35.8	4	91.33	96.04	66.75	3.58	3.27	6.55
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	71.2	4	124.2	199	80.57	14.82	18.41	36.82
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	70.8	4	105.29	182.5	64.02	16.36	17.22	34.45
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	70.8	4	93.4	139.33	65.08	8.38	7.83	15.66
Perfluoro-1-[13C8]octanesulfonamide	35.4	4	77.6	92.62	26.68	15.42	11.97	23.94
N-methyl-d3-perfluoro-1-octanesulfonamide	35.4	4	59.82	74.05	13.51	16.42	9.82	19.64
N-ethyl-d5-perfluoro-1-octanesulfonamide	35.4	4	58.44	70.18	11.93	16.53	9.66	19.31
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	70.8	4	87.42	113.46	21.46	7.32	6.4	12.8
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	70.8	4	85.78	105.67	11.68	8.21	7.04	14.09
N-methyl-d7-perfluorooctanesulfonamidoethanol	354	4	61.92	77.01	10.99	18.55	11.48	22.97
N-ethyl-d9-perfluorooctane sulfonamidoethanol	354	4	57.63	72.61	7.99	19.62	11.31	22.61
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	142	4	101.07	112.5	91.83	2.02	2.04	4.08

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation

Table F-10 All Solid Sample EIS Spike Recovery

Compound	Mean Concentration Spike (µg/kg)	Number EIS Spikes (n =)	Mean Percent Recovery	Highest % Recovery	Lowest % Recovery	RSD [1]	SD [2]	2x SD [3]
Perfluoro-n-[13C4]butanoic acid	29.5	4	82.23	101.9	27.03	36.52	30.03	60.05
Perfluoro-n-[13C5]pentanoic acid	14.7	4	90.28	107.34	52.2	8.60	7.76	15.52
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	7.37	4	95.66	99.03	89.78	1.46	1.40	2.80
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	7.37	4	90.96	97.00	79.69	4.31	3.92	7.85
Perfluoro-n-[13C8]octanoic acid	7.44	4	96.11	100.81	90.46	1.89	1.81	3.63
Perfluoro-n-[13C9]nonanoic acid	3.68	4	95.85	100.07	90.3	1.67	1.6	3.19
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	3.68	4	95.16	98.36	83.28	3.35	3.19	6.38
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	3.68	4	94.08	106.06	71.19	12.05	11.34	22.68
Perfluoro-n-[1,2-13C2]dodecanoic acid	3.68	4	79.21	96.33	37.19	22.66	17.95	35.91
Perfluoro-n-[1,2-13C2]tetradecanoic acid	3.68	4	68.56	101.68	27.13	36.42	24.97	49.94
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	7.37	4	99.60	107.08	88.83	4.91	4.89	9.79
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	7.44	4	98.28	102.33	93.8	1.59	1.56	3.12
Perfluoro-1-[13C8]octanesulfonic acid	7.44	4	99.62	104.09	91.90	2.11	2.10	4.21
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	14.8	4	154.31	214.83	120.42	22.08	34.07	68.15
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	14.7	4	122.48	229.75	91.58	28.71	35.16	70.32
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	14.7	4	130.53	220.25	104.09	23.92	31.23	62.45
Perfluoro-1-[13C8]octanesulfonamide	7.37	4	100.14	110.77	87.44	6.61	6.62	13.23
N-methyl-d3-perfluoro-1-octanesulfonamide	7.37	4	48.65	71.3	8.08	35.09	17.07	34.14
N-ethyl-d5-perfluoro-1-octanesulfonamide	7.37	4	38.54	60.13	7.51	35.49	13.68	27.35
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	14.7	4	109.19	138.53	76.55	9.38	10.24	20.47
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	14.7	4	113.51	151.09	78.52	7.99	9.07	18.13
N-methyl-d7-perfluorooctanesulfonamidoethanol	73.7	4	56.11	93.19	5.20	57.19	32.09	64.19
N-ethyl-d9-perfluorooctane sulfonamidoethanol	73.7	4	48.72	84.64	0.37	61.13	29.79	59.57
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	29.5	4	97.86	106.17	89.36	2.51	2.46	4.92

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table F-11 All Media Sample EIS Spike Recovery

Compound	Number EIS Spikes (n =)	Mean Percent Recovery	Highest % Recovery	Lowest % Recovery	RSD [1]	SD [2]	2x SD [3]
Perfluoro-n-[13C4]butanoic acid	8	82.06	101.9	8.99	26.11	21.43	42.85
Perfluoro-n-[13C5]pentanoic acid	8	89.09	107.34	38.88	10.43	9.29	18.59
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	8	93.42	99.03	72.87	3.23	3.02	6.04
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	8	90.42	97.00	76.61	3.30	2.98	5.97
Perfluoro-n-[13C8]octanoic acid	8	94.23	100.81	87.27	2.53	2.38	4.77
Perfluoro-n-[13C9]nonanoic acid	8	93.53	100.07	82.37	3.05	2.85	5.70
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	8	91.32	98.36	70.56	5.45	4.98	9.96
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	8	89.87	106.06	56.25	10.46	9.41	18.81
Perfluoro-n-[1,2-13C2]dodecanoic acid	8	75.74	96.33	33.68	18.38	13.92	27.84
Perfluoro-n-[1,2-13C2]tetradecanoic acid	8	66.30	153.44	16.96	29.91	19.83	39.67
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	8	96.58	107.08	72.28	5.57	5.38	10.76
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	8	95.17	102.33	79.44	3.79	3.61	7.22
Perfluoro-1-[13C8]octanesulfonic acid	8	95.48	104.09	66.75	5.36	5.12	10.23
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	8	139.25	214.83	80.57	21.56	30.03	60.06
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	8	113.88	229.75	64.02	23.91	27.23	54.46
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	8	111.96	220.25	65.08	25.85	28.95	57.89
Perfluoro-1-[13C8]octanesulfonamide	8	88.87	110.77	26.68	16.89	15.01	30.03
N-methyl-d3-perfluoro-1-octanesulfonamide	8	54.24	74.05	8.08	26.20	14.21	28.42
N-ethyl-d5-perfluoro-1-octanesulfonamide	8	48.49	70.18	7.51	31.50	15.27	30.55
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	8	98.31	138.53	21.46	14.31	14.07	28.14
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	8	99.65	151.09	11.68	16.68	16.62	33.23
N-methyl-d7-perfluorooctanesulfonamidoethanol	8	59.02	93.19	5.20	38.17	22.53	45.06
N-ethyl-d9-perfluorooctane sulfonamidoethanol	8	53.18	84.64	0.37	40.23	21.39	42.79
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	8	99.47	112.5	89.36	2.72	2.71	5.41

py_Log Date 8/11/2021 12:11:50 PM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Appendix G

Injection Internal Standards (IIS) Recovery

APPENDIX G. INJECTED INTERNAL STANDARD (IIS) SPIKE RECOVERY TABLES

This appendix presents 11 summary tables by media of the IIS spike recoveries. IIS spike recovery tables indicate the extraction efficiency of the SLV method for isotopically labeled PFAS compounds. IIS summaries were generated for each of the eight environmental media: groundwater, wastewater, surface water, landfill leachate, soils, sediment, biosolids and tissue. For those individual media tables, the mean of the sample percent recoveries, and percent Relative Standard Deviation of the 7 isotopically labeled analog PFAS analytes are presented.

In each table the mean of the matrix mean percent recoveries; percent RSD, Standard Deviation (SD), 2× SD, 3× SD of the matrix mean percent recoveries for the seven IIS compounds, for aqueous, solid, and all media. Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021). Briefly, the tables include the following information.

Table Headings

- **Media** - Media type. Eight possible matrices
- **PFAS** - IIS Compound. Always seven.
- **n=** - Number of subsamples in the EDD for a matrix type with numeric values. Entries represented as N/A are not counted.
- **Mean % Recovery** - Mean of the subsample percent recovery, for a compound, for a matrix.
- **RSD**-Percent RSD of the mean of the sample percent recoveries.
- **SD**-SD of the sample percent recoveries.
- **2× SD** - SD of the sample percent recoveries times two.
- **3× SD** - SD of the sample percent recoveries times three.

Table G-1. Groundwater Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Groundwater	13C3-PFBA	60	76.60	10.53	8.07	16.13
Groundwater	13C2-PFHXA	60	73.57	10.68	7.86	15.72
Groundwater	13C4-PFOA	60	73.87	11.15	8.24	16.48
Groundwater	13C5-PFNA	60	75.99	11.50	8.74	17.47
Groundwater	13C2-PFDA	60	75.49	10.69	8.07	16.14
Groundwater	18O2-PFHXS	60	74.84	11.18	8.37	16.74
Groundwater	13C4-PFOS	60	73.64	13.14	9.68	19.36

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-2. Surface Water Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Surface Water	13C3-PFBA	39	80.36	14.00	11.25	22.50
Surface Water	13C2-PFHXA	39	77.06	13.59	10.47	20.95
Surface Water	13C4-PFOA	39	78.16	14.46	11.30	22.61
Surface Water	13C5-PFNA	39	79.29	13.86	10.99	21.98
Surface Water	13C2-PFDA	39	79.86	14.85	11.86	23.71
Surface Water	18O2-PFHXS	39	78.33	14.02	10.98	21.97
Surface Water	13C4-PFOS	39	76.86	13.91	10.69	21.39

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-3. Wasterwater Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Waste Water	13C3-PFBA	105	81.56	14.46	11.80	23.59
Waste Water	13C2-PFHXA	105	73.13	14.98	10.96	21.91
Waste Water	13C4-PFOA	105	77.06	13.52	10.42	20.84
Waste Water	13C5-PFNA	105	78.95	13.83	10.92	21.84
Waste Water	13C2-PFDA	105	64.52	34.85	22.48	44.97
Waste Water	18O2-PFHXS	105	76.85	14.39	11.06	22.12
Waste Water	13C4-PFOS	105	68.87	16.55	11.40	22.79

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-4. Landfill Leachate Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Landfill Leachate	13C3-PFBA	40	64.56	15.04	9.71	19.42
Landfill Leachate	13C2-PFHXA	40	49.42	25.88	12.79	25.58
Landfill Leachate	13C4-PFOA	40	62.71	11.77	7.38	14.76
Landfill Leachate	13C5-PFNA	40	67.22	13.13	8.83	17.66
Landfill Leachate	13C2-PFDA	40	67.15	12.61	8.46	16.93
Landfill Leachate	18O2-PFHXS	40	62.94	12.36	7.78	15.56
Landfill Leachate	13C4-PFOS	40	64.71	13.56	8.78	17.55

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-5. Soil Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Soil	13C3-PFBA	85	69.71	6.07	4.23	8.47
Soil	13C2-PFHXA	85	69.10	6.81	4.70	9.41
Soil	13C4-PFOA	85	70.30	5.98	4.20	8.40
Soil	13C5-PFNA	85	72.67	6.50	4.72	9.45
Soil	13C2-PFDA	85	73.86	5.88	4.34	8.69
Soil	18O2-PFHXS	85	70.49	5.95	4.19	8.38
Soil	13C4-PFOS	85	70.34	5.58	3.93	7.85

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-6. Sediment Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Sediment	13C3-PFBA	36	64.66	2.71	1.75	3.51
Sediment	13C2-PFHXA	36	62.98	3.35	2.11	4.22
Sediment	13C4-PFOA	36	64.04	3.59	2.30	4.59
Sediment	13C5-PFNA	36	65.46	3.20	2.09	4.19
Sediment	13C2-PFDA	36	66.01	3.75	2.48	4.96
Sediment	18O2-PFHXS	36	63.69	3.44	2.19	4.38
Sediment	13C4-PFOS	36	64.24	3.54	2.27	4.54

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-7. Biosolid Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Biosolid	13C3-PFBA	37	66.15	6.19	4.10	8.20
Biosolid	13C2-PFHXA	37	63.94	6.08	3.89	7.78
Biosolid	13C4-PFOA	37	67.12	6.21	4.17	8.33
Biosolid	13C5-PFNA	37	69.40	5.60	3.89	7.78
Biosolid	13C2-PFDA	37	66.22	9.81	6.50	12.99
Biosolid	18O2-PFHXS	37	65.58	5.92	3.88	7.77
Biosolid	13C4-PFOS	37	66.18	7.73	5.11	10.22

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-8. Tissue Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Tissue	13C3-PFBA	36	70.26	7.89	5.54	11.09
Tissue	13C2-PFHXA	36	58.74	19.35	11.37	22.73
Tissue	13C4-PFOA	36	69.45	9.53	6.62	13.24
Tissue	13C5-PFNA	36	71.97	11.10	7.99	15.97
Tissue	13C2-PFDA	36	67.39	19.36	13.04	26.09
Tissue	18O2-PFHXS	36	70.08	8.12	5.69	11.39
Tissue	13C4-PFOS	36	69.52	10.30	7.16	14.33

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-9. All Aqueous Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Aqueous	13C3-PFBA	4	75.77	10.25	7.77	15.53
Aqueous	13C2-PFHXA	4	68.30	18.60	12.70	25.41
Aqueous	13C4-PFOA	4	72.95	9.68	7.07	14.13
Aqueous	13C5-PFNA	4	75.36	7.47	5.63	11.25
Aqueous	13C2-PFDA	4	71.75	9.96	7.15	14.30
Aqueous	18O2-PFHXS	4	73.24	9.58	7.02	14.03
Aqueous	13C4-PFOS	4	71.02	7.52	5.34	10.67

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-10. All Solid Sample IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
Solid	13C3-PFBA	4	67.70	4.02	2.72	5.44
Solid	13C2-PFHXA	4	63.69	6.69	4.26	8.52
Solid	13C4-PFOA	4	67.73	4.13	2.80	5.60
Solid	13C5-PFNA	4	69.88	4.66	3.26	6.52
Solid	13C2-PFDA	4	68.37	5.43	3.71	7.43
Solid	18O2-PFHXS	4	67.46	4.98	3.36	6.72
Solid	13C4-PFOS	4	67.57	4.23	2.86	5.72

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Table G-11. All Samples IIS Spike Recovery

Media	PFAS	Number IIS Spikes (n =)	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]
All	13C3-PFBA	8	71.73	9.62	6.90	13.80
All	13C2-PFHXA	8	65.99	13.81	9.11	18.22
All	13C4-PFOA	8	70.34	8.11	5.70	11.41
All	13C5-PFNA	8	72.62	7.12	5.17	10.34
All	13C2-PFDA	8	70.06	7.96	5.58	11.15
All	18O2-PFHXS	8	70.35	8.47	5.96	11.91
All	13C4-PFOS	8	69.29	6.31	4.37	8.74

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation

Appendix H

Ongoing Precision and Recovery (OPR)

APPENDIX H. ONGOING PRECISION AND RECOVERY (OPR) SPIKE RECOVERY TABLES

This appendix presents 4 summary tables by media of the OPR spike recoveries. OPR limits are sometimes termed a "laboratory control sample" or a "quality control check sample. The limits generated in this single lab validation study will be used to develop specifications for the permissible range of recovery for each analyte. OPR indicates the performance of the sample preparation and detection method at a mid-range spike concentration in the study as a quality control check.

OPR spike recovery tables were generated for all aqueous media (groundwater, surface water, waste water, and landfill leachate), solids (sediment, soil, and biosolids), and for the fish and clam tissue samples. Each table includes the calculated the mean of the OPR subsample percent recoveries and percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes, from the 32 samples, for aqueous media and solid media (including biosolid, sediment, and soil, but excluding tissue). A separate table is included with the calculated the mean of the OPR subsample percent recoveries, percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes from the tissue matrix. All sample percent recoveries values in the EDD were used except that for those data that had B flagged values (where the results was associated with a contaminated blanks.

In addition to the aqueous, solid, and tissue OPR tables, a table of "all matrices" is included. These include the mean of the OPR matrix mean percent recoveries and percent RSD, SD, 2× SD, and 3× SD of the matrix mean percent recoveries for the 64 analytes, from all media (i.e., aqueous, solid, and tissue).

Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021). Briefly, the tables include the following information.

Table Headings

- **Compound –** 64 PFAS analytes.
- **n=** - Number of OPR subsamples that do not have B flagged values for the analyte in the EDD, for the media type (e.g., aqueous, solid (biosolid, soil, sediment), tissue).
- **% Mean Recovery** - Mean of the OPR subsample percent recoveries of the EDD entries.
- **RSD** - Percent Relative Standard Deviation of the mean of the sample percent recoveries.
- **SD**- Standard Deviation of the sample mean percent recoveries.
- **2× SD** - Standard Deviation of the sample mean percent recoveries times two.
- **3× SD** - Standard Deviation of the sample mean percent recoveries times three.

Table H-1. Aqueous OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluorobutanoic acid	22	100.91	6.13	6.19	12.37	18.56
Perfluoropentanoic acid	22	105.41	7.61	8.02	16.04	24.07
Perfluorohexanoic acid	22	99.78	5.64	5.63	11.25	16.88
Perfluoroheptanoic acid	22	99.91	5.05	5.05	10.09	15.14
Perfluorooctanoic acid	22	99.48	6.35	6.32	12.64	18.97
Perfluorononanoic acid	22	100.45	5.45	5.48	10.95	16.43
Perfluorodecanoic acid	22	103.51	5.78	5.98	11.96	17.94
Perfluoroundecanoic acid	22	100.61	5.77	5.80	11.60	17.40
Perfluorododecanoic acid	22	103.19	9.41	9.71	19.42	29.13
Perfluorotridecanoic acid	22	105.53	6.28	6.63	13.26	19.89
Perfluorotetradecanoic acid	22	102.62	6.50	6.67	13.33	20.00
Perfluorobutanesulfonic acid	22	101.55	7.28	7.39	14.78	22.17
Perfluoropentanesulfonic acid	22	100.65	6.93	6.98	13.95	20.93
Perfluorohexanesulfonic acid	22	108.40	5.04	5.47	10.93	16.40
Perfluoroheptanesulfonic acid	22	100.01	7.08	7.08	14.16	21.23
Perfluorooctanesulfonic acid	22	105.46	7.06	7.44	14.89	22.33
Perfluorononanesulfonic acid	22	104.36	8.97	9.36	18.73	28.09
Perfluorodecanesulfonic acid	22	95.50	5.95	5.69	11.37	17.06
Perfluorododecanesulfonic acid	22	90.15	6.63	5.98	11.95	17.93
4:2 fluorotelomersulfonic acid	22	105.24	6.72	7.07	14.14	21.22
6:2 fluorotelomersulfonic acid	18	104.99	11.39	11.95	23.91	35.86
8:2 fluorotelomersulfonic acid	22	111.41	5.45	6.08	12.15	18.23
Perfluorooctanesulfonamide	22	106.50	7.06	7.52	15.04	22.56
N-methyl perfluorooctanesulfonamide	22	97.66	7.20	7.04	14.07	21.11
N-ethyl perfluorooctanesulfonamide	22	95.91	6.55	6.29	12.57	18.86
N-methyl perfluorooctanesulfonamidoacetic acid	22	100.52	9.73	9.78	19.57	29.35
N-ethyl perfluorooctanesulfonamidoacetic acid	22	104.31	9.24	9.64	19.28	28.92
N-methyl perfluorooctanesulfonamidoethanol	22	103.30	5.45	5.62	11.25	16.87
N-ethyl perfluorooctanesulfonamidoethanol	22	104.79	6.44	6.75	13.50	20.25
Hexafluoropropylene oxide dimer acid	22	100.84	8.46	8.54	17.07	25.61
4,8-dioxa-3H-perfluorononanoic acid	22	97.06	10.15	9.85	19.71	29.56

Table H-1. Aqueous OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-3-methoxypropanoic acid	22	101.28	9.27	9.39	18.78	28.17
Perfluoro-4-methoxybutanoic acid	22	97.80	8.77	8.58	17.15	25.73
Perfluoro-3,6-dioxaheptanoic acid	22	97.35	21.11	20.55	41.10	61.65
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	22	100.09	9.89	9.89	19.79	29.68
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	22	95.95	10.43	10.01	20.02	30.03
Perfluoro(2-ethoxyethane)sulfonic acid	22	100.20	7.55	7.56	15.13	22.69
2H, 2H, 3H, 3H-perfluorohexanoic acid	22	96.71	15.78	15.26	30.52	45.79
2H, 2H, 3H, 3H-perfluorooctanoic acid	22	98.18	7.44	7.30	14.60	21.91
2H, 2H, 3H, 3H-perfluorodecanoic acid	22	98.76	8.51	8.40	16.80	25.21
Perfluoro-n-[13C4]butanoic acid	22	97.90	4.91	4.80	9.61	14.41
Perfluoro-n-[13C5]pentanoic acid	22	97.70	6.87	6.71	13.43	20.14
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	22	95.54	6.65	6.35	12.71	19.06
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	22	94.45	6.07	5.74	11.47	17.21
Perfluoro-n-[13C8]octanoic acid	22	95.67	6.18	5.91	11.82	17.72
Perfluoro-n-[13C9]nonanoic acid	22	95.51	5.95	5.68	11.36	17.04
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	22	94.90	5.92	5.62	11.23	16.85
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	22	96.57	6.40	6.18	12.36	18.55
Perfluoro-n-[1,2-13C2]dodecanoic acid	22	86.85	7.86	6.82	13.65	20.47
Perfluoro-n-[1,2-13C2]tetradecanoic acid	22	85.06	6.75	5.75	11.49	17.24
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	22	99.07	5.64	5.59	11.17	16.76
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	22	94.09	4.85	4.56	9.12	13.68
Perfluoro-1-[13C8]octanesulfonic acid	22	97.90	6.32	6.19	12.38	18.57
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	22	111.97	11.00	12.31	24.62	36.93
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	22	107.71	18.96	20.42	40.84	61.26
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	22	103.74	15.91	16.50	33.00	49.50
Perfluoro-1-[13C8]octanesulfonamide	22	82.62	15.80	13.05	26.10	39.16
N-methyl-d3-perfluoro-1-octanesulfonamide	22	61.62	18.55	11.43	22.86	34.29
N-ethyl-d5-perfluoro-1-octanesulfonamide	22	63.29	16.25	10.28	20.57	30.85
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	22	91.56	13.87	12.70	25.41	38.11
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	22	89.20	14.52	12.95	25.91	38.86
N-methyl-d7-perfluorooctanesulfonamidoethanol	22	70.44	20.06	14.13	28.26	42.39

Table H-1. Aqueous OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
N-ethyl-d9-perfluorooctane sulfonamidoethanol	22	66.97	17.48	11.71	23.41	35.12
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	22	104.45	8.08	8.44	16.89	25.33

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table H-2. Soils, Sediments and Biosolids OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluorobutanoic acid	17	100.27	3.97	3.98	7.97	11.95
Perfluoropentanoic acid	17	104.64	5.01	5.24	10.48	15.73
Perfluorohexanoic acid	17	97.67	4.66	4.55	9.09	13.64
Perfluoroheptanoic acid	17	97.86	4.77	4.67	9.34	14.01
Perfluorooctanoic acid	17	97.91	4.17	4.08	8.16	12.24
Perfluorononanoic acid	17	99.71	5.96	5.94	11.89	17.83
Perfluorodecanoic acid	17	103.44	6.92	7.16	14.32	21.48
Perfluoroundecanoic acid	17	101.25	4.67	4.73	9.46	14.19
Perfluorododecanoic acid	17	103.40	7.60	7.86	15.72	23.58
Perfluorotridecanoic acid	17	106.82	8.32	8.89	17.78	26.67
Perfluorotetradecanoic acid	17	100.78	4.50	4.53	9.07	13.60
Perfluorobutanesulfonic acid	17	101.02	4.86	4.91	9.83	14.74
Perfluoropentanesulfonic acid	17	100.37	5.86	5.88	11.76	17.64
Perfluorohexanesulfonic acid	17	104.84	4.01	4.21	8.41	12.62
Perfluoroheptanesulfonic acid	17	96.13	4.26	4.10	8.19	12.29
Perfluorooctanesulfonic acid	17	104.32	5.15	5.37	10.74	16.11
Perfluorononanesulfonic acid	17	96.74	10.50	10.16	20.32	30.49
Perfluorodecanesulfonic acid	17	95.25	6.07	5.79	11.57	17.36
Perfluorododecanesulfonic acid	17	88.19	6.61	5.83	11.65	17.48
4:2 fluorotelomersulfonic acid	17	99.98	6.39	6.39	12.78	19.16
6:2 fluorotelomersulfonic acid	17	113.13	23.38	26.45	52.90	79.35
8:2 fluorotelomersulfonic acid	17	115.65	5.12	5.92	11.83	17.75
Perfluorooctanesulfonamide	17	103.85	4.69	4.87	9.73	14.60
N-methyl perfluorooctanesulfonamide	17	103.89	6.07	6.31	12.62	18.93
N-ethyl perfluorooctanesulfonamide	17	105.61	4.36	4.60	9.21	13.81
N-methyl perfluorooctanesulfonamidoacetic acid	17	101.46	5.61	5.69	11.38	17.07
N-ethyl perfluorooctanesulfonamidoacetic acid	17	102.06	7.42	7.58	15.16	22.73
N-methyl perfluorooctanesulfonamidoethanol	17	103.06	4.31	4.44	8.88	13.32
N-ethyl perfluorooctanesulfonamidoethanol	17	105.59	4.58	4.83	9.67	14.50
Hexafluoropropylene oxide dimer acid	17	99.88	9.89	9.88	19.76	29.64
4,8-dioxa-3H-perfluorononanoic acid	17	99.79	12.12	12.10	24.20	36.30

Table H-2. Soils, Sediments and Biosolids OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-3-methoxypropanoic acid	17	100.76	8.06	8.12	16.24	24.35
Perfluoro-4-methoxybutanoic acid	17	102.83	8.57	8.82	17.63	26.45
Perfluoro-3,6-dioxaheptanoic acid	17	97.38	19.99	19.46	38.92	58.39
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	17	105.15	12.42	13.06	26.12	39.18
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	17	101.78	12.39	12.61	25.22	37.83
Perfluoro(2-ethoxyethane)sulfonic acid	17	98.60	5.08	5.01	10.02	15.04
2H, 2H, 3H, 3H-perfluorohexanoic acid	17	96.13	10.56	10.15	20.30	30.46
2H, 2H, 3H, 3H-perfluorooctanoic acid	17	90.43	5.93	5.37	10.73	16.10
2H, 2H, 3H, 3H-perfluorodecanoic acid	17	89.52	8.02	7.18	14.36	21.54
Perfluoro-n-[13C4]butanoic acid	17	102.22	3.30	3.37	6.75	10.12
Perfluoro-n-[13C5]pentanoic acid	17	95.24	7.96	7.58	15.16	22.73
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	17	98.76	3.60	3.55	7.10	10.65
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	17	94.92	2.70	2.56	5.12	7.68
Perfluoro-n-[13C8]octanoic acid	17	98.12	3.09	3.04	6.07	9.11
Perfluoro-n-[13C9]nonanoic acid	17	98.28	4.02	3.95	7.89	11.84
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	17	97.58	5.99	5.84	11.69	17.53
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	17	103.36	5.98	6.18	12.36	18.54
Perfluoro-n-[1,2-13C2]dodecanoic acid	17	89.60	9.37	8.40	16.80	25.19
Perfluoro-n-[1,2-13C2]tetradecanoic acid	17	90.61	9.05	8.20	16.40	24.60
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	17	102.68	3.09	3.17	6.34	9.50
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	17	99.18	3.62	3.59	7.19	10.78
Perfluoro-1-[13C8]octanesulfonic acid	17	101.68	3.45	3.51	7.02	10.53
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	17	134.29	4.09	5.50	10.99	16.49
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	17	121.00	6.98	8.44	16.88	25.32
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	17	108.38	6.96	7.54	15.08	22.62
Perfluoro-1-[13C8]octanesulfonamide	17	82.65	10.22	8.44	16.89	25.33
N-methyl-d3-perfluoro-1-octanesulfonamide	17	44.26	21.95	9.72	19.44	29.15
N-ethyl-d5-perfluoro-1-octanesulfonamide	17	38.19	25.96	9.92	19.83	29.75
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	17	97.82	5.94	5.81	11.61	17.42
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	17	96.82	6.18	5.98	11.97	17.95
N-methyl-d7-perfluorooctanesulfonamidoethanol	17	55.52	18.65	10.36	20.71	31.07

Table H-2. Soils, Sediments and Biosolids OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
N-ethyl-d9-perfluorooctane sulfonamidoethanol	17	51.94	19.54	10.15	20.30	30.45
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	17	103.81	10.15	10.53	21.07	31.60

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table H-3. Tissue OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluorobutanoic acid	4	99.88	5.12	5.11	10.23	15.34
Perfluoropentanoic acid	4	104.78	4.33	4.53	9.06	13.59
Perfluorohexanoic acid	4	100.62	5.19	5.22	10.44	15.65
Perfluoroheptanoic acid	4	102.53	7.78	7.97	15.95	23.92
Perfluorooctanoic acid	4	97.70	8.19	8.00	15.99	23.99
Perfluorononanoic acid	4	102.70	7.74	7.95	15.90	23.85
Perfluorodecanoic acid	4	98.35	7.14	7.02	14.04	21.05
Perfluoroundecanoic acid	4	103.53	6.29	6.51	13.03	19.54
Perfluorododecanoic acid	4	108.97	14.72	16.04	32.08	48.12
Perfluorotridecanoic acid	4	119.75	5.72	6.85	13.70	20.55
Perfluorotetradecanoic acid	4	100.90	5.14	5.19	10.38	15.57
Perfluorobutanesulfonic acid	4	102.88	6.63	6.82	13.64	20.45
Perfluoropentanesulfonic acid	4	100.80	5.80	5.85	11.69	17.54
Perfluorohexanesulfonic acid	4	106.92	7.64	8.17	16.33	24.50
Perfluoroheptanesulfonic acid	4	97.05	5.88	5.70	11.41	17.11
Perfluorooctanesulfonic acid	4	110.75	6.18	6.85	13.70	20.55
Perfluorononanesulfonic acid	4	99.50	7.08	7.05	14.09	21.14
Perfluorodecanesulfonic acid	4	93.90	8.67	8.14	16.28	24.41
Perfluorododecanesulfonic acid	4	68.43	28.97	19.82	39.64	59.46
4:2 fluorotelomersulfonic acid	4	96.40	3.36	3.24	6.48	9.72
6:2 fluorotelomersulfonic acid	4	105.35	6.46	6.81	13.62	20.43
8:2 fluorotelomersulfonic acid	4	118.75	7.09	8.42	16.84	25.26
Perfluorooctanesulfonamide	4	108.95	5.75	6.26	12.52	18.79
N-methyl perfluorooctanesulfonamide	4	101.55	7.52	7.63	15.27	22.90
N-ethyl perfluorooctanesulfonamide	4	108.75	8.51	9.25	18.50	27.75
N-methyl perfluorooctanesulfonamidoacetic acid	4	104.72	5.67	5.94	11.87	17.81
N-ethyl perfluorooctanesulfonamidoacetic acid	4	103.58	6.69	6.93	13.85	20.78
N-methyl perfluorooctanesulfonamidoethanol	4	231.25	24.47	56.58	113.15	169.73
N-ethyl perfluorooctanesulfonamidoethanol	4	109.90	22.29	24.50	48.99	73.49
Hexafluoropropylene oxide dimer acid	4	100.03	6.92	6.92	13.83	20.75
4,8-dioxa-3H-perfluorononanoic acid	4	109.03	10.69	11.65	23.31	34.96

Table H-3. Tissue OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-3-methoxypropanoic acid	4	97.60	5.97	5.83	11.65	17.48
Perfluoro-4-methoxybutanoic acid	4	100.45	8.12	8.15	16.31	24.46
Perfluoro-3,6-dioxaheptanoic acid	4	85.70	17.17	14.72	29.43	44.15
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	4	110.25	7.08	7.80	15.61	23.41
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	4	116.25	9.54	11.09	22.17	33.26
Perfluoro(2-ethoxyethane)sulfonic acid	4	97.38	4.91	4.78	9.56	14.35
2H, 2H, 3H, 3H-perfluorohexanoic acid	4	83.28	25.45	21.20	42.39	63.59
2H, 2H, 3H, 3H-perfluorooctanoic acid	4	138.53	21.91	30.35	60.69	91.04
2H, 2H, 3H, 3H-perfluorodecanoic acid	4	119.00	8.26	9.83	19.66	29.50
Perfluoro-n-[13C4]butanoic acid	4	100.00	2.38	2.38	4.76	7.14
Perfluoro-n-[13C5]pentanoic acid	4	95.97	3.82	3.66	7.33	10.99
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	4	93.12	2.68	2.49	4.99	7.48
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	4	90.95	6.21	5.65	11.30	16.96
Perfluoro-n-[13C8]octanoic acid	4	93.78	4.37	4.10	8.19	12.29
Perfluoro-n-[13C9]nonanoic acid	4	95.22	3.03	2.88	5.77	8.65
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	4	96.88	3.64	3.52	7.05	10.57
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	4	98.75	5.29	5.23	10.46	15.69
Perfluoro-n-[1,2-13C2]dodecanoic acid	4	88.95	10.61	9.43	18.87	28.30
Perfluoro-n-[1,2-13C2]tetradecanoic acid	4	59.85	41.55	24.87	49.73	74.60
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	4	100.15	2.74	2.75	5.50	8.25
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	4	96.70	3.15	3.05	6.09	9.14
Perfluoro-1-[13C8]octanesulfonic acid	4	98.95	1.86	1.84	3.68	5.51
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	4	223.25	15.28	34.12	68.24	102.36
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	4	132.75	6.04	8.02	16.03	24.05
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	4	191.75	29.30	56.18	112.36	168.54
Perfluoro-1-[13C8]octanesulfonamide	4	103.80	7.76	8.06	16.12	24.18
N-methyl-d3-perfluoro-1-octanesulfonamide	4	18.32	42.76	7.84	15.67	23.51
N-ethyl-d5-perfluoro-1-octanesulfonamide	4	26.40	55.67	14.70	29.39	44.09
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	4	169.75	7.70	13.07	26.15	39.22
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	4	199.00	5.99	11.92	23.83	35.75
N-methyl-d7-perfluorooctanesulfonamidoethanol	4	2.56	103.07	2.64	5.27	7.91

Table H-3. Tissue OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
N-ethyl-d9-perfluorooctane sulfonamidoethanol	4	14.07	65.76	9.26	18.51	27.77
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	4	93.22	6.68	6.23	12.46	18.69

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table H-4 All Media OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluorobutanoic acid	3	100.35	0.52	0.52	1.05	1.57
Perfluoropentanoic acid	3	104.94	0.39	0.41	0.82	1.23
Perfluorohexanoic acid	3	99.36	1.53	1.52	3.04	4.57
Perfluoroheptanoic acid	3	100.10	2.33	2.34	4.67	7.01
Perfluorooctanoic acid	3	98.36	0.99	0.97	1.95	2.92
Perfluorononanoic acid	3	100.95	1.54	1.56	3.12	4.68
Perfluorodecanoic acid	3	101.77	2.91	2.96	5.92	8.88
Perfluoroundecanoic acid	3	101.80	1.50	1.53	3.06	4.60
Perfluorododecanoic acid	3	105.19	3.12	3.28	6.56	9.85
Perfluorotridecanoic acid	3	110.70	7.10	7.86	15.73	23.59
Perfluorotetradecanoic acid	3	101.43	1.01	1.03	2.06	3.08
Perfluorobutanesulfonic acid	3	101.81	0.94	0.95	1.91	2.86
Perfluoropentanesulfonic acid	3	100.61	0.22	0.22	0.44	0.66
Perfluorohexanesulfonic acid	3	106.72	1.68	1.79	3.58	5.37
Perfluoroheptanesulfonic acid	3	97.73	2.08	2.03	4.06	6.09
Perfluorooctanesulfonic acid	3	106.84	3.21	3.43	6.86	10.29
Perfluorononanesulfonic acid	3	100.20	3.85	3.86	7.71	11.57
Perfluorodecanesulfonic acid	3	94.88	0.91	0.86	1.72	2.58
Perfluorododecanesulfonic acid	3	82.25	14.61	12.02	24.03	36.05
4:2 fluorotelomersulfonic acid	3	100.54	4.42	4.44	8.89	13.33
6:2 fluorotelomersulfonic acid	3	107.82	4.27	4.60	9.20	13.80
8:2 fluorotelomersulfonic acid	3	115.27	3.20	3.69	7.37	11.06
Perfluorooctanesulfonamide	3	106.43	2.40	2.55	5.10	7.65
N-methyl perfluorooctanesulfonamide	3	101.04	3.11	3.15	6.29	9.44
N-ethyl perfluorooctanesulfonamide	3	103.42	6.47	6.69	13.39	20.08
N-methyl perfluorooctanesulfonamidoacetic acid	3	102.24	2.16	2.21	4.41	6.62
N-ethyl perfluorooctanesulfonamidoacetic acid	3	103.31	1.11	1.15	2.30	3.44
N-methyl perfluorooctanesulfonamidoethanol	3	145.87	50.69	73.94	147.89	221.83
N-ethyl perfluorooctanesulfonamidoethanol	3	106.76	2.57	2.75	5.50	8.25
Hexafluoropropylene oxide dimer acid	3	100.25	0.52	0.52	1.03	1.55
4,8-dioxa-3H-perfluorononanoic acid	3	101.96	6.15	6.27	12.54	18.81

Table H-4 All Media OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-3-methoxypropanoic acid	3	99.88	1.99	1.99	3.98	5.98
Perfluoro-4-methoxybutanoic acid	3	100.36	2.51	2.52	5.03	7.55
Perfluoro-3,6-dioxaheptanoic acid	3	93.48	7.21	6.74	13.47	20.21
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	3	105.16	4.83	5.08	10.16	15.24
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	3	104.66	9.99	10.45	20.91	31.36
Perfluoro(2-ethoxyethane)sulfonic acid	3	98.73	1.43	1.42	2.83	4.25
2H, 2H, 3H, 3H-perfluorohexanoic acid	3	92.04	8.25	7.60	15.19	22.79
2H, 2H, 3H, 3H-perfluorooctanoic acid	3	109.04	23.68	25.82	51.65	77.47
2H, 2H, 3H, 3H-perfluorodecanoic acid	3	102.43	14.72	15.08	30.15	45.23
Perfluoro-n-[13C4]butanoic acid	3	100.04	2.16	2.16	4.32	6.48
Perfluoro-n-[13C5]pentanoic acid	3	96.30	1.31	1.26	2.52	3.79
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	3	95.81	2.95	2.83	5.66	8.49
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	3	93.44	2.32	2.17	4.34	6.51
Perfluoro-n-[13C8]octanoic acid	3	95.86	2.27	2.18	4.36	6.54
Perfluoro-n-[13C9]nonanoic acid	3	96.34	1.75	1.69	3.38	5.06
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	3	96.45	1.44	1.39	2.78	4.17
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	3	99.56	3.48	3.47	6.94	10.40
Perfluoro-n-[1,2-13C2]dodecanoic acid	3	88.47	1.62	1.44	2.87	4.31
Perfluoro-n-[1,2-13C2]tetradecanoic acid	3	78.50	20.88	16.39	32.78	49.18
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	3	100.64	1.84	1.85	3.71	5.56
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	3	96.66	2.63	2.54	5.09	7.63
Perfluoro-1-[13C8]octanesulfonic acid	3	99.51	1.96	1.95	3.90	5.85
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	3	156.51	37.62	58.87	117.74	176.61
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	3	120.49	10.40	12.53	25.06	37.59
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	3	134.62	36.79	49.53	99.06	148.58
Perfluoro-1-[13C8]octanesulfonamide	3	89.69	13.62	12.22	24.44	36.66
N-methyl-d3-perfluoro-1-octanesulfonamide	3	41.40	52.63	21.79	43.58	65.37
N-ethyl-d5-perfluoro-1-octanesulfonamide	3	42.63	44.20	18.84	37.68	56.52
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	3	119.71	36.29	43.45	86.89	130.34
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	3	128.34	47.77	61.31	122.62	183.93
N-methyl-d7-perfluorooctanesulfonamidoethanol	3	42.84	83.27	35.67	71.35	107.02

Table H-4 All Media OPR Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
N-ethyl-d9-perfluorooctane sulfonamidoethanol	3	44.33	61.49	27.26	54.51	81.77
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	3	100.50	6.27	6.30	12.61	18.91

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Appendix I

Limit of Quantitation Verification (LOQVER)

APPENDIX I. LIMIT OF QUANTITATION VERIFICATION SPIKE RECOVERY TABLES

This appendix presents summary tables by combined media of the Limit of Quantitation Verification (LOQVER) samples [also known as Low-Level OPR, or LLOPR]. The verification was accomplished using clean control material (e.g., reagent water, Ottawa sand, vegetable oil) spiked with PFAS target compounds and labeled compounds and carried through the entire analytical process (sample preparation and analysis). The limits generated in this single lab validation study may be used to develop specifications for the permissible range of recovery for each analyte.

LOQVER spike recovery tables were generated for all aqueous media (groundwater, surface water, wastewater, and landfill leachate), solids (sediment, soil, and biosolids), and for the fish and clam tissue samples. Each table includes the calculated the mean of the LOQVER subsample percent recoveries and percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes, from the 32 samples, for aqueous media and solid media (including biosolid, sediment, and soil, but excluding tissue). A separate table is included with the calculated the mean of the LOQVER subsample percent recoveries, percent RSD, SD, 2× SD, and 3× SD for 64 PFAS analytes from the tissue matrix. All sample percent recoveries values in the EDD were used except that for those data that had B flagged values (where the results were associated with a contaminated blanks).

In addition to the aqueous, solid, and tissue LOQVER tables, a table of "all matrices" is included. These include the mean of the LOQVER matrix mean percent recoveries and percent RSD, SD, 2× SD, and 3× SD of the matrix mean percent recoveries for the 64 analytes, from all media (i.e., aqueous, solid, and tissue).

Details of the data compilation and analyses process are presented in Appendix B (IDA, 2021). Briefly, the tables include the following information.

Table Headings

- **Compound –** 64 PFAS analytes.
- **n=** - Number of LOQVER subsamples that do not have B flagged values for the analyte in the EDD, for the media type (e.g., aqueous, solid (biosolid, soil, sediment), tissue).
- **% Mean Recovery** - Mean of the LOQVER subsample percent recoveries of the EDD entries.
- **RSD** - Percent Relative Standard Deviation of the mean of the sample percent recoveries.
- **SD**- Standard Deviation of the sample mean percent recoveries.
- **2× SD** - Standard Deviation of the sample mean percent recoveries times two.
- **3× SD** - Standard Deviation of the sample mean percent recoveries times three.

Table I-1 Aqueous Media LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluorobutanoic acid	22	105.08	8.30	8.72	17.44	26.15
Perfluoropentanoic acid	22	109.19	9.02	9.85	19.70	29.55
Perfluorohexanoic acid	22	104.86	10.41	10.91	21.82	32.73
Perfluoroheptanoic acid	22	103.90	8.59	8.93	17.86	26.79
Perfluorooctanoic acid	22	106.84	10.25	10.95	21.90	32.85
Perfluorononanoic acid	22	104.36	7.82	8.16	16.31	24.47
Perfluorodecanoic acid	22	105.48	8.51	8.97	17.95	26.92
Perfluoroundecanoic acid	22	103.11	8.24	8.49	16.98	25.48
Perfluorododecanoic acid	22	101.98	10.00	10.20	20.40	30.59
Perfluorotridecanoic acid	22	107.77	7.09	7.64	15.28	22.92
Perfluorotetradecanoic acid	22	103.91	6.71	6.97	13.94	20.91
Perfluorobutanesulfonic acid	22	104.04	9.24	9.61	19.23	28.84
Perfluoropentanesulfonic acid	22	102.25	7.58	7.75	15.51	23.26
Perfluorohexanesulfonic acid	22	112.50	8.18	9.20	18.40	27.59
Perfluoroheptanesulfonic acid	22	99.92	6.33	6.32	12.65	18.97
Perfluorooctanesulfonic acid	22	108.39	9.01	9.76	19.53	29.29
Perfluorononanesulfonic acid	22	102.84	12.43	12.78	25.57	38.35
Perfluorodecanesulfonic acid	22	96.65	10.02	9.68	19.37	29.05
Perfluorododecanesulfonic acid	22	88.30	5.45	4.81	9.63	14.44
4:2 fluorotelomersulfonic acid	22	106.14	8.65	9.18	18.35	27.53
6:2 fluorotelomersulfonic acid	18	118.93	23.70	28.19	56.38	84.58
8:2 fluorotelomersulfonic acid	22	115.92	9.50	11.02	22.03	33.05
Perfluorooctanesulfonamide	22	107.30	9.53	10.23	20.46	30.69
N-methyl perfluorooctanesulfonamide	22	78.96	15.31	12.09	24.18	36.26
N-ethyl perfluorooctanesulfonamide	22	79.02	15.65	12.37	24.74	37.11
N-methyl perfluorooctanesulfonamidoacetic acid	22	101.66	10.40	10.57	21.15	31.72
N-ethyl perfluorooctanesulfonamidoacetic acid	22	103.10	15.41	15.89	31.78	47.66
N-methyl perfluorooctanesulfonamidoethanol	22	99.43	6.82	6.78	13.55	20.33
N-ethyl perfluorooctanesulfonamidoethanol	22	99.22	6.75	6.70	13.39	20.09
Hexafluoropropylene oxide dimer acid	22	102.38	11.45	11.72	23.44	35.16
4,8-dioxa-3H-perfluorononanoic acid	22	98.20	11.03	10.83	21.66	32.50

Table I-1 Aqueous Media LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-3-methoxypropanoic acid	22	103.02	7.91	8.15	16.30	24.44
Perfluoro-4-methoxybutanoic acid	22	102.37	9.33	9.55	19.09	28.64
Perfluoro-3,6-dioxaheptanoic acid	22	100.44	27.27	27.39	54.77	82.16
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	22	101.63	9.93	10.09	20.18	30.28
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	22	95.93	8.57	8.23	16.45	24.68
Perfluoro(2-ethoxyethane)sulfonic acid	22	104.70	8.72	9.13	18.26	27.39
2H, 2H, 3H, 3H-perfluorohexanoic acid	22	94.36	14.82	13.98	27.96	41.94
2H, 2H, 3H, 3H-perfluorooctanoic acid	22	99.81	11.25	11.23	22.46	33.68
2H, 2H, 3H, 3H-perfluorodecanoic acid	22	101.45	10.08	10.23	20.45	30.68
Perfluoro-n-[13C4]butanoic acid	22	97.32	3.45	3.35	6.71	10.06
Perfluoro-n-[13C5]pentanoic acid	22	96.17	6.41	6.17	12.33	18.50
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	22	93.57	5.84	5.47	10.93	16.40
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	22	92.56	5.76	5.33	10.66	15.99
Perfluoro-n-[13C8]octanoic acid	22	95.42	4.45	4.25	8.49	12.74
Perfluoro-n-[13C9]nonanoic acid	22	94.45	4.04	3.82	7.64	11.46
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	22	94.56	6.13	5.80	11.60	17.40
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	22	96.73	6.24	6.04	12.07	18.11
Perfluoro-n-[1,2-13C2]dodecanoic acid	22	87.03	7.66	6.67	13.33	20.00
Perfluoro-n-[1,2-13C2]tetradecanoic acid	22	85.18	8.64	7.36	14.72	22.08
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	22	98.04	5.24	5.13	10.27	15.40
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	22	94.28	3.80	3.58	7.16	10.75
Perfluoro-1-[13C8]octanesulfonic acid	22	97.43	3.88	3.78	7.57	11.35
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	22	115.73	9.70	11.22	22.44	33.67
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	22	110.10	16.27	17.92	35.83	53.75
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	22	105.67	14.01	14.80	29.61	44.41
Perfluoro-1-[13C8]octanesulfonamide	22	81.18	14.82	12.03	24.06	36.08
N-methyl-d3-perfluoro-1-octanesulfonamide	22	57.27	17.31	9.92	19.83	29.75
N-ethyl-d5-perfluoro-1-octanesulfonamide	22	58.33	15.10	8.80	17.61	26.41
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	22	91.09	10.85	9.88	19.77	29.65
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	22	89.27	12.65	11.29	22.58	33.87
N-methyl-d7-perfluorooctanesulfonamidoethanol	22	67.21	19.23	12.93	25.85	38.78

Table I-1 Aqueous Media LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
N-ethyl-d9-perfluorooctane sulfonamidoethanol	22	63.92	15.78	10.09	20.18	30.27
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	22	104.52	5.83	6.09	12.18	18.27

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table I-2. Soil, Sediment, and Biosolids LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluorobutanoic acid	17	100.96	7.20	7.27	14.53	21.80
Perfluoropentanoic acid	17	100.44	9.77	9.81	19.63	29.44
Perfluorohexanoic acid	17	97.24	6.62	6.43	12.87	19.30
Perfluoroheptanoic acid	17	97.36	6.79	6.61	13.22	19.83
Perfluorooctanoic acid	17	98.34	8.04	7.90	15.80	23.71
Perfluorononanoic acid	17	98.82	5.64	5.58	11.16	16.73
Perfluorodecanoic acid	17	100.06	7.04	7.04	14.09	21.13
Perfluoroundecanoic acid	17	97.56	7.13	6.95	13.91	20.86
Perfluorododecanoic acid	17	98.55	7.82	7.71	15.42	23.13
Perfluorotridecanoic acid	17	102.25	7.34	7.51	15.02	22.53
Perfluorotetradecanoic acid	17	100.28	6.75	6.77	13.55	20.32
Perfluorobutanesulfonic acid	17	100.66	7.12	7.17	14.34	21.52
Perfluoropentanesulfonic acid	17	97.71	8.34	8.15	16.30	24.45
Perfluorohexanesulfonic acid	17	103.94	7.67	7.97	15.94	23.91
Perfluoroheptanesulfonic acid	17	93.24	6.52	6.08	12.16	18.24
Perfluorooctanesulfonic acid	17	100.27	8.92	8.95	17.89	26.84
Perfluorononanesulfonic acid	17	88.45	13.28	11.75	23.50	35.25
Perfluorodecanesulfonic acid	17	91.68	8.68	7.96	15.91	23.87
Perfluorododecanesulfonic acid	17	85.00	8.77	7.45	14.90	22.35
4:2 fluorotelomersulfonic acid	17	96.69	6.93	6.70	13.39	20.09
6:2 fluorotelomersulfonic acid	16	128.41	53.88	69.18	138.37	207.55
8:2 fluorotelomersulfonic acid	17	113.05	7.85	8.87	17.75	26.62
Perfluorooctanesulfonamide	17	104.66	7.01	7.33	14.67	22.00
N-methyl perfluorooctanesulfonamide	17	95.91	9.24	8.87	17.73	26.60
N-ethyl perfluorooctanesulfonamide	17	99.75	9.38	9.36	18.71	28.07
N-methyl perfluorooctanesulfonamidoacetic acid	17	92.45	14.12	13.05	26.10	39.15
N-ethyl perfluorooctanesulfonamidoacetic acid	17	99.38	11.43	11.36	22.71	34.07
N-methyl perfluorooctanesulfonamidoethanol	17	97.56	5.63	5.49	10.99	16.48
N-ethyl perfluorooctanesulfonamidoethanol	17	100.80	6.16	6.21	12.43	18.64
Hexafluoropropylene oxide dimer acid	17	95.55	11.92	11.39	22.77	34.16
4,8-dioxa-3H-perfluorononanoic acid	17	92.54	12.40	11.47	22.94	34.42

Table I-2. Soil, Sediment, and Biosolids LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-3-methoxypropanoic acid	17	96.12	7.19	6.91	13.82	20.73
Perfluoro-4-methoxybutanoic acid	17	95.32	10.87	10.36	20.72	31.08
Perfluoro-3,6-dioxaheptanoic acid	17	90.95	24.78	22.54	45.07	67.61
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	17	97.06	11.70	11.35	22.70	34.05
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	17	94.19	14.03	13.22	26.43	39.65
Perfluoro(2-ethoxyethane)sulfonic acid	17	93.18	9.56	8.91	17.81	26.72
2H, 2H, 3H, 3H-perfluorohexanoic acid	17	88.01	10.50	9.24	18.47	27.71
2H, 2H, 3H, 3H-perfluorooctanoic acid	17	86.91	6.91	6.01	12.02	18.03
2H, 2H, 3H, 3H-perfluorodecanoic acid	17	86.06	8.62	7.42	14.83	22.25
Perfluoro-n-[13C4]butanoic acid	17	102.26	3.44	3.52	7.03	10.55
Perfluoro-n-[13C5]pentanoic acid	17	95.68	8.03	7.68	15.36	23.04
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	17	99.08	4.10	4.06	8.13	12.19
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	17	95.31	4.56	4.34	8.68	13.03
Perfluoro-n-[13C8]octanoic acid	17	98.42	3.94	3.88	7.75	11.63
Perfluoro-n-[13C9]nonanoic acid	17	98.99	4.97	4.92	9.84	14.75
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	17	97.68	3.98	3.89	7.78	11.66
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	17	101.82	6.13	6.24	12.48	18.72
Perfluoro-n-[1,2-13C2]dodecanoic acid	17	91.36	7.24	6.61	13.22	19.84
Perfluoro-n-[1,2-13C2]tetradecanoic acid	17	88.42	9.34	8.26	16.51	24.77
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	17	103.06	3.84	3.96	7.92	11.88
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	17	98.15	3.17	3.11	6.23	9.34
Perfluoro-1-[13C8]octanesulfonic acid	17	101.51	4.27	4.33	8.67	13.00
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	17	143.29	4.70	6.73	13.47	20.20
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	17	122.47	6.08	7.45	14.90	22.35
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	17	109.51	6.12	6.70	13.40	20.10
Perfluoro-1-[13C8]octanesulfonamide	17	84.56	11.83	10.00	20.01	30.01
N-methyl-d3-perfluoro-1-octanesulfonamide	17	42.41	26.90	11.41	22.81	34.22
N-ethyl-d5-perfluoro-1-octanesulfonamide	17	35.94	33.93	12.19	24.39	36.58
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	17	98.52	9.02	8.88	17.77	26.65
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	17	97.76	10.18	9.95	19.91	29.86
N-methyl-d7-perfluorooctanesulfonamidoethanol	17	54.89	20.11	11.04	22.08	33.11

Table I-2. Soil, Sediment, and Biosolids LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
N-ethyl-d9-perfluorooctane sulfonamidoethanol	17	51.39	20.79	10.68	21.36	32.05
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	17	106.15	10.09	10.71	21.43	32.14

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table I-3. Tissue LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluorobutanoic acid	4	104.45	6.77	7.07	14.14	21.22
Perfluoropentanoic acid	4	108.62	8.55	9.29	18.57	27.86
Perfluorohexanoic acid	4	104.90	10.60	11.12	22.24	33.36
Perfluoroheptanoic acid	4	102.15	5.31	5.42	10.85	16.27
Perfluorooctanoic acid	4	103.05	8.59	8.85	17.70	26.55
Perfluorononanoic acid	4	106.25	4.82	5.12	10.25	15.37
Perfluorodecanoic acid	4	104.12	7.23	7.53	15.06	22.60
Perfluoroundecanoic acid	4	118.25	3.04	3.59	7.19	10.78
Perfluorododecanoic acid	4	101.95	10.63	10.84	21.68	32.52
Perfluorotridecanoic acid	4	126.00	8.65	10.89	21.79	32.68
Perfluorotetradecanoic acid	4	105.67	7.37	7.79	15.58	23.37
Perfluorobutanesulfonic acid	4	103.25	7.94	8.20	16.39	24.59
Perfluoropentanesulfonic acid	4	101.38	8.32	8.44	16.87	25.31
Perfluorohexanesulfonic acid	4	111.00	6.62	7.35	14.70	22.05
Perfluoroheptanesulfonic acid	4	98.47	8.37	8.25	16.49	24.74
Perfluorooctanesulfonic acid	4	126.75	3.43	4.35	8.70	13.05
Perfluorononanesulfonic acid	4	102.28	14.68	15.02	30.04	45.06
Perfluorodecanesulfonic acid	4	96.55	10.41	10.05	20.11	30.16
Perfluorododecanesulfonic acid	4	61.35	11.53	7.07	14.15	21.22
4:2 fluorotelomersulfonic acid	4	99.38	7.63	7.59	15.17	22.76
6:2 fluorotelomersulfonic acid	4	116.75	9.87	11.53	23.06	34.59
8:2 fluorotelomersulfonic acid	4	128.00	9.53	12.19	24.39	36.58
Perfluorooctanesulfonamide	4	116.75	5.61	6.55	13.10	19.65
N-methyl perfluorooctanesulfonamide	4	99.23	3.41	3.38	6.76	10.14
N-ethyl perfluorooctanesulfonamide	4	109.83	6.98	7.67	15.33	23.00
N-methyl perfluorooctanesulfonamidoacetic acid	4	87.55	15.46	13.53	27.07	40.60
N-ethyl perfluorooctanesulfonamidoacetic acid	4	107.35	6.79	7.29	14.58	21.87
N-methyl perfluorooctanesulfonamidoethanol	4	237.25	30.60	72.60	145.19	217.79
N-ethyl perfluorooctanesulfonamidoethanol	4	125.50	14.34	17.99	35.98	53.97
Hexafluoropropylene oxide dimer acid	4	95.00	6.20	5.89	11.78	17.67
4,8-dioxa-3H-perfluorononanoic acid	4	109.42	11.21	12.26	24.53	36.79

Table I-3. Tissue LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-3-methoxypropanoic acid	4	94.62	6.11	5.78	11.56	17.34
Perfluoro-4-methoxybutanoic acid	4	102.05	8.44	8.61	17.23	25.84
Perfluoro-3,6-dioxaheptanoic acid	4	92.80	11.28	10.47	20.94	31.41
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	4	113.50	9.61	10.91	21.82	32.73
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	4	117.00	10.42	12.19	24.39	36.58
Perfluoro(2-ethoxyethane)sulfonic acid	4	96.72	8.95	8.66	17.32	25.98
2H, 2H, 3H, 3H-perfluorohexanoic acid	4	81.53	23.94	19.52	39.03	58.55
2H, 2H, 3H, 3H-perfluorooctanoic acid	4	136.07	20.30	27.63	55.26	82.89
2H, 2H, 3H, 3H-perfluorodecanoic acid	4	117.00	8.58	10.03	20.07	30.10
Perfluoro-n-[13C4]butanoic acid	4	98.23	3.54	3.48	6.95	10.43
Perfluoro-n-[13C5]pentanoic acid	4	100.85	4.46	4.49	8.99	13.48
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	4	96.47	2.75	2.65	5.31	7.96
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	4	90.62	8.58	7.78	15.56	23.34
Perfluoro-n-[13C8]octanoic acid	4	97.05	5.60	5.43	10.86	16.30
Perfluoro-n-[13C9]nonanoic acid	4	97.08	3.81	3.69	7.39	11.08
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	4	99.62	4.59	4.57	9.14	13.71
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	4	99.92	5.97	5.96	11.93	17.89
Perfluoro-n-[1,2-13C2]dodecanoic acid	4	95.35	8.90	8.49	16.98	25.46
Perfluoro-n-[1,2-13C2]tetradecanoic acid	4	47.90	18.67	8.94	17.89	26.83
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	4	96.10	3.39	3.26	6.52	9.79
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	4	96.20	3.92	3.77	7.54	11.31
Perfluoro-1-[13C8]octanesulfonic acid	4	99.70	6.57	6.55	13.11	19.66
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	4	221.50	10.82	23.97	47.93	71.90
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	4	127.00	2.65	3.37	6.73	10.10
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	4	180.75	29.92	54.08	108.16	162.25
Perfluoro-1-[13C8]octanesulfonamide	4	103.85	8.14	8.45	16.90	25.35
N-methyl-d3-perfluoro-1-octanesulfonamide	4	18.27	13.75	2.51	5.03	7.54
N-ethyl-d5-perfluoro-1-octanesulfonamide	4	19.65	13.00	2.55	5.11	7.66
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	4	171.25	7.57	12.97	25.94	38.91
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	4	203.50	7.28	14.82	29.64	44.46
N-methyl-d7-perfluorooctanesulfonamidoethanol	4	1.52	44.37	0.67	1.35	2.02

Table I-3. Tissue LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
N-ethyl-d9-perfluorooctane sulfonamidoethanol	4	10.04	41.84	4.20	8.40	12.60
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	5	96.00	8.63	8.28	16.57	24.85

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table I-4. All Media LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluorobutanoic acid	3	103.50	2.14	2.22	4.44	6.66
Perfluoropentanoic acid	3	106.09	4.62	4.90	9.79	14.69
Perfluorohexanoic acid	3	102.33	4.31	4.41	8.82	13.23
Perfluoroheptanoic acid	3	101.13	3.35	3.38	6.77	10.15
Perfluorooctanoic acid	3	102.74	4.15	4.26	8.52	12.78
Perfluorononanoic acid	3	103.14	3.74	3.86	7.72	11.58
Perfluorodecanoic acid	3	103.22	2.73	2.82	5.64	8.46
Perfluoroundecanoic acid	3	106.31	10.07	10.71	21.42	32.13
Perfluorododecanoic acid	3	100.83	1.95	1.97	3.94	5.91
Perfluorotridecanoic acid	3	112.01	11.10	12.43	24.86	37.29
Perfluorotetradecanoic acid	3	103.29	2.66	2.75	5.51	8.26
Perfluorobutanesulfonic acid	3	102.65	1.72	1.76	3.53	5.29
Perfluoropentanesulfonic acid	3	100.44	2.40	2.41	4.81	7.22
Perfluorohexanesulfonic acid	3	109.15	4.19	4.58	9.15	13.73
Perfluoroheptanesulfonic acid	3	97.21	3.62	3.52	7.04	10.56
Perfluorooctanesulfonic acid	3	111.80	12.13	13.57	27.13	40.70
Perfluorononanesulfonic acid	3	97.85	8.33	8.15	16.29	24.44
Perfluorodecanesulfonic acid	3	94.96	2.99	2.84	5.68	8.51
Perfluorododecanesulfonic acid	3	78.22	18.79	14.70	29.40	44.09
4:2 fluorotelomersulfonic acid	3	100.73	4.83	4.87	9.74	14.60
6:2 fluorotelomersulfonic acid	3	121.36	5.11	6.20	12.39	18.59
8:2 fluorotelomersulfonic acid	3	118.99	6.67	7.93	15.86	23.80
Perfluorooctanesulfonamide	3	109.57	5.80	6.35	12.71	19.06
N-methyl perfluorooctanesulfonamide	3	91.37	11.90	10.87	21.74	32.61
N-ethyl perfluorooctanesulfonamide	3	96.20	16.33	15.71	31.41	47.12
N-methyl perfluorooctanesulfonamidoacetic acid	3	93.89	7.63	7.16	14.33	21.49
N-ethyl perfluorooctanesulfonamidoacetic acid	3	103.28	3.86	3.99	7.98	11.97
N-methyl perfluorooctanesulfonamidoethanol	3	144.75	55.35	80.11	160.23	240.34
N-ethyl perfluorooctanesulfonamidoethanol	3	108.51	13.58	14.74	29.47	44.21
Hexafluoropropylene oxide dimer acid	3	97.64	4.21	4.11	8.23	12.34
4,8-dioxa-3H-perfluorononanoic acid	3	100.05	8.59	8.59	17.19	25.78

Table I-4. All Media LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-3-methoxypropanoic acid	3	97.92	4.57	4.48	8.96	13.44
Perfluoro-4-methoxybutanoic acid	3	99.91	3.99	3.98	7.97	11.95
Perfluoro-3,6-dioxaheptanoic acid	3	94.73	5.31	5.03	10.06	15.09
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	3	104.06	8.15	8.49	16.97	25.46
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	3	102.37	12.40	12.70	25.39	38.09
Perfluoro(2-ethoxyethane)sulfonic acid	3	98.20	6.01	5.90	11.80	17.71
2H, 2H, 3H, 3H-perfluorohexanoic acid	3	87.96	7.30	6.42	12.84	19.26
2H, 2H, 3H, 3H-perfluorooctanoic acid	3	107.60	23.69	25.49	50.98	76.47
2H, 2H, 3H, 3H-perfluorodecanoic acid	3	101.50	15.24	15.47	30.94	46.41
Perfluoro-n-[13C4]butanoic acid	3	99.27	2.65	2.63	5.26	7.89
Perfluoro-n-[13C5]pentanoic acid	3	97.57	2.92	2.85	5.71	8.56
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	3	96.37	2.86	2.75	5.51	8.26
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	3	92.83	2.53	2.35	4.70	7.06
Perfluoro-n-[13C8]octanoic acid	3	96.96	1.55	1.50	3.00	4.50
Perfluoro-n-[13C9]nonanoic acid	3	96.84	2.35	2.28	4.56	6.84
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	3	97.29	2.63	2.56	5.11	7.67
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	3	99.49	2.58	2.57	5.14	7.71
Perfluoro-n-[1,2-13C2]dodecanoic acid	3	91.25	4.56	4.16	8.33	12.49
Perfluoro-n-[1,2-13C2]tetradecanoic acid	3	73.84	30.50	22.52	45.04	67.56
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	3	99.07	3.63	3.59	7.19	10.78
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	3	96.21	2.01	1.93	3.87	5.80
Perfluoro-1-[13C8]octanesulfonic acid	3	99.54	2.05	2.04	4.09	6.13
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	3	160.17	34.26	54.87	109.74	164.61
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	3	119.86	7.30	8.75	17.50	26.25
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	3	131.97	32.04	42.28	84.57	126.85
Perfluoro-1-[13C8]octanesulfonamide	3	89.86	13.61	12.23	24.46	36.69
N-methyl-d3-perfluoro-1-octanesulfonamide	3	39.32	50.06	19.68	39.36	59.04
N-ethyl-d5-perfluoro-1-octanesulfonamide	3	37.97	51.14	19.42	38.84	58.26
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	3	120.29	36.82	44.29	88.58	132.87
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	3	130.18	48.89	63.64	127.28	190.92
N-methyl-d7-perfluorooctanesulfonamidoethanol	3	41.21	84.74	34.92	69.84	104.76

Table I-4. All Media LOQVER Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
N-ethyl-d9-perfluorooctane sulfonamidoethanol	3	41.78	67.48	28.19	56.39	84.58
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	3	102.22	5.33	5.45	10.90	16.35

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Appendix J

Method Blank Recovery

Table J-1. Aqueous Method Blank Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-n-[13C4]butanoic acid	22	95.95	5.39	5.17	10.35	15.52
Perfluoro-n-[13C5]pentanoic acid	22	96.70	7.69	7.44	14.88	22.31
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	22	93.34	6.57	6.13	12.26	18.39
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	22	92.47	5.66	5.23	10.46	15.69
Perfluoro-n-[13C8]octanoic acid	22	94.09	5.86	5.52	11.04	16.55
Perfluoro-n-[13C9]nonanoic acid	22	93.77	5.29	4.96	9.91	14.87
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	22	92.05	7.25	6.68	13.35	20.03
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	22	94.32	6.72	6.34	12.67	19.01
Perfluoro-n-[1,2-13C2]dodecanoic acid	22	84.48	9.84	8.31	16.62	24.93
Perfluoro-n-[1,2-13C2]tetradecanoic acid	22	82.75	10.51	8.70	17.39	26.09
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	22	97.90	4.54	4.45	8.89	13.34
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	22	94.10	5.18	4.88	9.75	14.63
Perfluoro-1-[13C8]octanesulfonic acid	22	96.00	7.10	6.81	13.62	20.43
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	22	119.35	10.36	12.36	24.72	37.08
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	22	113.13	17.57	19.88	39.75	59.63
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	22	108.87	15.27	16.62	33.24	49.87
Perfluoro-1-[13C8]octanesulfonamide	22	80.41	16.13	12.97	25.94	38.91
N-methyl-d3-perfluoro-1-octanesulfonamide	22	59.40	19.28	11.45	22.90	34.36
N-ethyl-d5-perfluoro-1-octanesulfonamide	22	60.33	17.90	10.80	21.60	32.39
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	22	90.97	13.18	11.99	23.97	35.96
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	22	88.40	13.45	11.89	23.79	35.68
N-methyl-d7-perfluorooctanesulfonamidoethanol	22	69.03	21.74	15.01	30.02	45.03
N-ethyl-d9-perfluorooctane sulfonamidoethanol	22	64.19	19.49	12.51	25.02	37.54
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	22	101.23	9.72	9.84	19.69	29.53

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table J-2. Soil, Sediment and Biosolids Method Blank Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-n-[13C4]butanoic acid	17	101.25	3.38	3.42	6.84	10.27
Perfluoro-n-[13C5]pentanoic acid	17	94.95	7.23	6.86	13.73	20.59
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	17	97.48	5.08	4.95	9.90	14.84
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	17	94.11	2.79	2.63	5.26	7.89
Perfluoro-n-[13C8]octanoic acid	17	97.75	3.16	3.09	6.18	9.27
Perfluoro-n-[13C9]nonanoic acid	17	96.48	3.56	3.43	6.87	10.30
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	17	96.06	4.10	3.94	7.88	11.82
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	17	101.04	6.73	6.80	13.60	20.40
Perfluoro-n-[1,2-13C2]dodecanoic acid	17	88.27	12.04	10.63	21.26	31.89
Perfluoro-n-[1,2-13C2]tetradecanoic acid	17	88.49	11.49	10.17	20.34	30.51
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	17	101.46	3.87	3.93	7.85	11.78
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	17	97.46	3.41	3.32	6.64	9.96
Perfluoro-1-[13C8]octanesulfonic acid	17	100.40	5.08	5.10	10.21	15.31
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	17	143.18	4.63	6.64	13.27	19.91
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	17	120.12	6.65	7.98	15.97	23.95
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	17	107.68	6.19	6.66	13.33	19.99
Perfluoro-1-[13C8]octanesulfonamide	17	85.07	10.88	9.25	18.51	27.76
N-methyl-d3-perfluoro-1-octanesulfonamide	17	43.79	21.89	9.58	19.17	28.75
N-ethyl-d5-perfluoro-1-octanesulfonamide	17	37.28	27.20	10.14	20.28	30.41
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	17	96.61	8.89	8.59	17.19	25.78
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	17	97.35	11.41	11.11	22.21	33.32
N-methyl-d7-perfluorooctanesulfonamidoethanol	17	56.20	23.33	13.11	26.22	39.33
N-ethyl-d9-perfluorooctane sulfonamidoethanol	17	52.35	25.50	13.35	26.69	40.04
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	17	101.38	12.11	12.28	24.56	36.84

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table J-3. Tissues Method Blank Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-n-[13C4]butanoic acid	4	99.95	2.45	2.45	4.90	7.35
Perfluoro-n-[13C5]pentanoic acid	4	96.82	5.80	5.61	11.22	16.83
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	4	96.55	3.68	3.55	7.10	10.66
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	4	91.65	9.10	8.34	16.68	25.02
Perfluoro-n-[13C8]octanoic acid	4	95.50	3.52	3.36	6.73	10.09
Perfluoro-n-[13C9]nonanoic acid	4	96.47	5.96	5.75	11.50	17.25
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	4	97.10	4.94	4.80	9.60	14.40
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	4	102.25	3.13	3.20	6.40	9.60
Perfluoro-n-[1,2-13C2]dodecanoic acid	4	94.20	8.09	7.62	15.24	22.87
Perfluoro-n-[1,2-13C2]tetradecanoic acid	4	65.75	41.11	27.03	54.06	81.09
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	4	95.97	5.58	5.35	10.70	16.05
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	4	96.22	3.28	3.16	6.31	9.47
Perfluoro-1-[13C8]octanesulfonic acid	4	99.65	2.21	2.20	4.40	6.60
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	4	199.25	13.01	25.93	51.86	77.78
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	4	126.50	3.51	4.43	8.87	13.30
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	4	167.75	35.92	60.26	120.51	180.77
Perfluoro-1-[13C8]octanesulfonamide	4	110.00	7.42	8.16	16.33	24.49
N-methyl-d3-perfluoro-1-octanesulfonamide	4	37.10	34.93	12.96	25.92	38.88
N-ethyl-d5-perfluoro-1-octanesulfonamide	4	37.45	50.68	18.98	37.96	56.94
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	4	156.00	9.93	15.49	30.98	46.48
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	4	191.00	4.06	7.75	15.49	23.24
N-methyl-d7-perfluorooctanesulfonamidoethanol	4	5.32	128.22	6.82	13.64	20.46
N-ethyl-d9-perfluorooctane sulfonamidoethanol	4	26.27	73.17	19.22	38.45	57.67
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	4	94.20	17.31	16.30	32.61	48.91

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Table J-4. All Media Method Blank Spike Recovery

Compound	n =	Mean Percent Recovery	RSD [1]	SD [2]	2x SD [3]	3x SD [4]
Perfluoro-n-[13C4]butanoic acid	3	99.05	2.78	2.76	5.52	8.28
Perfluoro-n-[13C5]pentanoic acid	3	96.16	1.09	1.05	2.10	3.14
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	3	95.79	2.27	2.17	4.34	6.51
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	3	92.74	1.35	1.25	2.51	3.76
Perfluoro-n-[13C8]octanoic acid	3	95.78	1.93	1.84	3.69	5.53
Perfluoro-n-[13C9]nonanoic acid	3	95.57	1.64	1.56	3.13	4.69
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	3	95.07	2.81	2.67	5.33	8.00
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	3	99.20	4.31	4.27	8.54	12.82
Perfluoro-n-[1,2-13C2]dodecanoic acid	3	88.98	5.50	4.90	9.80	14.69
Perfluoro-n-[1,2-13C2]tetradecanoic acid	3	79.00	14.97	11.83	23.66	35.48
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	3	98.45	2.83	2.78	5.57	8.35
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	3	95.93	1.77	1.70	3.40	5.10
Perfluoro-1-[13C8]octanesulfonic acid	3	98.68	2.39	2.35	4.71	7.06
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	3	153.93	26.65	41.02	82.04	123.05
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	3	119.92	5.58	6.69	13.37	20.06
1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	3	128.10	26.81	34.34	68.69	103.03
Perfluoro-1-[13C8]octanesulfonamide	3	91.83	17.32	15.91	31.82	47.73
N-methyl-d3-perfluoro-1-octanesulfonamide	3	46.76	24.47	11.44	22.88	34.32
N-ethyl-d5-perfluoro-1-octanesulfonamide	3	45.02	29.45	13.26	26.52	39.78
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	3	114.52	31.46	36.03	72.06	108.09
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	3	125.58	45.25	56.83	113.66	170.48
N-methyl-d7-perfluorooctanesulfonamidoethanol	3	43.52	77.43	33.69	67.39	101.08
N-ethyl-d9-perfluorooctane sulfonamidoethanol	3	47.60	40.75	19.40	38.80	58.19
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	3	98.94	4.15	4.10	8.21	12.31

py_Log Date 8/9/2021 10:16:00 AM

Notes:

1. RSD = Relative Standard Deviation
2. SD = Standard Deviation
3. 2X SD = 2 times Standard Deviation
4. 3X SD = 3 times Standard Deviation

Appendix K

Holding Time Study (USEPA Report)

PFAS Holding Time in Aqueous, Solid, and Biosolids Samples Study Report

Prepared for

Adrian Hanley
U.S. Environmental Protection Agency
Office of Water
Office of Science and Technology
Engineering and Analysis Division (4303T)
1200 Pennsylvania Avenue, NW
Washington, DC 20460

Prepared by

General Dynamics Information Technology
6361 Walker Lane
Suite 300
Alexandria, VA 22310

Prepared under

EPA Scientific and Technical Support Contract EP-C-17-024

September 21, 2021

Executive Summary

Per- and polyfluoroalkyl substances (PFAS) have been found to have environmental persistence and mobility, and have been routinely detected at trace levels in various environmental media. For this reason, there exists an immediate need to evaluate groundwater, wastewater, surface water, soil, sediment, biosolids, landfill leachate, and tissue matrices. Currently there are no published methods for these various media; therefore, the Department of Defense (DOD) has completed a single-laboratory validation study (SLVS) of a method for the analysis of PFAS in these media using isotope dilution liquid chromatography mass spectrometry/mass spectrometry (LC-MS/MS) prior to beginning a multi-laboratory validation study (MLVS) and with an end goal to submit a method and supporting data package to the EPA Office of Water (OW) for consideration as a 1600-Series method. As part of the SLVS validation, a holding time study was designed, and EPA will use the results of this study to establish the holding times and preservation conditions for a new EPA PFAS Method.

The 2020 holding time study was performed by SGS AXYS funded through a contract with the U.S. Army Engineering and Support Center, Huntsville Engineering and Support Center (CEHNC), U.S. Army Corps of Engineers (USACE) and by the EPA Engineering and Analysis Division (EAD) through a GDIT purchase order to SGS AXYS. The study data evaluation and this final report was performed by GDIT through a separate contract to EPA EAD.

The goal of the study was to assess how long environmental samples and extracts of samples can be held, at various temperatures, before PFAS degrade, become unextractable, or otherwise change concentrations so that the samples are no longer representative of the media from which they are derived.

The study included one groundwater, one soil, one sediment, and one biosolids sample matrix as well as extracts of each of these matrices. The matrices were spiked at the mid-level concentrations and analyzed with three (3) replicate samples for each time increment. The samples were put into the appropriate sample containers as though they were field samples.

SGS AXYS performed a separate holding time in 2018, which included reagent water, one river surface water and two wastewater effluents. That study was designed to cover different sample containers, three storage temperatures, a holding time of up to 180 days and tested 29 of the 40 PFAS compounds tested by the current DOD study. EPA decided to incorporate the data acquired from the AXYS study with the data acquired in the DOD study. For the purpose of this report, only the data that matched the design of the DOD study, in containers, storage temperature and storage length of time, were included in the statistical comparison. The AXYS study only tested the storage conditions for the actual samples and not the storage conditions for the extracts; therefore, the statistical analysis for extracts in this report consists only of the results gathered from the DOD study.

Based on the statistical analysis of the data gathered, EPA determined that aqueous samples may be held for up to 28 days when stored at 4 ºC, with the caveat that the study data showed signs of transformation of some precursors to other PFAS when the sample is stored beyond 7 days, and this is likely to increase the observed concentrations of some PFAS compounds if precursors are present in the sample. For greater stability of PFAS compounds, the aqueous samples can be stored at -20 ºC for up to 90 days.

For biosolids, all PFAS compounds were stable for up to 90 days when stored at either 4 ºC or -20 ºC. Because microbiological activity in biosolids samples at 0 - 6 ºC may lead to production of gases which may cause the sample to be expelled from the container when it is opened, as well as producing noxious odors, EPA strongly recommends that samples be frozen if they need to be stored for more than a few days before extraction.

For solids samples, EPA found that samples could be held for 90 days when stored at either 4 ºC or -20 ºC, except when analyzing for NFDHA, which was not stable at either of the temperatures tested and must be analyzed as soon as possible after collection if it is an analyte of concern.

Once the PFAS compounds are extracted, they are stable in the extracts for up to 90 days when stored at 4 ºC, with the exception of NFDHA in solids and the ether sulfonates in aqueous samples, which are only stable for 28 days. (Although the extracts were analyzed at 29 days, EPA has decided to maintain consistency with holding times for other analytes that are often specified in multiples of 7, such that 28 days would be 4 weeks).

The holding times for each matrix and storage temperature are summarized below. Results are further discussed in Sections 4 and 5 of this report.

Summary of Holding Times per Matrix and Storage Temperature				
	Stored at 4 ºC		**Stored at -20 ºC**	
Matrices	**Holding Time**	**Exceptions**	**Holding Time**	**Exceptions**
Samples				
Aqueous	28 days	Precursor degradation after 7 days	90 days	-
Solids	90 days	NFDHA - analyze as soon as possible	90 days	NFDHA - analyze as soon as possible
Biosolids	90 days	-	90 days	
Extracts				
Aqueous	90 days	28 days for ether sulfonates	NA	-
Solids	90 days	28 days for NFDHA	NA	-
Biosolids	90 days	-	NA	-

Table of Contents

List of Tables

List of Figures

Section 1.0 Introduction and Background

Per- and polyfluoroalkyl substances (PFAS) have been widely used for decades in industrial processes and consumer products. Due to their environmental persistence and mobility, the presence of some PFAS has been routinely detected at trace levels in various environmental media. U.S. Environmental Protection Agency (EPA) has published a recommended Health Advisory Level for two PFAS, perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS), and several states have established or promulgated standards for these and other PFAS. Currently there are no published methods for the scope of media proposed for this study. There exists an immediate need to evaluate these media (e.g., groundwater, wastewater, surface water, soil, sediment, biosolids, landfill leachate) using a published method that is single- and multi-laboratory validated that can achieve the sensitivity and defensibility needed to support environmental decision making.

The Department of Defense (DOD) is currently completing a single-laboratory validation study (SLVS) of a method for the analysis of PFAS in these media using isotope dilution liquid chromatography mass spectroscopy/mass spectroscopy (LC-MS/MS). DOD will use the results of the SLVS validation study to make any necessary adjustments to the method prior to a multi-laboratory validation study (MLVS) effort that is proposed to follow. The end goal is to submit a method and supporting data package to the EPA Office of Water (OW) for consideration as a 1600-Series method, including the information that EPA Office of Land and Emergency Management (OLEM) needs to consider for an EPA SW-846 method. OW will distribute the method/data package to OLEM. A holding time study was required as part of the SLVS. EPA will use the results of this study to establish the holding times and preservation conditions for a new EPA PFAS method.

Section 2.0 Study Scope and Details

2.1 Study Scope

A holding time study was performed in 2020 for the single-laboratory validation study for PFAS by SGS AXYS funded through a contract with the U.S. Army Engineering and Support Center, Huntsville Engineering and Support Center (CEHNC), U.S. Army Corps of Engineers (USACE) and by the EPA EAD through a GDIT PO. The study data evaluation and final report was performed by GDIT through a separate contract to EPA EAD. The goal of the holding time study was to assess how long environmental samples and extracts of samples can be held, at various temperatures, before PFAS degrade, become unextractable, or otherwise change concentrations so that the samples are no longer representative of the media from which they are derived.

The study analyzed the stability of PFAS compounds on groundwater, soil, sediment, and biosolid matrices stored at two different temperatures for up to 90 days, as well as the stability of the compounds in the extracts for each of these matrices. The matrices were spiked at the mid-level concentrations and three (3) replicate samples were analyzed at each time increment. The telomer alcohols and perfluorooctane sulfonamidoethanols (FOSEs) are PFAS precursors that were included in standards added to samples for evaluation of holding times. The samples were put into the appropriate sample containers as though they were field samples.

SGS AXYS had performed a separate holding time study in 2018, which included reagent water, one surface water, and two wastewater effluents. That study was designed to cover three different sample containers (polypropylene, glass, and high-density polyethylene), three storage temperatures (20 ºC, 4 ºC and -20 ºC), and holding times up to 180 days. However, this study also tested only 29 of the 40 PFAS compounds tested by the DOD study. The compounds not included were the per- and polyfluoroether carboxylates, ether sulfonates, and fluorotelomer carboxylates. EPA decided to incorporate the data acquired from the 2018 AXYS study with the data acquired from the 2020 study. For the purpose of this report, only the data that matched the design of the 2020 study, in containers, storage temperature and storage time, were included in the statistical comparison. The 2018 study only tested the storage conditions for the actual samples and not the storage conditions for the extracts; therefore, the statistical analysis for extracts in this report consist only of the results gathered from the 2020 study.

2.2 Analytical Method and Analytes

Aqueous samples were extracted into a weak anion-exchange (WAX) SPE and cleaned up with carbon prior to analysis. Solid samples were extracted by shaking in a methanolic ammonium hydroxide solution and the extract cleaned up with carbon and WAX SPE cartridge. Extracts were analyzed by LC-MS/MS and quantitated by isotope dilution, in general accordance with the procedures described in Draft Method 1633. Up to thirty containers were prepared for each matrix; therefore, the multiple extractions required for analysis of each sample were performed on individual sample aliquots. The target compounds are listed in Table 1 according to their compound classification system, which is used in the discussion of results.

Table 1. Names, Abbreviations, and CAS Registry Numbers for Native Per- and Polyfluoroalkyl Substances (PFAS)

Abbreviation	***Name – Acid/Neutral Form***	***CAS#***
Perfluoroalkyl carboxylates		
PFBA	Perfluorobutanoic acid	375-22-4
PFPeA	Perfluoropentanoic acid	2706-90-3
PFHxA	Perfluorohexanoic acid	307-24-4
PFHpA	Perfluoroheptanoic acid	375-85-9
PFOA	Perfluorooctanoic acid	335-67-1
PFNA	Perfluorononanoic acid	375-95-1

Table 1. Names, Abbreviations, and CAS Registry Numbers for Native Per- and Polyfluoroalkyl Substances (PFAS)		
Abbreviation	***Name – Acid/Neutral Form***	***CAS#***
PFDA	Perfluorodecanoic acid	335-76-2
PFUnA	Perfluoroundecanoic acid	2058-94-8
PFDoA	Perfluorododecanoic acid	307-55-1
PFTrDA	Perfluorotridecanoic acid	72629-94-8
PFTeDA	Perfluorotetradecanoic acid	376-06-7
Perfluoroalkyl sulfonates		
PFBS	Perfluorobutanesulfonic acid	375-73-5
PFPeS	Perfluoropentanesulfonic acid	2706-91-4
PFHxS	Perfluorohexanesulfonic acid	355-46-4
PFHpS	Perfluoroheptanesulfonic acid	375-92-8
PFOS	Perfluorooctanesulfonic acid	1763-23-1
PFNS	Perfluorononanesulfonic acid	68259-12-1
PFDS	Perfluorodecanesulfonic acid	335-77-3
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
Fluorotelomer sulfonates		
4:2FTS	4:2 fluorotelomersulfonic acid	757124-72-4
6:2FTS	6:2 fluorotelomersulfonic acid	27619-97-2
8:2FTS	8:2 fluorotelomersulfonic acid	39108-34-4
Perfluorooctane sulfonamides		
PFOSA	Perfluorooctanesulfonamide	754-91-6
NMeFOSA	N-Methylperfluorooctanesulfonamide	31506-32-8
NEtFOSA	N-Ethylperfluorooctanesulfonamide	4151-50-2
Perfluorooctane sulfonamidoacetic acids		
NMeFOSAA	N-Methylperfluorooctanesulfonamidoacetic acid	2355-31-9
NEtFOSAA	N-Ethylperfluorooctanesulfonamidoacetic acid	2991-50-6
Perfluorooctane sulfonamidoethanols		
NMeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
NEtFOSE	N-Ethylperfluorooctanesulfonamidoethanol	1691-99-2
Per- and Polyfluoroether carboxylates*		
HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6
ADONA	4,8-dioxa-3H-perfluorononanoic acid	919005-14-4
PFMPA	Perfluoro-4-methoxypropanoic acid	377-73-1
PFMBA	Perfluoro-3-methoxybutanoic acid	863090-89-5
NFDHA	Perfluoro-3,6-dioxaheptanoic acid	151772-58-6
Ether sulfonates*		
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
PFEESA	Perfluoro(2-ethoxyethane) sulfonic acid	113507-82-7
Fluorotelomer carboxylates*		
3:3FTCA	2H, 2H, 3H, 3H-perfluorohexanoic acid	356-02-5
5:3FTCA	2H, 2H, 3H, 3H-perfluorooctanoic acid	914637-49-3
7:3FTCA	2H, 2H, 3H, 3H-perfluorodecanoic acid	812-70-4

*These compounds were not included in the 2018 holding time study performed by SGS AXYS.

2.3 Holding Time and Preservation Conditions

The study was designed to investigate the stability of PFAS in reagent water, surface water, wastewater, groundwater, soil, sediment, and biosolids matrices. The parameters investigated were sample and extract storage conditions (≤ -20 ºC or 4 ºC), and length of storage (from Day 0 up to 90 days after preparation). All samples were collected in HDPE containers. Statistical relevance of the results was evaluated by the use of replicate samples for each set of conditions. All samples were spiked with known amounts of the target analytes at the beginning of the study to provide concentrations of all target compounds that could be reliably quantified by the analytical methods. For the 2020 study, the spiked samples were stored at either ≤ -20 ºC or 4 ºC overnight and the first analyses were conducted the following day. For the 2018 study, the spiked samples were analyzed on the first day, and then stored at either ≤ -20 ºC or 4 ºC for the duration of the study. The analyte background levels for each sample were determined by triplicate analysis and the average value was subtracted from the spiked value. The summary of background levels for all the matrices can be found in Appendix A at the end of the report.

Table 2. Holding Time Study Conditions and Parameters

Matrix	Storage Temp.	Number of Replicates				
		Day 0	Day 7	Day 14	Day 28	Day 90
Phase I. Sample Storage						
Surface Water	4 ºC	3	3	3	3	3
	-20 ºC	0	3	3	3	3
Wastewater	4 ºC	3	3	3	3	3
	-20 ºC	0	3	3	3	3
Groundwater Sample	4 ºC	3	3	3	3	3
	-20 ºC	3	3	3	3	3
Soil Sample	4 ºC	3	3	3	3	3
	-20 ºC	3	3	3	3	3
Sediment Sample	4 ºC	3	3	3	3	3
	-20 ºC	3	3	3	3	3
Biosolids Sample	4 ºC	3	3	3	3	3
	-20 ºC	3	3	3	3	3
Phase II. Extract Storage						
Groundwater Extract	4 ºC	3	3	3	3	3
Soil Extract	4 ºC	3	0	3	3	3
Sediment Extract	4 ºC	3	0	3	3	3
Biosolids Extract	4 ºC	3	0	3	3	3

2.4 Preparation of Samples for the Holding Time Study

Spiking Solution

All samples were spiked at least 10 times higher than the LOQ. The samples were spiked with all the 40 target compounds (except for the 2018 study), 24 extracted internal standards (EIS) (a.k.a. labeled compounds) and seven (7) non-extracted internal standards (NIS). The samples contained the same amount of target analytes as the on-going precision and recovery sample (OPR) used as part of the batch quality control, but at a higher concentration. The spiking solution and NIS were prepared in 100% methanol, while the EIS solution was prepared in methanol with 0.4% isopropanol. All the final standard concentrations were high enough so that less than 100 µL of each solution were used for each sample. The spike levels were as follows:

Table 3. Nominal Concentrations of Spiking Solutions Added to Samples		
	Amount Added (ng)	
Analyte	**DOD Study**	**AXYS Study**
Target Compounds		
PFBA	20	79.4
PFPeA	10	39.8
PFHxA	5	19.9
PFHpA	5	19.9
PFOA	5	19.9
PFNA	5	20
PFDA	5	19.9
PFUnA	5	19.9
PFDoA	5	20.1
PFTrDA	5	20.2
PFTeDA	5	19.9
PFBS	5	19.7
PFPeS	5	18.8
PFHxS	5	20
PFHpS	5	19
PFOS	5	19.8
PFNS	5	19.2
PFDS	5	19.8
PFDoS	5	19.4
4:2FTS	20	80.2
6:2FTS	18	72.1
8:2FTS	20	80
PFOSA	5	19.9
NMeFOSA	5.75	23
NEtFOSA	12.5	50
NMeFOSAA	5	20
NEtFOSAA	5	20
NMeFOSE	50	19.8
NEtFOSE	37.5	14.9
HFPO-DA	19	N/A
ADONA	20	N/A
9Cl-PF3ONS	20	N/A
11Cl-PF3OUdS	20	N/A
3:3FTCA	20	N/A
5:3FTCA	125	N/A
7:3FTCA	125	N/A
PFEESA	5	N/A
PFMBA	5	N/A
PFMPA	10	N/A
NFDHA	10	N/A
Extracted Internal Standards (EIS)		
$^{13}C_4$-PFBA	40	
$^{13}C_5$-PFPeA	20	
$^{13}C_5$-PFHxA	10	
$^{13}C_4$-PFHpA	10	

Table 3. Nominal Concentrations of Spiking Solutions Added to Samples		
	Amount Added (ng)	
Analyte	DOD Study	AXYS Study
$^{13}C_8$-PFOA	10	
$^{13}C_9$-PFNA	5	
$^{13}C_6$-PFDA	5	
$^{13}C_7$-PFUnA	5	
$^{13}C_2$-PFDoA	5	
$^{13}C_2$-PFTeDA	5	
$^{13}C_3$-PFBS	10	
$^{13}C_3$-PFHxS	10	
$^{13}C_8$-PFOS	10	
$^{13}C_2$-4:2FTS	20	
$^{13}C_2$-6:2FTS	20	
$^{13}C_2$-8:2FTS	20	
$^{13}C_8$-PFOSA	10	
D_3-NMeFOSA	10	
D_5-NEtFOSA	10	
D_3-NMeFOSAA	20	
D_5-NEtFOSAA	20	
D_7-NMeFOSE	100	
D_9-NEtFOSE	100	
$^{13}C_3$-HFPO-DA	40	
Non-extracted Internal Standards (NIS)		
$^{13}C_3$-PFBA	20	
$^{13}C_2$-PFHxA	10	
$^{13}C_4$-PFOA	10	
$^{13}C_5$-PFNA	5	
$^{13}C_2$-PFDA	5	
$^{18}O_2$-PFHxS	10	
$^{13}C_4$-PFOS	10	

Aqueous Sample Storage and Preparation

The laboratory received one groundwater sample from the Colorado School of Mines on May 21, 2020. The sample collection and shipping were arranged by Hydrogeologic, Inc. The sample was collected in thirty (30) separate 1-L HDPE containers. The shipment was received at a temperature of 19ºC.

On September 16, 2020, the sample was homogenized and subsampled into 59 individual 500-mL HDPE containers. The sample was given the internal tracking number L33488, with each container having a dash followed by a number (e.g., L33488-1). Five sets of three containers were spiked with the full list of target compounds. Samples L33488-1, -2, -3, -7, -8, -9, -13, -14, -15, -19, -20, -21, -25, -26, and -27 were stored in a refrigerator at 4 °C, while samples L33488-4, -5, -6, -10, -11, -12, -16, -17, -18, -22, -23, -24, -28, -29, and -30 were stored in a freezer at ≤ -20 °C. The samples were stored until analysis which started on September 17, 2020, which was established as Day 0. The samples were extracted and analyzed as follows:

Sample Storage Temperature Summary

Storage Day	4 °C	≤ -20 °C
Day 0 (9/17/2020)	L33488-1, -2, -3	L33488-4, -5, -6
Day 7	L33488-7, -8, -9	L33488-10, -11, -12
Day 14	L33488-13, -14, -15	L33488-16, -17, -18
Day 28	L33488-19, -20, -21	L33488-22, -23, -24
Day 90	L33488-25, -26, -27	L33488-28, -29, -30

The aqueous samples were prepared by adjusting the pH to 6.5 ± 0.5 and then passing the sample through a WAX SPE cartridge. The compounds were eluted with 1% methanolic ammonium hydroxide. Acetic acid and carbon were added to the eluate , and after mixing, the extract was filtered through a 0.2-µm filter into a collection tube for analysis and storage.

After analysis on Day 0, one set of three extracts was stored in polypropylene tubes at 4 °C and analyzed at storage day 7, 15, 29, and 90 from the date of original analysis. These extracts were given a new internal tracking number for each analysis day and analyzed as follows:

Extract Storage Summary

Storage Day	Sample ID
Day 7	L33593-1, -2, -3
Day 15	L33594-1, -2, -3
Day 29	L33595-1, -2, -3
Day 90	L33596-1, -2, -3

For the samples used in the 2018 holding time study, the laboratory measured out 50 mL of each sample and then diluted them to 500 mL with deionized water prior to the sample preparation described above. This step was taken because of initial concerns about the effects of adding 4 mL of methanol to the 50-mL sample prior to the solid-phase extraction process. The laboratory had previously determined that using 4 mL of methanol in a 500-mL sample did not cause analyte breakthrough, so they diluted the holding time samples to that volume as a precaution.

Solid Samples Storage and Preparation

The laboratory analyzed one soil (5 g dry-weight), one sediment (5 g dry-weight), and one biosolids (0.5 g dry-weight) sample at different time intervals.

The soil sample was purchased from the North American Proficiency Testing (NAPT) Program and had already been air dried, grounded, and sieved. The soil was received on 01/14/2020 as a bulk sample, at 5 °C. The sample was then subsampled on 9/9/2020 into 24 separate HDPE containers with internal tracking number L33556. After subsampling, the soil was wetted with 2.5 g of deionized water to simulate a normal soil sample and allow for the spiked compounds to be better distributed within the sample. The biosolids sample was received on 01/08/2020 from Hydrogeologic, Inc. in eight (8) 60-mL amber glass containers at 0.8 °C. The sample was composited, homogenized, and subsampled into 24 separate HDPE containers with internal tracking number L33557 on 09/10/2020. The sediment sample came from a river basin containing high total organic carbon (TOC) and was received on 01/08/2020 in nine (9) 60-mL amber glass containers at 0.8 °C from Hydrogeologic, Inc. The laboratory composited all the contents together, homogenized the sample and subsampled it into 24 separate HDPE containers with internal tracking number L33558 on 09/11/2020. The percent moisture was determined for the biosolids and sediment samples prior to subsampling in order to establish the wet-weight sample size that would give the required dry-weight before analysis. The samples were extracted and analyzed as follows:

	Sample Storage Temperature	
Storage Day	**4 °C**	**≤ -20 °C**
Soil Sample		
Day 0	L33556-1, -2, -3	L33556-4, -5, -6
Day 14	L33556-7, -8, -9	L33556-10, -11, -12
Day 28	L33556-13, -14, -15	L33556-16, -17, -18
Day 90	L33556-19, -20, -21	L33556-22, -23, -24
Biosolids Sample		
Day 0	L33557-1, -2, -3	L33557-4, -5, -6
Day 14	L33557-7, -8, -9	L33557-10, -11, -12
Day 28	L33557-13, -14, -15	L33557-16, -17, -18
Day 90	L33557-19, -20, -21	L33557-22, -23, -24
Sediment Sample		
Day 0 (9/15/2020)	L33558-1, -2, -3	L33558-4, -5, -6
Day 14	L33558-7, -8, -9	L33558-10, -11, -12
Day 28	L33558-13, -14, -15	L33558-16, -17, -18
Day 90	L33558-19, -20, -21	L33558-22, -23, -24

The solid samples were extracted by shaking into a 0.3% methanolic ammonium hydroxide solution, in general accordance with the procedures described in Draft Method 1633. The extract was then cleaned up with carbon. The cleaned extract was concentrated under nitrogen, then diluted with water, pH adjusted and passed through a WAX SPE cartridge. The compounds were eluted with 1% methanolic ammonium hydroxide. Acetic acid was added to the sample extract, mixed, then filtered through a 0.2-µm filter into a collection tube for analysis and storage.

After analysis on Day 0, three extracts for each of the solid matrices were stored in polypropylene tubes at 4 °C and analyzed at storage day 15, 29 and 90 from the date of original analysis. Aliquots of these extracts were given new internal tracking numbers for each new analysis day and analyzed as follows:

	Sample ID		
Storage Day	**Soi**	**Sediment**	**Biosolids**
Day 15	L33597-1, -2, -3	L33600-1, -2, -3	L33603-1, -2, -3
Day 29	L33598-1, -2, -3	L33601-1, -2, -3	L33604-1, -2, -3
Day 90	L33599-1, -2, -3	L33602-1, -2 -3	L33605-1, -2, -3

Section 3.0 Statistical Analysis

3.1 Classification of Compounds

PFAS compounds mainly are divided into two classes: polymers and non-polymers. Polymers are formed by the combination of monomers in a repeating pattern; however, they are not considered to pose as much of an immediate human health or ecological risk as non-polymers. Because non-polymer PFAS compounds are the most commonly detected in the environment, they are the class on which detection is mostly focused. The non-polymers are divided into two subclasses: perfluoroalkyl and polyfluoroalkyl.

Perfluoroalkyl substances are fully fluorinated alkane molecules with a chain of two or more carbon atoms with a functional group attached at one end. The functional groups are commonly either carboxylates or sulfonates, but other forms are also possible. They are divided into two main classes: perfluoroalkyl acids (PFAAs) and perfluoroalkane sulfonamides (FOSAs).

PFAAs are the compounds most commonly tested in the environment since they can be formed through degradation of polyfluoroalkyl substances, referred to as "precursors." PFAAs are further subdivided into two main groups: perfluoroalkyl carboxylic acids or perfluoroalkyl carboxylates (PFCAs), and perfluoroalkane sulfonic acids or perfluoroalkane sulfonates (PFSAs). PFAAs can be described as either short-chain or long-chain. For the purposes of this report, short-chain refers to perfluoroalkyl carboxylic acids with seven or fewer carbons (e.g., PFBA, PFHxA) and perfluoroalkane sulfonates with five or fewer carbons (e.g., PFBS, PFPeS), while long-chain refers to perfluoroalkyl carboxylic acids with eight or more carbons (e.g., PFOA, PFNA) and perfluoroalkane sulfonates with six or more carbons (e.g., PFOS, PFNS).

Polyfluoroalkyl substances are not fully fluorinated, but instead have a non-fluorine atom (typically hydrogen or oxygen) attached to at least one carbon atom in the alkyl chain, with a minimum of two carbon atoms in the chain tail that are fully fluorinated. Since the carbon-hydrogen bond is not as strong as the carbon-fluorine bond, polyfluoroalkyl substances can fragment through degradation, producing precursor compounds which have the potential of transforming into PFAAs.

Polyfluoroalkyl substances can be divided into two classes: fluorotelomers and perfluoroalkane sulfonamido substances. Fluorotelomers are polyfluoroalkyl compounds produced by fluorotelomerization and their degradation is a potential source of PFCAs in the environment. Fluorotelomers are divided into three groups which are detected in the environment: fluorotelomer alcohols (FTOH), fluorotelomer sulfonic acids (FTSA), and fluorotelomer carboxylic acids (FTCA). Perfluoroalkane sulfonamido substances are divided into the following groups: N-alkyl perfluoroalkane sulfonamides, perfluoroalkane sulfonamidoethanols, N-alkyl perfluoroalkane sulfonamidoethanols, N-alkyl perfluoroalkane sulfonamido acetic acids, and perfluoroalkane sulfonamido acetic acids.

Names for the anionic and acid forms nomenclatures are commonly used interchangeably; however, they have different physical and chemical properties. Most PFAAs are found in the anionic form in the environment and although the acid form is usually reported, the method actually measures the anionic form. The PFAS compounds analyzed under this method were reported as either the acid or neutral form names.

Many PFAS compounds have linear and branched isomers depending on their manufacturing process. For example, fluorotelomerization produces linear PFAA isomers while electrochemical fluorination (ECF) produces a mixture of branched and linear isomers. The method tested both types of isomers; however, the results are reported as the sum of both linear and branched for the following compounds: PFOS, PFHxS, NEtFOSAA, and NMeFOSAA.

Because of PFAS persistence and bioaccumulation, replacement PFAS have been commercially introduced. Some of these replacement compounds have also been detected in the environment and as such, some of them have been included as part of this study (e.g., ADONA, PFMBA).

3.2 Statistical Tests

The number and complexity of PFAS compounds becomes a limitation when performing statistical analysis for any one group of chemicals, because these groups may behave differently under similar conditions. Therefore, holding time evaluations were performed separately for each compound, chemical group, and treatment. Identification of statistical differences for each compound was performed using multi-step processes that involved a one-way Analysis of Variance (ANOVA) and a Dunnett's post-hoc test for pairwise differences among holding times.

In the first step, an ANOVA model was fit for each treatment, then the F-test from that model was used to determine whether there was an overall difference between holding times (e.g., whether recovery at one holding time was significantly different from recovery for at least one other holding time). If the ANOVA determined that there was no difference in analyte recovery between any of the different holding times, then the evaluation of the results for that analyte and treatment was stopped.

If the ANOVA determined that there was a difference, a second step was performed, which involved pairwise comparisons between Day 0 and each of the other holding times using Dunnett's test. Dunnett's test was used to specifically compare results from one reference time (Day 0) to all other holding times, without directly comparing the individual non-zero times to each other.

Because fewer comparisons are run using Dunnett's test than for other pairwise comparison procedures, Dunnett's test yields more statistical power for the given set of analyses. In addition, because the Dunnett's test compared all of the results back to the Day 0 result, it ensured that small changes over time were not overlooked (e.g., a gradual downward trend might not be apparent when comparing results from adjacent days).

Based on the ANOVA and the Dunnett's test, GDIT established whether or not there was a statistically significant difference in the mean result for each analyte on each day of the study (e.g., 7, 14, 28 and 90), compared to the results for the analyte on Day 0. The differences for each analyte were classified as significant (Y) or not significant (N).

3.3 Magnitude of Statistical Differences

The anticipated analytical precision between compounds within a single study design can have such a degree of variation that the statistical evaluation would yield non-meaningful differences that are statistically significant for some compounds, while having meaningful differences that are not statistically significant for other compounds. Therefore, GDIT did not rely solely on the ANOVA and Dunnett results.

GDIT calculated the magnitude of the difference for each analyte, and storage time in terms of percent difference in recovery compared to the recovery on Day 0, using the following equation:

$$\%Diff = \frac{(R_x - R_0)}{R_0} * 100\%$$

where R_x is the mean recovery for Day x and R_0 is the mean recovery for Day 0 for the given analyte.

Using a 20% difference (positive or negative) in recoveries as an indication of the minimum change that could affect a decision about the data in a real-world sample, GDIT counted the number of analytes for which there was a statistically significant change (e.g., Significant [Y] or Not Significant [N] in the tables that follow) and the magnitude of that change was greater than 20%. In addition to excluding statistically significant differences of small magnitude, the 20% concentration difference also identified instances where the magnitude was large due to large analytical variability, but not statistically significant. GDIT

tabulated the number of analytes in each family that had significant differences greater than 20% and those that did not. If there was a statistically significant change with a magnitude less than 20%, GDIT did not count that change in this evaluation of holding time (e.g., a change of 5% in concentration, while perhaps statistically significant, is not sufficient reason to establish a holding time).

Twenty percent (20%) was selected by EPA as the maximum threshold because similar criteria are commonly used in calibration models within many EPA analytical methods. While other threshold values have been utilized in other holding time studies, EPA felt that when considering the variability associated with this analysis and the difficult and broad range of matrices such as biosolids included in this study, that 20% was an appropriate choice. Other threshold values could be employed and would alter the conclusions.

While other approaches to holding times determinations are available, ANOVA, along with the use of a Dunnett's test, was more statistically powerful than using the t-test for each compound.

Given the practical resource limitations in the study (the number matrices which needed to be covered and the number of replicate analysis which could be reasonably performed), the statistical analysis was performed at a 95% confidence level rather than the 99% confidence level used in ASTM D4841, to increase the statistical power (the ability to identify a holding time difference). The danger with this approach is that it introduces a 5% risk of concluding there is a holding time effect when there is not (rather than 1%, as it would be if the tests were run at the 99% confidence level). However, in cases where the variability between replicate analyses in this study is low, utilizing the 20% cutoff mentioned above decreases the 5% risk of concluding there is an effect when there is not.

Final Presentation of Statistical Results

The comparison results were summarized for each chemical group by determining the number of analytes that "survived" on each day of the study (e.g., the mean did not differ by more than 20% from the mean on Day 0). Each total was divided by the total number of analytes in that chemical group, to arrive at the "percent survival" for each treatment/family/day combination (Tables 64 - 67). The survival rates for all 40 target analytes in each treatment were also calculated.

Section 4.0 Study Results and Discussion

The study examined the effects of storage temperatures as well as the length of storage at those temperatures, on aqueous (surface water, wastewater, and groundwater) and solids (soil, biosolids, and sediment) samples. The samples were spiked with the analytes of interest as described in Section 2.0, but the samples also contained "background" levels of some analytes which was subtracted from the final values prior to determining the percent recoveries. The goal of both temperature treatments was to reduce the biological activity of the samples, as well as reduce the rates of any chemical reactions in the samples that might affect the analytes of interest. The storage length of time at each of the storage temperatures was also used to observe any potential changes in analyte concentration within the samples. For this purpose, the matrices were analyzed at varying points in time to determine any possible effects of holding times. The study results and the discussion of those results are below in Sections 4.1 and 4.2.

Due to the logistical considerations encountered when analyzing a large number of samples extracts, holding times often vary; therefore, any potential changes in analyte concentration within the extracts also need to be evaluated and the extract holding times established. For this reason, a variety of sample extracts were also analyzed at varying points in time to determine any possible effects of analytical delay. The results and discussion of these results are below in Section 4.3.

For the statistical analysis, each holding time day was compared separately against Day 0. It was assumed that the determined holding time following this approach would be the latest time before the first statistically significant difference from Day 0. For example, if Day 7 and Day 14 each were not statistically significantly different from Day 0 for a specific matrix/compound, but Day 28 results were different, the statistically determined holding time was set at 14 days, regardless of how the Day 90 results compare to Day 0. If the first non-zero time was significantly different from Day 0, then the statistically determined holding time was set to 0.

GDIT also examined the percent recoveries for all the replicates for each compound. For each day, the average recovery was calculated, as well as the minimum and maximum recoveries for each compound. An interim recovery limit of 70 – 130% was used for the holding time study.

4.1 Aqueous Samples

The results for both temperature preservation treatments for all aqueous samples are summarized below. The aqueous samples (i.e., reagent water, effluent #1, effluent #2, and river water) analyzed in the 2018 holding time study only include twenty-nine (29) PFAS compounds.

Reagent Water

Table 4 presents the mean recoveries for reagent water stored at 4 °C. PFOS and most of the compounds analyzed had recoveries between 70 and 130%, with a few exceptions. PFDoS had low recoveries across the board regardless of the holding day, with an average recovery of 75%, a minimum recovery of 57% and maximum of 90%. All of the fluorotelomer sulfonates and NMeFOSAA had recoveries consistently above 100%, with maximum recoveries above 130%. The recoveries decrease slightly between Day 0 and Day 90, but the recoveries remained above 100%. NMeFOSA and NEtFOSA showed decreasing recoveries after Day 7, with average recoveries ranging from 50 to 85%.

Table 4. Mean Recoveries for Reagent Water Stored at 4 ºC								
	PFAS	Mean Recovery per Storage Day					Recovery (%)	
Chemical Group	Acronym	Day 0	Day 7	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	116	114	112	114	101	99	117
	PFDA	99	103	101	108	105	93	111
	PFDoA	115	117	118	105	113	103	121
	PFHpA	118	118	115	113	97	96	119
	PFHxA	113	114	109	110	101	100	116
	PFNA	106	107	104	107	104	101	112
	PFOA	113	109	111	108	99	96	117
	PFPeA	106	108	106	116	98	96	116
	PFTeDA	109	97	99	103	100	96	113
	PFTrDA	106	100	100	106	115	97	122
	PFUnA	108	99	100	107	104	92	115
Perfluoroalkyl sulfonates	PFBS	106	102	103	110	103	101	114
	PFDS	107	112	109	102	89	79	115
	PFDoS	78	86	79	66	68	57	90
	PFHpS	98	98	104	111	103	95	113
	PFHxS	95	94	96	105	100	89	106
	PFNS	101	98	98	105	96	94	110
	PFOS	90	91	92	109	96	88	114
	PFPeS	106	104	103	114	98	97	116
Fluorotelomer sulfonates	4:2FTS	125	127	122	114	109	108	132
	6:2FTS	128	125	122	118	102	98	132
	8:2FTS	139	136	133	124	119	115	143
Perfluorooctane sulfonamides	NEtFOSA	83	71	54	67	80	52	83
	NMeFOSA	96	89	71	74	85	67	97
	PFOSA	102	111	99	101	102	97	116
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	100	102	97	102	94	87	108
	NMeFOSAA	128	125	121	120	109	99	133
Perfluorooctane sulfonamidoethanols	NEtFOSE	102	85	75	84	95	72	102
	NMeFOSE	103	93	83	84	98	82	104

Based on the ANOVA and the Dunnett's test, there were six (6) compounds with sufficient statistically significant differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 5 and 6).

Table 5. Statistical Difference for Reagent Water Stored at 4 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 15	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	N	Y	N	Y	7
	PFDA	N	N	N	N	90
	PFDoA	N	N	Y	N	14
	PFHpA	N	N	Y	Y	14
	PFHxA	N	N	N	Y	28
	PFNA	N	N	N	N	90

Table 5. Statistical Difference for Reagent Water Stored at 4 ºC, Compared to Day 0						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 15**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFOA	N	N	N	Y	28
	PFPeA	N	N	Y	Y	14
	PFTeDA	Y	Y	Y	Y	0
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	Y	28
	PFDoS	N	N	Y	N	14
	PFHpS	N	N	Y	N	14
	PFHxS	N	N	Y	N	14
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	N	14
	PFPeS	N	N	Y	Y	14
Fluorotelomer sulfonates	4:2FTS	N	N	N	Y	28
	6:2FTS	N	N	N	Y	28
	8:2FTS	N	N	Y	Y	14
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	Y	Y	0
	NMeFOSE	Y	Y	Y	Y	0
Perfluorooctane sulfonamides	NEtFOSA	Y	Y	Y	N	0
	NMeFOSA	N	Y	Y	Y	7
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	Y	28

Based on a percent difference of 20%, NEtFOSE, NMeFOSA, and NEtFOSA have a holding time of 7 days, while PFOS has a holding time of 14 days while the remaining compounds would have a holding time of 90 days. The percent difference for PFOS on Day 28 was 21%; however, it decreased to below 20% at Day 90.

Table 6. Selected Holding Time Based on 20% Difference for Reagent Water Sample Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90

Table 6. Selected Holding Time Based on 20% Difference for Reagent Water Sample Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	N	N	N	N	90
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	N	14
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	Y	N	N	7
	NMeFOSA	N	Y	Y	N	7
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	Y	N	N	7
	NMeFOSE	N	N	N	N	90

Table 7 presents the mean recoveries for reagent water stored at -20 ºC. Most of the compounds analyzed had recoveries between 70 and 130%, with a few exceptions. Results were comparable with those seen in the samples stored at 4 °C with the exception of PFTrDA. This compound had high recovery after storage Day 28.

Table 7. Mean Recoveries for Reagent Water Stored at -20 ºC							
		Mean Recovery per Storage Day				**Recovery (%)**	
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Min**	**Max**
Perfluoroalkyl carboxylates	PFBA	115	113	114	101	101	116
	PFDA	101	101	106	103	98	107
	PFDoA	114	127	106	114	97	131
	PFHpA	115	116	108	96	94	119
	PFHxA	111	112	108	99	97	115
	PFNA	107	106	106	103	101	111
	PFOA	110	109	108	105	104	112
	PFPeA	108	107	116	97	96	119
	PFTeDA	103	105	116	107	101	123
	PFTrDA	100	101	109	132	96	139
	PFUnA	105	97	107	101	94	111
Perfluoroalkyl sulfonates	PFBS	105	102	108	102	99	110
	PFDS	106	111	93	94	82	115
	PFDoS	77	87	62	69	58	92
	PFHpS	100	107	110	105	99	112
	PFHxS	97	95	105	101	93	109
	PFNS	97	98	102	96	95	106
	PFOS	93	92	110	97	90	113
	PFPeS	106	101	115	98	97	119
Fluorotelomer sulfonates	4:2FTS	127	129	113	107	100	133
	6:2FTS	124	125	113	104	100	128
	8:2FTS	138	140	130	114	112	149

Table 7. Mean Recoveries for Reagent Water Stored at -20 ºC							
		Mean Recovery per Storage Day				Recovery (%)	
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Min	Max
Perfluorooctane sulfonamides	NEtFOSA	76	70	83	91	66	96
	NMeFOSA	90	85	89	99	83	102
	PFOSA	109	104	103	103	99	112
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	98	100	102	94	90	103
	NMeFOSAA	124	119	117	109	106	127
Perfluorooctane sulfonamidoethanols	NEtFOSE	90	83	95	97	80	103
	NMeFOSE	95	91	93	99	85	103

Based on the ANOVA and the Dunnett's test, there were seven (7) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days, as described at the beginning of Section 4.0; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 8 and 9).

Table 8. Statistical Difference for Reagent Water Stored at -20 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	N	Y	N	Y	7
	PFDA	N	N	Y	N	14
	PFDoA	N	Y	N	N	7
	PFHpA	N	N	Y	Y	14
	PFHxA	N	N	N	Y	28
	PFNA	N	N	N	N	90
	PFOA	N	N	N	Y	28
	PFPeA	N	N	Y	Y	14
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	Y	28
	PFUnA	N	Y	N	N	7
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	Y	Y	14
	PFDoS	N	N	Y	N	14
	PFHpS	N	Y	Y	Y	7
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	Y	14
	PFPeS	N	N	Y	Y	14
Fluorotelomer sulfonates	4:2FTS	N	N	N	Y	28
	6:2FTS	N	N	Y	Y	14
	8:2FTS	N	N	N	Y	28
Perfluorooctane sulfonamides	NEtFOSA	N	Y	N	N	7
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	Y	28
	NMeFOSAA	N	N	Y	Y	14
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	N	N	0
	NMeFOSE	N	Y	N	N	7

Based on a percent difference of 20%, PFDoS and PFOS have a holding time of 14 days, PFDS has a holding time of 28 days, while the remaining compounds have a holding time of 90 days. It is important

to note that although the percent difference for PFDoS is above 20% at Day 28, technically giving it a holding time of 14 days, the value was only 21%, and decreased to below 20% on Day 90.

Table 9. Selected Holding Time Based on 20% Difference for Reagent Water Sample Stored at -20 ºC

Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Chosen HT
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	Y	28
	PFDoS	N	N	Y	N	14
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	N	14
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	N	90
	NMeFOSE	N	N	N	N	90

Wastewater Samples

The laboratory analyzed two different effluent samples. Table 10 presents the mean recoveries for Effluent Sample #1 stored at 4 ºC. Most of the compounds analyzed had recoveries between 70 and 130%, with the following exceptions. PFDoS and NEtFOSA had low recoveries across the board, regardless of the holding day, with an average recovery of 61% and 76%, respectively. Recoveries ranged from 55 – 72% for PFDoS and 61 – 86% for NEtFOSA. The fluorotelomer sulfonates as a whole had recoveries consistently above 100%, with maximum recoveries above 130%. The recoveries decrease slightly between Day 0 and Day 90 for 8:2FTS; however, the recoveries remained above 80%. NMeFOSA showed decreasing recoveries after Day 7, with average recoveries ranging from 50 to 85%.

Table 10. Mean Recoveries for Effluent Sample #1 Stored at 4 ºC								
	PFAS	Mean Recovery per Storage Day					Recovery (%)	
Chemical Group	Acronym	Day 0	Day 7	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	115	113	111	114	109	108	116
	PFDA	95	100	99	106	112	91	117
	PFDoA	112	110	106	98	116	93	116
	PFHpA	118	116	116	109	104	102	120
	PFHxA	114	108	109	112	111	103	116
	PFNA	104	102	104	109	120	99	123
	PFOA	113	110	109	110	104	97	115
	PFPeA	109	111	107	114	103	102	116
	PFTeDA	107	91	82	86	96	79	108
	PFTrDA	122	100	88	88	120	84	124
	PFUnA	104	97	95	101	108	92	111
Perfluoroalkyl sulfonates	PFBS	105	101	101	112	105	97	114
	PFDS	105	102	99	97	93	92	111
	PFDoS	70	61	57	57	58	55	72
	PFHpS	100	100	100	109	111	95	118
	PFHxS	94	89	95	104	108	85	109
	PFNS	98	97	91	102	104	89	110
	PFOS	90	91	88	108	107	85	112
	PFPeS	105	103	104	115	106	101	118
Fluorotelomer sulfonates	4:2FTS	127	125	124	112	115	109	135
	6:2FTS	126	126	123	114	105	102	134
	8:2FTS	135	126	111	99	85	81	145
Perfluorooctane sulfonamides	NEtFOSA	83	72	64	77	83	61	86
	NMeFOSA	100	88	74	77	80	72	102
	PFOSA	108	114	124	130	135	104	142
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	99	105	128	158	149	93	167
	NMeFOSAA	122	152	191	241	236	118	250
Perfluorooctane sulfonamidoethanols	NEtFOSE	97	73	67	69	71	64	100
	NMeFOSE	101	77	72	63	63	61	102

Based on the ANOVA and the Dunnett's test, there were thirteen (13) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 11 and 12).

Table 11. Statistical Difference Effluent Sample #1 Stored at 4 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	N	Y	N	Y	7
	PFDA	N	N	Y	Y	14
	PFDoA	N	N	Y	N	14
	PFHpA	N	N	Y	Y	14
	PFHxA	N	N	N	N	90
Perfluoroalkyl carboxylates	PFNA	N	N	Y	Y	14
	PFOA	N	N	N	N	90
	PFPeA	N	N	Y	Y	14
	PFTeDA	Y	Y	Y	Y	0

Table 11. Statistical Difference Effluent Sample #1 Stored at 4 ºC, Compared to Day 0						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFTrDA	Y	Y	Y	N	0
	PFUnA	N	Y	N	N	7
Perfluoroalkyl sulfonates	PFBS	N	N	Y	N	14
	PFDS	N	N	N	Y	28
	PFDoS	Y	Y	Y	Y	0
	PFHpS	N	N	Y	Y	14
	PFHxS	N	N	Y	Y	14
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	Y	14
	PFPeS	N	N	Y	N	14
Fluorotelomer sulfonates	4:2FTS	N	N	Y	Y	14
	6:2FTS	N	N	N	Y	28
	8:2FTS	N	Y	Y	Y	7
Perfluorooctane sulfonamides	NEtFOSA	Y	Y	N	N	0
	NMeFOSA	Y	Y	Y	Y	0
	PFOSA	N	Y	Y	Y	7
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	Y	Y	Y	7
	NMeFOSAA	Y	Y	Y	Y	0
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	Y	Y	0
	NMeFOSE	Y	Y	Y	Y	0

Based on a percent difference of 20%, PFTeDA, PFTrDA, NMeFOSA, NEtFOSA, NEtFOSAA, NEtFOSE, and NMeFOSE have a holding time of zero (0) or seven (7) days, while 8:2FTS has a holding time of 14 days and PFOSA of 28 days. PFTeDA and NEtFOSA have a percent difference >20% but <23% at Day 14 (23% and 22% respectively); however, the values decreased <20% on Days 28 and 90. All other compounds have a holding time of 90 days based on a 20% percent difference criteria.

Table 12. Selected Holding Time Based on 20% Difference for Effluent Sample #1 Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
Perfluoroalkyl carboxylates	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	Y	N	N	7
	PFTrDA	N	Y	Y	N	7
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	N	N	N	N	90
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	N	N	90

Table 12. Selected Holding Time Based on 20% Difference for Effluent Sample #1 Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl sulfonates	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	Y	Y	14
Perfluorooctane sulfonamides	NEtFOSA	N	Y	N	N	7
	NMeFOSA	N	Y	Y	Y	7
	PFOSA	N	N	N	Y	28
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	Y	Y	Y	7
	NMeFOSAA	Y	Y	Y	Y	0
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	Y	Y	0
	NMeFOSE	Y	Y	Y	Y	0

Table 13 presents the mean recoveries for Effluent Sample #1 stored at -20 ºC. Most of the compounds analyzed had recoveries between 70 and 130%, with very few exceptions. PFDoS had low recoveries across the board regardless of the holding day, with an average recovery of 76%, with a recovery range of 65 – 84%. The fluorotelomer sulfonates as a whole had recoveries consistently above 100%, with maximum recoveries above 130%. The recoveries decrease slightly between Day 0 and Day 90 for 4:2FTS and 8:2FTS; however, the recoveries remained above 100%.

Table 13. Mean Recoveries for Effluent Sample #1 Stored at -20 ºC							
		Mean Recovery per Storage Day				**Recovery (%)**	
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Min**	**Max**
Perfluoroalkyl carboxylates	PFBA	112	111	112	101	100	113
	PFDA	97	95	106	104	92	110
	PFDoA	115	108	104	111	102	117
	PFHpA	114	113	110	97	95	115
	PFHxA	109	105	105	99	96	111
	PFNA	103	102	105	101	98	108
	PFOA	107	107	106	101	98	111
	PFPeA	105	106	112	95	94	114
	PFTeDA	110	106	121	99	93	127
	PFTrDA	102	99	112	116	95	121
	PFUnA	99	98	102	99	93	106
Perfluoroalkyl sulfonates	PFBS	103	103	111	102	99	113
	PFDS	96	100	101	90	79	104
	PFDoS	76	82	76	69	65	84
	PFHpS	101	101	114	103	98	115
	PFHxS	91	95	101	101	90	104
	PFNS	93	91	108	97	90	110
	PFOS	90	87	112	100	86	114
	PFPeS	102	102	112	98	95	114
Fluorotelomer sulfonates	4:2FTS	121	126	111	106	100	131
	6:2FTS	120	119	114	99	97	125
	8:2FTS	134	130	127	114	110	140

Table 13. Mean Recoveries for Effluent Sample #1 Stored at -20 ºC							
		Mean Recovery per Storage Day				Recovery (%)	
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Min**	**Max**
Perfluorooctane sulfonamides	NEtFOSA	83	76	90	88	74	95
	NMeFOSA	101	96	100	93	91	107
	PFOSA	101	103	105	103	97	110
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	97	92	98	95	87	102
	NMeFOSAA	116	117	116	108	105	124
Perfluorooctane sulfonamidoethanols	NEtFOSE	92	87	101	93	86	103
	NMeFOSE	98	94	100	95	93	101

Based on the ANOVA and the Dunnett's test, there were eight (8) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 14 and 15).

Table 14. Statistical Difference for Effluent Sample #1 Stored at -20 ºC, Compared to Day 0						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	Y	Y	Y	Y	0
	PFDA	N	N	Y	Y	14
	PFDoA	N	N	Y	N	14
	PFHpA	Y	Y	Y	Y	0
	PFHxA	N	Y	Y	Y	7
	PFNA	N	N	N	N	90
	PFOA	N	N	N	Y	28
	PFPeA	N	N	N	Y	28
	PFTeDA	N	N	Y	N	14
	PFTrDA	Y	Y	Y	N	0
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	N	Y	N	N	7
	PFHpS	N	N	Y	N	14
	PFHxS	N	N	Y	Y	14
	PFNS	N	Y	Y	N	7
	PFOS	N	N	Y	Y	14
	PFPeS	N	N	Y	Y	14
Fluorotelomer sulfonates	4:2FTS	N	N	Y	Y	14
	6:2FTS	N	N	N	Y	28
	8:2FTS	N	N	N	Y	28
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	Y	28
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	Y	N	N	7
	NMeFOSE	N	Y	N	Y	7

When looking at a percent difference of 20%, PFOS has a holding time of 14 days, 6:2FTS has a holding time of 28 days, while the other compounds have a holding time of 90 days. It is important to note that 6:2FTS had a percent difference slightly above 20% (21.5%) for Day 90.

Table 15. Selected Holding Time Based on 20% Difference for Effluent Sample #1 Stored at -20 ºC

Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Chosen HT
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
	PFBS	N	N	N	N	90
Perfluoroalkyl sulfonates	PFDS	N	N	N	N	90
	PFDoS	N	N	N	N	90
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	N	14
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	Y	28
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	N	90
	NMeFOSE	N	N	N	N	90

Table 16 presents the mean recoveries for Effluent Sample #2 stored at 4 ºC. Most of the compounds analyzed had recoveries between 70 and 130%, with the following exceptions. PFDoS and NEtFOSA had low recoveries across the board regardless of the holding day, with an average recovery of 74% for both, with a recovery ranges of 59 – 83% and 62 – 79%, respectively. Both, the fluorotelomer sulfonates and NMeFOSAA, had recoveries consistently above 100%, with maximum recoveries above 130%. Compound 8:2FTS had recoveries consistently above 130%. The recoveries decrease slightly for the fluorotelomers between Day 0 and Day 90; however, the recoveries still remained above 100%.

Table 16. Mean Recoveries for Effluent Sample #2 Stored at 4 ºC								
	PFAS	Mean Recovery per Storage Day					Recovery (%)	
Chemical Group	Acronym	Day 0	Day 7	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	115	111	111	113	108	108	117
	PFDA	101	96	97	105	109	92	116
	PFDoA	117	110	107	100	116	97	118
	PFHpA	117	114	116	110	104	104	119
	PFHxA	114	107	108	110	110	105	116
	PFNA	108	99	106	102	110	95	111
	PFOA	112	108	107	107	108	103	117
Perfluoroalkyl carboxylates	PFPeA	109	106	106	113	101	100	114
	PFTeDA	106	97	97	108	105	95	111
	PFTrDA	109	100	95	105	117	92	120
	PFUnA	99	104	99	100	108	91	109
Perfluoroalkyl sulfonates	PFBS	105	103	102	110	107	100	113
	PFDS	108	103	98	98	91	81	112
	PFDoS	81	75	76	64	72	59	83
	PFHpS	99	97	101	110	107	90	114
	PFHxS	91	91	96	106	107	90	111
	PFNS	98	98	91	101	99	81	103
	PFOS	88	89	89	107	103	86	113
	PFPeS	103	101	101	116	103	99	117
Fluorotelomer sulfonates	4:2FTS	131	126	123	112	117	105	132
	6:2FTS	128	122	120	118	106	105	137
	8:2FTS	139	137	131	131	122	116	145
Perfluorooctane sulfonamides	NEtFOSA	77	75	67	74	76	62	79
	NMeFOSA	96	99	86	87	84	82	104
	PFOSA	110	119	110	107	106	104	121
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	94	94	96	98	101	92	104
	NMeFOSAA	125	117	121	131	144	115	167
Perfluorooctane sulfonamidoethanols	NEtFOSE	97	84	85	92	96	82	101
	NMeFOSE	102	93	91	91	97	88	103

Based on the ANOVA and the Dunnett's test, there were ten (10) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 17 and 18).

Table 17. Statistical Difference for Effluent Sample #2 Stored at 4 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	Y	Y	N	Y	0
	PFDA	N	N	N	N	90
	PFDoA	Y	Y	Y	N	0
	PFHpA	Y	N	Y	Y	14
	PFHxA	Y	Y	N	N	0
	PFNA	Y	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	Y	Y	14
	PFTeDA	Y	Y	N	N	0

Table 17. Statistical Difference for Effluent Sample #2 Stored at 4 ºC, Compared to Day 0						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFTrDA	Y	Y	N	N	0
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	Y	28
	PFDoS	N	N	Y	N	14
	PFHpS	N	N	Y	Y	14
Perfluoroalkyl sulfonates	PFHxS	N	Y	Y	Y	7
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	Y	14
	PFPeS	N	N	Y	N	14
Fluorotelomer sulfonates	4:2FTS	N	N	Y	Y	14
	6:2FTS	N	N	N	Y	28
	8:2FTS	N	N	N	Y	28
Perfluorooctane sulfonamides	NEtFOSA	N	Y	N	N	7
	NMeFOSA	N	Y	N	Y	7
	PFOSA	Y	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	Y	N	0
	NMeFOSE	Y	Y	Y	Y	0

Based on a percent difference of 20%, PFDoS and PFOS have holding time of 14 days while the other compounds have a holding time of 90 days. However PFDoS and PFOS had percent differences slightly above 20% (21.7% and 21% respectively) at Day 28, which decreased to below 20% for Day 90.

Table 18. Selected Holding Time Based on 20% Difference for Effluent Sample #2 Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	N	N	Y	N	14
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	N	14
	PFPeS	N	N	N	N	90

Table 18. Selected Holding Time Based on 20% Difference for Effluent Sample #2 Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	N	90
	NMeFOSE	N	N	N	N	90

Table 19 presents the mean recoveries for Effluent Sample #2 stored at -20 ºC. Most of the compounds analyzed had recoveries between 70 and 130%, with the following two exceptions. PFDoS had low recoveries across the board regardless of the holding day, with an average recovery of 74% for both, with a recovery range of 65 – 80%. Compound 8:2FTS had recoveries consistently above 100%, with an average of 125% and a range of 111 – 138%.

Table 19. Mean Recoveries for Effluent Sample #2 Stored at -20 ºC							
		Mean Recovery per Storage Day				**Recovery (%)**	
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Min**	**Max**
Perfluoroalkyl carboxylates	PFBA	110	111	112	100	99	112
	PFDA	95	95	103	103	90	108
	PFDoA	108	115	101	109	96	118
	PFHpA	114	113	110	97	95	118
	PFHxA	107	108	108	104	103	111
	PFNA	101	103	103	102	98	107
Perfluoroalkyl carboxylates	PFOA	107	108	105	97	95	109
	PFPeA	104	104	113	94	93	115
	PFTeDA	97	97	113	105	91	119
	PFTrDA	99	94	106	116	87	123
	PFUnA	90	94	95	100	86	106
Perfluoroalkyl sulfonates	PFBS	100	102	106	101	98	109
	PFDS	102	96	96	91	86	104
	PFDoS	77	78	70	72	65	80
	PFHpS	96	98	107	105	93	112
	PFHxS	87	96	103	98	83	104
	PFNS	91	89	100	98	88	101
	PFOS	86	86	107	99	80	108
	PFPeS	99	101	112	97	95	113
Fluorotelomer sulfonates	4:2FTS	119	121	109	108	104	124
	6:2FTS	124	118	119	104	96	128
	8:2FTS	130	131	129	111	111	138
Perfluorooctane sulfonamides	NEtFOSA	77	77	91	95	74	96
	NMeFOSA	94	97	98	101	90	104
	PFOSA	107	108	110	105	101	114
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	89	97	97	88	83	103
	NMeFOSAA	115	116	113	110	102	120

Table 19. Mean Recoveries for Effluent Sample #2 Stored at -20 ºC							
		Mean Recovery per Storage Day				Recovery (%)	
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Min	Max
Perfluorooctane sulfonamidoethanols	NEtFOSE	85	87	97	97	84	98
	NMeFOSE	93	96	97	98	90	99

Based on the ANOVA and the Dunnett's test, there were ten (10) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 20 and 21).

Table 20. Statistical Difference for Effluent Sample #2 Stored at -20 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	Y	Y	Y	Y	0
	PFDA	N	N	N	N	90
	PFDoA	N	N	Y	N	14
	PFHpA	N	N	Y	Y	14
	PFHxA	Y	Y	Y	Y	0
	PFNA	Y	N	N	N	90
	PFOA	N	N	N	Y	28
	PFPeA	Y	Y	Y	Y	0
	PFTeDA	N	N	N	N	90
	PFTrDA	N	Y	N	N	7
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	Y	Y	Y	7
	PFDoS	N	N	Y	Y	14
	PFHpS	N	N	N	N	90
	PFHxS	N	Y	Y	Y	7
	PFNS	Y	Y	N	N	0
	PFOS	N	N	Y	Y	14
	PFPeS	N	N	Y	Y	14
Fluorotelomer sulfonates	4:2FTS	Y	Y	Y	Y	0
	6:2FTS	N	N	N	Y	28
	8:2FTS	N	N	N	Y	28
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	N	N	0
	NMeFOSE	Y	Y	Y	Y	0
Perfluorooctane sulfonamides	NEtFOSA	N	N	Y	Y	14
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	Y	Y	14

Based on a percent difference of 20%, PFOS has a holding time of 14 days while NEtFOSA has a holding time of 28 days. It is important to note that PFOS had a percent difference of 21% on Day 28 while it was <20% at Day 90. Also, NEtFOSA had a percent difference of 22.3% on Day 90. All other compounds had a holding time of 90 days.

Table 21. Selected Holding Time Based on 20% Difference for Effluent Sample #2 Stored at -20 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
Perfluoroalkyl carboxylates	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	N	N	N	N	90
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	N	14
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	Y	28
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	N	90
	NMeFOSE	N	N	N	N	90

Surface Waters

Table 22 presents the mean recoveries for river water stored at 4 ºC. Most of the compounds analyzed had recoveries between 70 and 130%, with the following exceptions. PFDoS and NEtFOSA had low recoveries across the board regardless of the holding day, with an average recovery of 72% and 75%, respectively. Recoveries ranged from 61 – 93% for PFDoS and 64 – 85% for NEtFOSA. The fluorotelomer sulfonates as a whole as well as NMeFOSAA, had recoveries consistently above 100% with maximum recoveries above 130%. Compound 8:2FTS had recoveries consistently above 100%, with an average of 127% and a range of 100 – 141%. NMeFOSAA had recoveries above 100% from Day 0 through Day 28, with an average of 132%. However, at Day 90, the recovery drastically increased to above 400%. NEtFOSA had an average recovery of 99% from Day 0 through Day 28 and increased to 155% on Day 90. NMeFOSE had an average recovery of 94% from Day 0 through Day 28 and decreased on Day 90 to 65%.

Table 22. Mean Recoveries for River Water Stored at 4 ºC								
		Mean Recovery per Storage Day					Recovery (%)	
Chemical Group	PFAS Acronym	Day 0	Day 7	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	116	114	113	113	105	101	118
	PFDA	99	98	96	104	104	93	111
	PFDoA	117	108	109	98	113	98	120
	PFHpA	119	116	116	109	102	97	124
	PFHxA	111	110	111	110	101	98	115
	PFNA	107	105	103	106	108	100	110
	PFOA	113	111	109	105	107	103	115
	PFPeA	108	109	109	116	99	96	116
	PFTeDA	105	96	97	108	100	91	109
	PFTrDA	110	96	94	108	108	89	121
	PFUnA	103	95	100	97	102	91	110
Perfluoroalkyl sulfonates	PFBS	106	103	104	109	106	101	111
	PFDS	110	95	99	97	85	75	113
	PFDoS	87	67	72	67	65	61	93
	PFHpS	102	99	103	109	109	97	115
	PFHxS	95	95	97	107	102	91	111
	PFNS	99	95	93	102	97	90	107
	PFOS	90	91	89	109	103	86	112
	PFPeS	106	105	101	114	100	97	118
Fluorotelomer sulfonates	4:2FTS	128	126	127	112	110	104	137
	6:2FTS	121	122	124	116	110	108	128
	8:2FTS	133	128	135	130	108	100	141
Perfluorooctane sulfonamides	NEtFOSA	81	73	64	82	74	64	85
	NMeFOSA	97	93	85	89	81	75	98
	PFOSA	106	119	108	104	113	102	133
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	99	97	100	100	155	95	183
	NMeFOSAA	123	131	135	140	435	122	510
Perfluorooctane sulfonamidoethanols	NEtFOSE	97	87	83	95	86	79	99
	NMeFOSE	100	94	90	92	65	56	102

Based on the ANOVA and the Dunnett's test, there were five (5) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 23 and 24).

Table 23. Statistical Difference for River Water Stored at 4 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 15	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	N	N	N	Y	28
	PFDA	N	N	N	N	90
	PFDoA	Y	Y	Y	N	0
	PFHpA	N	N	Y	Y	14
	PFHxA	N	N	N	Y	28
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	Y	Y	14
	PFTeDA	N	N	N	N	90

Table 23. Statistical Difference for River Water Stored at 4 ºC, Compared to Day 0						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 15**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	Y	N	N	Y	28
	PFDoS	Y	Y	Y	Y	0
	PFHpS	N	N	N	N	90
	PFHxS	N	N	Y	N	14
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	Y	14
Perfluoroalkyl sulfonates	PFPeS	N	N	Y	N	14
Fluorotelomer sulfonates	4:2FTS	N	N	Y	Y	14
	6:2FTS	N	N	N	Y	28
	8:2FTS	N	N	N	Y	28
Perfluorooctane sulfonamides	NEtFOSA	Y	Y	N	Y	0
	NMeFOSA	N	Y	N	Y	7
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	Y	28
	NMeFOSAA	N	N	N	Y	28
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	N	Y	0
	NMeFOSE	N	N	N	Y	28

Based on a 20% difference from Day 0, PFDoS and PFOS have a holding time of 14 days, while PFDS, NEtFOSAA, NMeFOSAA, and NMeFOSE have a holding time of 28 days. PFDoS and PFOS had percent differences just above 20% (22.9% and 20.5% respectively) at Day 28, which decreased to <20% on Day 90. All other compounds had a holding time of 90 days based on the 20% difference criteria.

Table 24. Selected Holding Time Based on 20% Difference for River Water Sample Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	Y	28
	PFDoS	Y	N	Y	Y	14
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	N	14
	PFPeS	N	N	N	N	90

Table 24. Selected Holding Time Based on 20% Difference for River Water Sample Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	Y	28
	NMeFOSAA	N	N	N	Y	28
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	N	90
	NMeFOSE	N	N	N	Y	28

Table 25 presents the mean recoveries for river water stored at -20 ºC. Most of the compounds analyzed had recoveries between 70 and 130%, with the following exceptions. PFDoS had low recoveries across the board regardless of the holding day, with an average recovery of 75%, and a range from 62 – 80%. The fluorotelomer sulfonates as a whole, had recoveries consistently above 100% with maximum recoveries at or above 130%.

Table 25. Mean Recoveries for River Water Stored at -20 ºC							
		Mean Recovery per Storage Day				**Recovery (%)**	
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Min**	**Max**
Perfluoroalkyl carboxylates	PFBA	112	113	112	101	101	115
	PFDA	93	95	111	102	90	116
	PFDoA	115	111	98	110	95	124
	PFHpA	114	115	109	96	95	117
	PFHxA	112	111	110	100	100	116
	PFNA	107	104	105	105	101	110
	PFOA	109	109	104	100	99	111
	PFPeA	108	107	116	96	95	117
	PFTeDA	103	97	108	105	94	114
	PFTrDA	100	96	106	111	93	114
	PFUnA	97	96	100	100	90	105
Perfluoroalkyl sulfonates	PFBS	103	103	111	101	101	112
	PFDS	101	99	103	88	86	106
	PFDoS	74	77	69	79	62	80
	PFHpS	102	105	113	103	100	115
	PFHxS	94	97	106	100	92	107
	PFNS	94	91	107	94	90	111
	PFOS	90	89	110	97	88	113
	PFPeS	102	102	112	97	95	114
Fluorotelomer sulfonates	4:2FTS	127	127	109	108	103	133
	6:2FTS	125	123	114	101	100	130
	8:2FTS	136	128	129	110	106	148
Perfluorooctane sulfonamides	NEtFOSA	83	77	92	91	75	94
	NMeFOSA	103	93	100	99	90	111
	PFOSA	110	110	110	102	99	113

Table 25. Mean Recoveries for River Water Stored at -20 ºC							
		Mean Recovery per Storage Day				Recovery (%)	
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Min	Max
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	98	96	97	87	81	104
	NMeFOSAA	117	117	119	113	111	120
Perfluorooctane sulfonamidoethanols	NEtFOSE	92	86	100	96	84	103
	NMeFOSE	98	95	99	97	94	104

Based on the ANOVA and the Dunnett's test, there were eight (8) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 26 and 27).

Table 26. Statistical Difference for River Water Stored at -20 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	Y	Y	Y	Y	0
	PFDA	N	N	Y	N	14
	PFDoA	N	N	Y	N	14
	PFHpA	Y	N	Y	Y	14
Perfluoroalkyl carboxylates	PFHxA	N	N	N	Y	28
	PFNA	N	N	N	N	90
	PFOA	N	N	Y	Y	14
	PFPeA	N	N	Y	Y	14
	PFTeDA	N	N	N	N	90
	PFTrDA	Y	Y	N	N	0
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	Y	Y	14
	PFDS	Y	Y	Y	Y	0
	PFDoS	Y	Y	Y	N	0
	PFHpS	N	N	Y	N	14
	PFHxS	N	N	Y	N	14
	PFNS	N	Y	Y	N	7
	PFOS	N	N	Y	Y	14
	PFPeS	Y	Y	Y	Y	0
Fluorotelomer sulfonates	4:2FTS	N	N	Y	Y	14
	6:2FTS	N	N	N	Y	28
	8:2FTS	N	N	N	Y	28
Perfluorooctane sulfonamides	NEtFOSA	N	N	Y	Y	14
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	Y	Y	N	Y	0
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	Y	N	N	7
	NMeFOSE	N	N	N	N	90

Based on a percent difference of 20%, PFDS, PFDoS, and PFOS have a holding time of 28 days while all other compounds have a holding time of 90 days. However, PFDoS and PFOS had percent differences barely above 20% (20.7% and 221.6 respectively) at Day 28, which decreased to <20% on Day 90. PFDS also had a percent difference barely above 20% (20.5%) at Day 90.

Table 27. Selected Holding Time Based on 20% Difference for River Water Sample Stored at -20 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
Perfluoroalkyl carboxylates	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	Y	28
	PFDoS	N	N	Y	N	14
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	Y	N	14
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	N	90
	NMeFOSE	N	N	N	N	90

Groundwater Sample

The groundwater sample was analyzed for 40 compounds. Table 28 presents the mean recoveries for groundwater stored at 4 ºC. Most of the compounds analyzed had recoveries between 70 and 130%, with the following exceptions. Compound 7:3FTCA had an average recovery of 94% from Day 0 through 28 with a decrease on Day 90 to 69%. NFDHA had a maximum recovery of 149% which was observed for one replicate on Day 28. Compound 11Cl-PF3OUdS had a minimum recovery of 67% which was observed for one replicate on Day 14.

NMeFOSAA had a significant difference in recovery starting at storage Day 28, where the recovery increased from 107% to 148%. By Day 90, the recovery of this compound had more than tripled and had a mean recovery of 484%. The statistics show that the maximum holding time for this compound is 14 days when stored at 4 ºC. For NEtFOSAA, there was no statistical difference between Day 0 through Day 28. On Day 90, however, there was a sharp increase in recovery to a range of 324 – 373%. Therefore, the maximum holding time for NEtFOSAA is 28 days when the sample is stored at 4 ºC.

Perfluorooctane sulfanomide ethanols have peak recoveries at Day 28 and then have a significant decrease in recovery at Day 90. Although there is a statistical difference between the recoveries when comparing Day 0 with each of the following days, up to Day 28, all the recoveries are still well within the

70 – 130% interim range that was selected for this study. Also, there is no statistical difference between the recoveries from Day 7 through Day 28, showing compound stability. However, on Day 90, the recoveries decreased drastically. For NEtFOSE, the recovery decreased from an average of 98%, up to Day 28, to recoveries ranging from 59 – 62% on Day 90. This recovery range may be within the limits of the final method calculated recovery range, but it is well outside the interim range. On the other hand, NMeFOSE recovery decreased from an average of 96%, up to Day 28, to recoveries ranging from 36 – 38% on Day 90. The obvious decrease shows that the maximum stability for these compounds in a sample stored at 4 °C is 28 days.

Table 28. Mean Recoveries for Groundwater Stored at 4 ºC

		Mean Recovery per Storage Day (%)					Recovery (%)	
Chemical Group	**PFAS Acronym**	**Day 0**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Min**	**Max**
Perfluoroalkyl carboxylates	PFBA	103	101	98	104	103	97	106
	PFDA	105	104	103	109	105	97	112
	PFDoA	103	97	92	97	95	88	107
	PFHpA	103	103	102	102	107	98	112
	PFHxA	100	98	98	102	102	93	105
	PFNA	104	100	101	104	106	97	111
	PFOA	100	102	93	100	104	91	108
	PFPeA	107	105	103	106	112	100	113
	PFTeDA	98	94	91	91	90	87	100
	PFTrDA	96	94	86	89	87	85	98
	PFUnA	104	100	95	103	104	93	108
Perfluoroalkyl sulfonates	PFBS	102	105	101	105	109	93	110
	PFDS	98	98	87	91	94	82	103
	PFDoS	90	82	75	73	76	70	94
	PFHpS	96	99	96	101	100	93	102
	PFHxS	109	109	99	107	109	91	112
	PFNS	99	103	96	105	103	89	106
	PFOS	96	105	96	112	98	75	118
	PFPeS	96	93	90	96	95	88	98
Fluorotelomer sulfonates	4:2FTS	103	99	100	105	101	96	108
	6:2FTS	100	100	97	103	102	95	105
	8:2FTS	111	111	108	116	103	102	119
Perfluorooctane sulfonamides	NEtFOSA	101	94	96	104	91	91	108
	NMeFOSA	93	95	98	108	93	90	114
	PFOSA	108	114	103	115	122	99	124
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	101	100	101	113	353	95	372
	NMeFOSAA	100	101	107	148	484	98	493
Perfluorooctane sulfonamidoethanols	NEtFOSE	108	97	93	94	61	59	108
	NMeFOSE	107	96	93	88	37	36	107
Per- and Polyfluoroether carboxylates	ADONA	87	82	82	91	107	75	109
	HFPO-DA	106	104	96	105	103	88	110
	NFDHA	107	119	104	121	111	74	149
	PFMBA	108	107	104	103	110	99	113
	PFMPA	96	104	102	103	109	93	111

Table 28. Mean Recoveries for Groundwater Stored at 4 ºC								
		Mean Recovery per Storage Day (%)					Recovery (%)	
Chemical Group	PFAS Acronym	Day 0	Day 7	Day 14	Day 28	Day 90	Min	Max
Ether sulfonates	11Cl-PF3OUdS	87	82	74	79	95	67	97
	9Cl-PF3ONS	89	85	81	92	110	74	113
	PFEESA	101	100	100	100	97	95	104
Fluorotelomer carboxylates	3:3FTCA	100	103	99	101	107	97	111
	5:3FTCA	97	94	88	90	88	85	100
	7:3FTCA	101	100	90	85	69	68	104

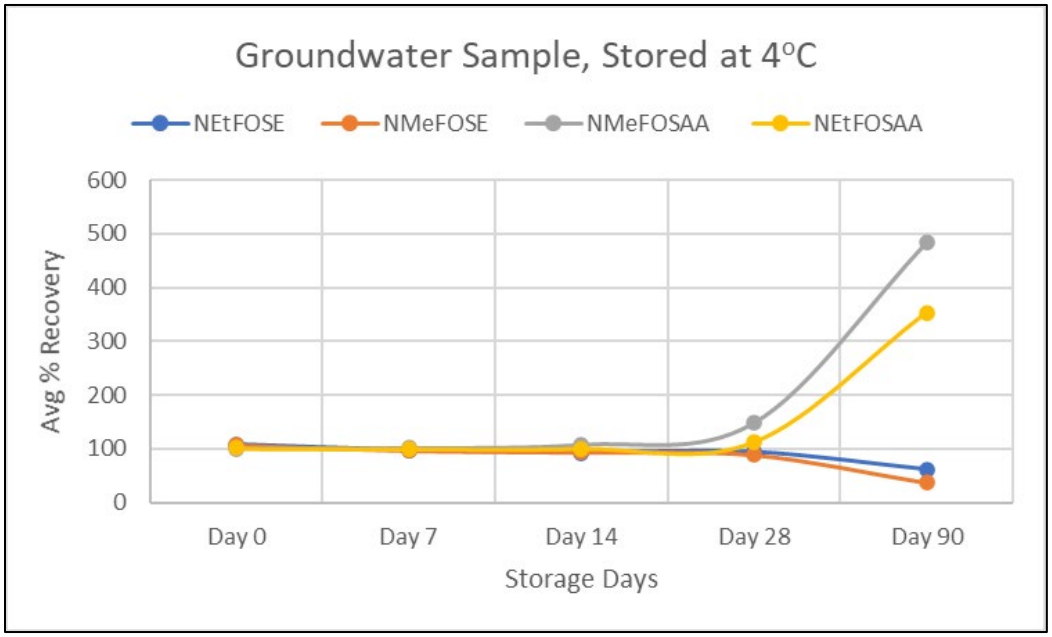


Figure 1. Plot of Mean Recovery for Perfluorooctane Sulfonamidoacetic Acids and Perfluorooctane Sulfonamidoethanols in Groundwater Sample Stored at 4 ºC

Based on the ANOVA and the Dunnett's test, there were fourteen (14) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 29 and 30).

Table 29. Statistical Difference for Groundwater Sample Stored at 4 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	N	Y	N	N	7
	PFDA	N	N	N	N	90
	PFDoA	N	Y	N	Y	7
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	Y	N	N	7
	PFPeA	N	N	N	N	90
	PFTeDA	N	Y	Y	Y	7
	PFTrDA	N	Y	Y	Y	7
	PFUnA	N	Y	N	N	7

Table 29. Statistical Difference for Groundwater Sample Stored at 4 ºC, Compared to Day 0						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	Y	N	N	7
	PFDoS	N	Y	Y	Y	7
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	N	N	90
	PFPeS	N	Y	N	N	7
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	Y	28
	NMeFOSA	N	N	Y	N	14
	PFOSA	N	N	N	Y	28
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	Y	28
	NMeFOSAA	N	N	Y	Y	14
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	Y	Y	0
	NMeFOSE	Y	Y	Y	Y	0
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	Y	28
	HFPO-DA	N	N	N	N	90
	NFDHA	N	N	N	N	90
	PFMBA	N	N	N	N	90
	PFMPA	Y	N	Y	Y	14
Ether sulfonates	11Cl-PF3OUdS	N	Y	N	N	7
	9Cl-PF3ONS	N	N	N	Y	28
	PFEESA	N	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	Y	28
	5:3FTCA	N	Y	Y	Y	7
	7:3FTCA	N	Y	Y	Y	7

For a percent difference of 20%, the holding times are as follows: NMeFOSAA at 14 days, NEtFOSAA, NEtFOSE, NMeFOSE, 9Cl-PF3ONS, 7:3FTCA, and ADONA at 28 days, all other compounds at 90 days. ADONA had a percent difference of 22% on Day 90.

Table 30. Selected Holding Time Based on 20% Difference for Groundwater Sample Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90

Table 30. Selected Holding Time Based on 20% Difference for Groundwater Sample Stored at 4 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	N	N	N	N	90
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	N	N	90
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	Y	28
	NMeFOSAA	N	N	Y	Y	14
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	Y	28
	NMeFOSE	N	N	N	Y	28
Ether sulfonates	11Cl-PF3OUdS	N	N	N	N	90
	9Cl-PF3ONS	N	N	N	Y	28
	PFEESA	N	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	Y	28
	HFPO-DA	N	N	N	N	90
	NFDHA	N	N	N	N	90
	PFMBA	N	N	N	N	90
	PFMPA	N	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	N	90
	5:3FTCA	N	N	N	N	90
	7:3FTCA	N	N	N	Y	28

Average percent recoveries showed that the compounds are stable up to 90 days when groundwater samples are stored at -20 ºC (Table 31). There were a few outliers during analysis. For Day 7, one of the replicates had a high recovery for NFDHA (154%) while that same analyte had a low recovery (67%) on a replicate from Day 90. On Day 28, one replicate had low recoveries for PFDoS (67%), PFMPA (49%), and 3:3FTCA (53%).

Table 31. Mean Recoveries for Groundwater Sample Stored at -20 ºC								
	PFAS	**Mean Recovery per Storage Day (%)**					**Recovery (%)**	
Chemical Group	**Acronym**	**Day 0**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Min**	**Max**
Perfluoroalkyl carboxylates	PFBA	103	100	98	96	103	76	108
	PFDA	105	99	100	111	108	96	112
	PFDoA	100	94	96	102	105	93	110
	PFHpA	102	101	99	102	105	94	107
	PFHxA	101	97	97	97	99	79	107
Perfluoroalkyl carboxylates	PFNA	102	99	97	105	103	96	108
	PFOA	99	95	94	102	103	90	107
	PFPeA	107	103	102	98	111	79	115
	PFTeDA	104	90	89	92	93	86	104

Table 31. Mean Recoveries for Groundwater Sample Stored at -20 ºC								
	PFAS	Mean Recovery per Storage Day (%)					Recovery (%)	
Chemical Group	Acronym	Day 0	Day 7	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFTrDA	97	91	90	93	91	87	100
	PFUnA	103	101	95	106	101	94	110
Perfluoroalkyl sulfonates	PFBS	103	102	99	97	106	79	108
	PFDS	100	93	92	92	97	89	105
	PFDoS	92	78	80	73	81	67	96
	PFHpS	99	94	96	99	96	92	101
	PFHxS	108	101	101	107	109	97	112
	PFNS	93	99	99	105	103	84	108
	PFOS	97	92	102	111	110	88	120
	PFPeS	96	90	90	94	94	87	100
Fluorotelomer sulfonates	4:2FTS	103	99	99	102	104	87	110
	6:2FTS	101	95	93	104	100	90	107
	8:2FTS	110	107	109	118	99	98	127
Perfluorooctane sulfonamides	NEtFOSA	102	95	100	104	103	91	109
	NMeFOSA	98	96	101	106	99	91	109
	PFOSA	110	112	102	112	111	99	116
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	102	95	95	101	102	92	108
	NMeFOSAA	99	96	96	103	101	90	109
Perfluorooctane sulfonamidoethanols	NEtFOSE	108	97	97	98	102	91	110
	NMeFOSE	108	96	98	99	103	92	110
Per- and Polyfluoroether carboxylates	ADONA	93	83	84	89	99	80	103
	HFPO-DA	106	101	99	103	99	89	114
	NFDHA	108	115	105	111	93	67	154
	PFMBA	105	105	103	97	112	79	115
	PFMPA	95	90	95	76	100	49	102
Ether sulfonates	11Cl-PF3OUdS	93	82	83	82	94	77	98
	9Cl-PF3ONS	95	86	85	93	103	82	105
	PFEESA	102	98	99	98	102	84	108
Fluorotelomer carboxylates	3:3FTCA	98	89	92	79	107	53	110
	5:3FTCA	97	91	91	95	99	88	103
	7:3FTCA	102	98	96	105	107	93	112

Based on the ANOVA and the Dunnett's test, there were fourteen (14) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 32 and 33).

Table 32. Statistics for Groundwater Sample Stored at -20 ºC, Compared to Day 0						
Chemical Group	PFAS Acronym	Day 7	Day 14	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90

Table 32. Statistics for Groundwater Sample Stored at -20 ºC, Compared to Day 0						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFPeA	N	N	N	N	90
	PFTeDA	Y	Y	Y	Y	0
	PFTrDA	N	N	N	N	90
	PFUnA	N	Y	N	N	7
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	Y	Y	Y	Y	0
	PFHpS	Y	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	Y	N	14
	PFOS	N	N	N	N	90
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	Y	28
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	Y	Y	N	N	0
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	Y	N	0
	NMeFOSE	Y	Y	Y	N	0
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	N	90
	HFPO-DA	N	N	N	N	90
	NFDHA	N	N	N	N	90
	PFMBA	N	N	N	N	90
	PFMPA	N	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	Y	Y	Y	N	0
	9Cl-PF3ONS	Y	Y	N	Y	0
	PFEESA	N	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	N	90
	5:3FTCA	N	N	N	N	90
	7:3FTCA	N	N	N	N	90

Based on a 20% difference from Day 0, PFDoS has a holding time of 14 days while the rest of the compounds have a holding time of 90 days. However, the percent difference for PFDoS was 21% on Day 28 and decreased to <20% on Day 90.

Table 33. Selected Holding Time Based on 20% Difference for Groundwater Sample Stored at -20 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90

Table 33. Selected Holding Time Based on 20% Difference for Groundwater Sample Stored at -20 ºC						
Chemical Group	**PFAS Acronym**	**Day 7**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	N	N	Y	N	14
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	N	N	90
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	N	90
	NMeFOSE	N	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	N	90
	9Cl-PF3ONS	N	N	N	N	90
	PFEESA	N	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	N	90
	HFPO-DA	N	N	N	N	90
	NFDHA	N	N	N	N	90
	PFMBA	N	N	N	N	90
	PFMPA	N	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	N	90
	5:3FTCA	N	N	N	N	90
	7:3FTCA	N	N	N	N	90

4.2 Solid Samples

The results for each the three solid samples (soil, biosolids, and sediment) stored at both temperatures are discussed below.

Soil Sample

Table 34 shows the average percent recoveries for the soil sample stored at 4 ºC. All recoveries, including the minimum and maximum recoveries, fall within the interim 70 – 130% recovery limit.

Table 34. Mean Recoveries for Soil Sample Stored at 4 ºC							
		Mean Recovery per Storage Day (%)				Recovery (%)	
Chemical Group	PFAS Acronym	Day 0	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	103	103	102	103	100	105
	PFDA	104	109	103	104	97	113
	PFDoA	101	102	104	100	95	107
	PFHpA	103	102	103	102	99	104
	PFHxA	100	104	102	101	93	106
	PFNA	100	101	98	103	97	109
	PFOA	95	100	97	100	93	105
	PFPeA	106	108	104	107	102	109
	PFTeDA	101	99	97	99	96	102
	PFTrDA	108	106	105	102	99	110
	PFUnA	104	104	102	100	99	106
Perfluoroalkyl sulfonates	PFBS	106	106	105	107	101	110
	PFDS	100	103	99	99	94	104
	PFDoS	87	86	87	95	83	97
	PFHpS	100	101	97	97	95	104
	PFHxS	110	109	106	106	104	112
	PFNS	108	111	111	105	103	113
	PFOS	108	107	103	106	102	111
	PFPeS	101	102	102	100	97	108
Fluorotelomer sulfonates	4:2FTS	102	104	106	100	99	109
	6:2FTS	103	99	101	98	94	118
	8:2FTS	113	119	117	112	108	122
Perfluorooctane sulfonamides	NEtFOSA	109	102	107	105	102	112
	NMeFOSA	106	100	101	100	96	107
	PFOSA	104	103	100	100	98	106
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	111	107	106	108	104	114
	NMeFOSAA	109	105	102	104	98	111
Perfluorooctane sulfonamidoethanols	NEtFOSE	107	108	106	109	104	112
	NMeFOSE	108	108	105	105	104	109
Per- and Polyfluoroether carboxylates	ADONA	96	91	94	115	89	115
	HFPO-DA	102	99	103	103	95	108
	NFDHA	102	97	113	97	86	128
	PFMBA	103	102	98	100	93	110
	PFMPA	101	101	97	101	93	104
Ether sulfonates	11Cl-PF3OUdS	105	101	107	125	94	126
	9Cl-PF3ONS	105	98	103	123	92	124
	PFEESA	101	101	99	100	96	103
Fluorotelomer carboxylates	3:3FTCA	97	96	95	97	93	100
	5:3FTCA	100	96	95	93	90	102
	7:3FTCA	105	96	95	85	83	108

Based on the ANOVA and the Dunnett's test, there were two (2) compounds with sufficient statistically differences to have determined holding times of zero (0) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 35 and 36).

Table 35. Statistical Difference for Soil Sample Stored at 4 ºC, Compared to Day 0					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	Y	N	14
	PFTrDA	N	N	Y	28
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	Y	28
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	Y	Y	N	0
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	Y	Y	14
Per- and Polyfluoroether carboxylates	ADONA	N	N	Y	28
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	Y	28
	9Cl-PF3ONS	N	N	Y	28
	PFEESA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	Y	28
	7:3FTCA	Y	Y	Y	0

Based on a percent difference of 20%, all the compounds have a holding time of 90 days.

Table 36. Selected Holding Time Based on 20% Difference for Soil Sample Stored at 4 ºC					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	90
	9Cl-PF3ONS	N	N	N	90
	PFEESA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

Table 37 shows the average percent recoveries for soil samples stored at -20 ºC. For NFDHA, two replicates, one each for Day 14 and Day 28, had recoveries >130%.

Table 37. Mean Recoveries for Soil Sample Stored at -20 ºC							
		Mean Recovery per Storage Day (%)				Recovery (%)	
Chemical Group	PFAS Acronym	Day 0	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	104	101	100	103	97	104
	PFDA	101	101	103	108	96	113
	PFDoA	100	97	94	101	86	104
	PFHpA	105	100	98	103	95	107
	PFHxA	100	95	100	102	93	105
	PFNA	101	100	94	105	92	107
	PFOA	98	96	97	102	93	103
	PFPeA	106	105	102	110	99	111
	PFTeDA	102	98	97	101	93	103
	PFTrDA	107	100	100	105	94	109
	PFUnA	102	101	104	104	99	106
Perfluoroalkyl sulfonates	PFBS	107	103	99	109	96	112
	PFDS	101	102	98	102	93	107
	PFDoS	87	84	86	95	80	95
	PFHpS	99	99	97	99	91	104
	PFHxS	108	108	107	107	104	111
	PFNS	107	108	109	107	104	116
	PFOS	107	103	103	110	99	113
	PFPeS	102	100	100	102	98	106
Fluorotelomer sulfonates	4:2FTS	104	100	108	102	97	121
	6:2FTS	100	99	98	100	93	109
	8:2FTS	116	113	111	112	105	118
Perfluorooctane sulfonamides	NEtFOSA	109	104	102	107	100	114
	NMeFOSA	106	98	95	101	93	107
	PFOSA	106	102	101	104	98	106
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	111	101	106	112	96	115
	NMeFOSAA	106	105	100	107	95	108
Perfluorooctane sulfonamidoethanols	NEtFOSE	107	105	105	110	102	111
	NMeFOSE	109	106	102	107	99	111
Per- and Polyfluoroether carboxylates	ADONA	99	99	90	114	86	117
	HFPO-DA	108	109	101	102	99	115
	NFDHA	89	117	125	105	72	144
	PFMBA	103	100	96	103	93	104
	PFMPA	101	100	94	103	92	105
Ether sulfonates	11Cl-PF3OUdS	111	110	103	124	99	130
	9Cl-PF3ONS	108	107	98	123	95	129
	PFEESA	101	95	100	101	94	104
Fluorotelomer carboxylates	3:3FTCA	97	92	92	99	88	100
	5:3FTCA	99	90	94	93	87	100
	7:3FTCA	104	93	96	93	89	105

Based on the ANOVA and the Dunnett's test, there were five (5) compounds with sufficient statistically differences to have determined holding times of zero (0) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 38 and 39).

Table 38. Statistical Difference for Soil Sample Stored at -20 ºC, Compared to Day 0					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	Y	N	14
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	Y	28
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	Y	N	14
	NMeFOSA	Y	Y	N	0
	PFOSA	N	Y	N	14
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	Y	N	14
Per- and Polyfluoroether carboxylates	ADONA	N	Y	Y	14
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	Y	N	14
	PFMPA	N	Y	N	14
Ether sulfonates	11Cl-PF3OUdS	N	N	Y	28
	9Cl-PF3ONS	N	Y	Y	14
	PFEESA	Y	N	N	0
Fluorotelomer carboxylates	3:3FTCA	Y	Y	N	0
	5:3FTCA	Y	N	N	0
	7:3FTCA	Y	Y	Y	0

Based on a 20% difference, all compounds have a holding time of 90 days except for NFDHA has a holding time of 0 days.

Table 39. Selected Holding Time Based on 20% Difference for Soil Sample Stored at -20 ºC					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	90
	9Cl-PF3ONS	N	N	N	90
	PFEESA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	Y	Y	N	0
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90

Biosolids Sample

The biosolids sample was analyzed for 40 compounds. Table 40 presents the mean recoveries for the biosolids stored at 4 °C. All the compounds analyzed had recoveries between 70 and 130%, with the exception of PFDoS. This compound had low recoveries across all holding times, with an average recovery of 59% across Day 0 through 90, and a recovery range of 52 – 66%.

Table 40. Mean Recoveries for Biosolids Sample Stored at 4 ºC							
	PFAS	Mean Recovery per Storage Day (%)				Recovery (%)	
Chemical Group	Acronym	Day 0	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	104	94	102	103	91	106
	PFDA	106	96	106	109	94	112
	PFDoA	97	92	101	101	86	106
	PFHpA	104	93	102	105	86	108
	PFHxA	102	92	103	102	88	109
	PFNA	105	95	101	104	93	107
	PFOA	101	93	99	101	91	106
	PFPeA	106	98	105	109	95	111
	PFTeDA	100	89	94	92	86	101
	PFTrDA	107	93	101	105	90	117
	PFUnA	103	96	101	102	91	106
Perfluoroalkyl sulfonates	PFBS	105	91	99	110	89	112
	PFDS	92	84	83	92	82	95
	PFDoS	61	56	53	65	52	66
	PFHpS	96	89	98	94	86	101
	PFHxS	111	102	108	111	99	113
	PFNS	110	95	106	100	92	112
Perfluoroalkyl sulfonates	PFOS	107	98	107	105	93	109
	PFPeS	103	93	103	101	86	106
Fluorotelomer sulfonates	4:2FTS	104	94	106	96	90	109
	6:2FTS	104	92	100	99	90	110
	8:2FTS	118	113	118	115	109	122
Perfluorooctane sulfonamides	NEtFOSA	110	96	103	104	90	114
	NMeFOSA	106	95	98	98	90	107
	PFOSA	106	96	105	103	93	107
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	112	101	113	111	99	117
	NMeFOSAA	102	96	107	104	93	109
Perfluorooctane sulfonamidoethanols	NEtFOSE	118	104	122	115	103	123
	NMeFOSE	107	103	108	112	102	113
Per- and Polyfluoroether carboxylates	ADONA	97	88	95	106	86	109
	HFPO-DA	106	96	99	102	93	110
	NFDHA	111	100	91	96	71	129
	PFMBA	102	93	100	102	90	105
	PFMPA	97	91	93	100	89	101
Ether sulfonates	11Cl-PF3OUdS	97	84	85	109	80	110
	9Cl-PF3ONS	108	94	103	117	90	119
	PFEESA	104	93	100	96	90	107
Fluorotelomer carboxylates	3:3FTCA	95	85	92	97	82	98
	5:3FTCA	95	86	94	95	84	98
	7:3FTCA	100	88	92	98	85	103

Although all the compounds (except PFDoS) had average recoveries within the interim recovery range, the statistical analysis showed that there were enough differences between Day 0 and the other days that only three compounds had determined holding times greater than zero days (Table 41). The percent difference between the average recoveries was calculated and are shown in Table 42.

Table 41. Statistical Difference for Biosolids Sample Stored at 4 ºC, Compared to Day 0					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	Y	N	N	0
	PFDA	Y	N	N	0
	PFDoA	N	N	N	90
	PFHpA	Y	N	N	0
	PFHxA	Y	N	N	0
	PFNA	Y	Y	N	0
	PFOA	Y	N	N	0
	PFPeA	Y	N	N	0
	PFTeDA	Y	Y	Y	0
	PFTrDA	Y	N	N	0
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	Y	Y	N	0
	PFDS	Y	Y	N	0
	PFDoS	Y	Y	Y	0
	PFHpS	Y	N	N	0
	PFHxS	Y	N	N	0
	PFNS	Y	N	Y	0
	PFOS	Y	N	N	0
	PFPeS	Y	N	N	0
Fluorotelomer sulfonates	4:2FTS	Y	N	N	0
	6:2FTS	Y	N	N	0
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	Y	N	N	0
	NMeFOSA	Y	Y	Y	0
	PFOSA	Y	N	N	0
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	Y	N	N	0
	NMeFOSAA	Y	N	N	0
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	Y	Y	0
	NMeFOSE	Y	N	Y	0
Per- and Polyfluoroether carboxylates	ADONA	Y	N	Y	0
	HFPO-DA	Y	Y	N	0
	NFDHA	N	N	N	90
	PFMBA	Y	N	N	0
	PFMPA	Y	Y	N	0
Ether sulfonates	11Cl-PF3OUdS	Y	Y	Y	0
	9Cl-PF3ONS	Y	N	Y	0
	PFEESA	Y	N	Y	0
Fluorotelomer carboxylates	3:3FTCA	Y	N	N	0
	5:3FTCA	Y	N	N	0
	7:3FTCA	Y	Y	N	0

Based on a percent difference of 20%, all 40 compounds have a holding time of 90 days.

Table 42. Selected Holding Time Based on 20% Difference for Biosolids Stored at 4 ºC					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	90
	9Cl-PF3ONS	N	N	N	90
	PFEESA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

Table 43 presents the mean recoveries for the biosolids stored at -20 ºC. All the compounds analyzed had recoveries between 70 and 130%, with a few exceptions. PFDoS had low recoveries across all holding times, with an average recovery of 61% from Day 0 through 90, and a recovery range of 49 – 74%. NFDHA had an average recovery of 105% from Day 0 through 90, and a recovery range of 79 – 135%, with the recovery >130% from one replicate analyzed on Day 0. Compound 9Cl-PF3ONS had an average recovery of 105% from Day 0 through 90, and a recovery range of 89 – 132%, with the recovery >130% from one replicate analyzed on Day 90.

Table 43. Mean Recoveries for Biosolids Sample Stored at -20 ºC							
		Mean Recovery per Storage Day (%)				Recovery (%)	
Chemical Group	PFAS Acronym	Day 0	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	105	96	96	101	92	105
	PFDA	106	105	100	105	99	112
	PFDoA	97	97	96	103	83	107
	PFHpA	104	99	96	99	95	108
	PFHxA	104	95	95	103	93	107
	PFNA	102	97	96	100	93	104
	PFOA	101	96	94	101	91	105
	PFPeA	108	101	99	109	96	110
	PFTeDA	103	92	94	99	90	104
	PFTrDA	103	99	98	105	91	107
	PFUnA	103	94	99	100	91	105
Perfluoroalkyl sulfonates	PFBS	107	96	95	105	91	107
	PFDS	95	88	76	93	74	97
	PFDoS	66	58	50	72	49	74
	PFHpS	101	94	91	95	87	104
	PFHxS	111	103	102	110	99	114
	PFNS	115	99	99	103	95	118
	PFOS	112	103	98	107	95	112
	PFPeS	102	95	98	103	92	107
Fluorotelomer sulfonates	4:2FTS	101	94	100	98	90	104
	6:2FTS	105	94	97	100	89	106
	8:2FTS	117	109	107	113	100	119
Perfluorooctane sulfonamides	NEtFOSA	114	105	102	109	98	115
	NMeFOSA	106	99	96	102	92	107
	PFOSA	106	100	100	104	95	109
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	110	104	105	109	98	115
	NMeFOSAA	108	101	99	105	91	110
Perfluorooctane sulfonamidoethanols	NEtFOSE	117	111	116	115	106	119
	NMeFOSE	107	102	103	110	97	111
Per- and Polyfluoroether carboxylates	ADONA	97	88	89	112	83	122
	HFPO-DA	106	96	98	105	91	112
	NFDHA	119	102	101	97	79	135
	PFMBA	103	96	93	101	88	105
	PFMPA	100	93	90	101	86	102
Ether sulfonates	11Cl-PF3OUdS	98	85	80	114	75	122
	9Cl-PF3ONS	106	95	96	122	89	132
	PFEESA	103	93	93	97	90	108
Fluorotelomer carboxylates	3:3FTCA	96	87	85	98	81	101
	5:3FTCA	94	88	85	92	82	97
	7:3FTCA	102	91	83	99	80	104

Based on the ANOVA and the Dunnett's test, there were fifteen (15) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 44 and 45).

Table 44. Statistical Difference for Biosolids Sample Stored at -20 ºC, Compared to Day 0					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	Y	Y	N	0
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	Y	Y	Y	0
	PFHxA	Y	Y	N	0
	PFNA	N	N	N	90
	PFOA	N	Y	N	14
	PFPeA	N	Y	N	14
	PFTeDA	Y	Y	N	0
	PFTrDA	N	N	N	90
	PFUnA	Y	N	N	0
Perfluoroalkyl sulfonates	PFBS	Y	Y	N	0
	PFDS	Y	Y	N	0
	PFDoS	Y	Y	N	0
	PFHpS	N	Y	N	14
	PFHxS	N	Y	N	14
	PFNS	Y	Y	Y	0
	PFOS	Y	Y	N	0
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	Y	Y	N	0
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	Y	N	14
	PFOSA	Y	Y	N	0
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	Y	N	14
	PFMPA	N	Y	N	14
Ether sulfonates	11Cl-PF3OUdS	N	Y	Y	14
	9Cl-PF3ONS	N	N	N	90
	PFEESA	Y	Y	N	0
Fluorotelomer carboxylates	3:3FTCA	Y	Y	N	0
	5:3FTCA	N	Y	N	14
	7:3FTCA	Y	Y	N	0

Based on a 20% difference, PFDoS has a holding time of 14 days (% difference of 23.51% at Day 28) while all other compounds have a holding time of 90 days.

Table 45. Selected Holding Time Based on 20% Difference for Biosolids Stored at -20 ºC					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	Y	N	14
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	90
	9Cl-PF3ONS	N	N	N	90
	PFEESA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

Sediment Sample

Table 46 presents the mean recoveries for the sediments stored at 4 ºC. All the compounds analyzed had recoveries between 70 and 130%, with a few exceptions. Compound 6:2FTS had an average recovery of 106% from Day 0 through 90, and a recovery range of 100 – 132%, with the recovery >130% from one replicate analyzed on Day 28. NFDHA had an average recovery of 101% from Day 0 through 90, and a recovery range of 73 – 145%, with the recovery >130% from one replicate analyzed on Day 14.

Compound 11Cl-PF3OUdS had an average recovery of 109% from Day 0 through 90, and a recovery range of 99 – 135%, with the recovery >130% from one replicate analyzed on Day 90.

Table 46. Mean Recoveries for Sediment Sample Stored at 4 ºC

Chemical Group	PFAS Acronym	Mean Recovery per Storage Day (%)				Recovery (%)	
		Day 0	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	102	101	100	102	99	104
	PFDA	102	102	97	107	95	110
	PFDoA	104	101	95	100	91	107
	PFHpA	100	98	95	104	92	107
	PFHxA	104	97	102	97	94	107
	PFNA	102	99	98	101	94	104
	PFOA	101	95	97	98	94	105
	PFPeA	105	106	102	109	99	110
	PFTeDA	102	98	95	99	93	103
	PFTrDA	104	100	99	101	97	107
	PFUnA	100	104	100	99	97	105
Perfluoroalkyl sulfonates	PFBS	105	103	99	105	96	107
	PFDS	102	97	97	99	94	103
	PFDoS	98	94	93	98	90	100
	PFHpS	96	94	94	94	91	100
	PFHxS	115	113	110	110	105	115
	PFNS	106	104	106	107	103	110
	PFOS	111	108	108	112	107	113
	PFPeS	102	99	99	98	94	107
Fluorotelomer sulfonates	4:2FTS	100	98	101	104	96	105
	6:2FTS	103	103	111	105	100	132
	8:2FTS	117	113	106	110	100	122
Perfluorooctane sulfonamides	NEtFOSA	105	98	97	98	94	106
	NMeFOSA	101	95	91	91	86	103
	PFOSA	103	97	93	91	90	105
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	110	103	103	105	98	117
	NMeFOSAA	104	99	97	101	93	106
Perfluorooctane sulfonamidoethanols	NEtFOSE	107	104	104	104	102	108
	NMeFOSE	107	102	102	99	99	109
Per- and Polyfluoroether carboxylates	ADONA	92	93	89	112	83	117
	HFPO-DA	99	106	98	106	95	110
	NFDHA	91	127	106	81	73	145
	PFMBA	99	99	95	101	93	104
	PFMPA	100	99	95	102	94	102
Ether sulfonates	11Cl-PF3OUdS	104	102	101	128	99	135
	9Cl-PF3ONS	100	101	98	125	95	130
	PFEESA	105	96	98	97	94	107
Fluorotelomer carboxylates	3:3FTCA	92	87	87	90	84	94
	5:3FTCA	91	84	89	81	81	92
	7:3FTCA	99	85	90	81	78	100

Based on the ANOVA and the Dunnett's test, there were eleven (11) compounds with sufficient statistically differences to have determined holding times of zero (0) days; therefore, the percent

difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 47 and 48).

Table 47. Statistical Difference for Sediment Sample Stored at 4 ºC, Compared to Day 0

Chemical Group	PFAS Acronym	Day 14	Day 28	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	Y	N	Y	0
	PFNA	N	N	N	90
	PFOA	Y	N	N	0
	PFPeA	N	N	N	90
	PFTeDA	N	Y	N	14
	PFTrDA	N	Y	N	14
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	Y	N	14
	PFDS	N	Y	N	14
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	Y	Y	N	0
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	Y	Y	Y	0
	NMeFOSA	N	Y	Y	14
	PFOSA	Y	Y	Y	0
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	Y	N	Y	0
Per- and Polyfluoroether carboxylates	ADONA	N	N	Y	28
	HFPO-DA	N	N	N	90
	NFDHA	Y	N	N	0
	PFMBA	N	N	N	90
	PFMPA	N	Y	N	14
Ether sulfonates	11Cl-PF3OUdS	N	N	Y	28
	9Cl-PF3ONS	N	N	Y	28
	PFEESA	Y	Y	Y	0
Fluorotelomer carboxylates	3:3FTCA	Y	N	N	0
	5:3FTCA	Y	N	Y	0
	7:3FTCA	Y	Y	Y	0

Based on a 20% difference, NFDHA has a holding time of 0 days, 11Cl-PF3OUdS, 9Cl-PF3ONS, and ADONA have a holding time of 28 days, while the rest of the compounds have a holding time of 90 days.

For ADONA, the percent difference for Day 90 was 21.5%. It is important to note that NFDHA had percent difference of 40% on Day 14 which reflects on a recovery of 145% for one of the three replicates. The value decreased to <20% for Days 28 and 90. However, removing the outlier only decreases the percent difference to 26% for this compound. The percent differences for the ether sulfonates were < 25%.

Table 48. Selected Holding Time Based on 20% Difference for Sediment Sample Stored at 4 ºC					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	Y	28
	9Cl-PF3ONS	N	N	Y	28
	PFEESA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	Y	28
	HFPO-DA	N	N	N	90
	NFDHA	Y	N	N	0
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

Table 49 presents the mean recoveries for the sediment sample stored at -20 ºC. All the compounds analyzed had recoveries between 70 and 130% including the recovery ranges.

Table 49. Mean Recoveries for Sediment Sample Stored at -20 ºC

Chemical Group	PFAS Acronym	Mean Recovery per Storage Day (%)				Recovery (%)	
		Day 0	Day 14	Day 28	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	103	101	101	102	99	103
	PFPeA	106	104	103	109	101	110
	PFHxA	102	99	98	100	93	104
	PFHpA	103	102	100	102	98	106
	PFOA	96	97	97	99	93	100
	PFNA	101	97	98	102	95	104
	PFDA	102	103	100	106	94	109
	PFUnA	105	102	103	101	100	108
	PFDoA	102	96	99	99	90	104
	PFTrDA	106	99	102	100	96	110
	PFTeDA	99	98	97	99	95	101
Perfluoroalkyl sulfonates	PFBS	104	105	99	106	97	110
	PFPeS	103	100	100	100	99	107
	PFHxS	114	112	110	111	106	118
	PFHpS	98	99	96	96	93	100
	PFOS	112	109	108	111	107	113
	PFNS	108	104	107	103	101	110
Perfluoroalkyl sulfonates	PFDS	99	100	99	98	95	103
	PFDoS	98	97	93	98	93	100
Fluorotelomer sulfonates	4:2FTS	105	99	100	98	96	108
	6:2FTS	104	104	105	105	99	112
	8:2FTS	117	114	111	110	107	121
Perfluorooctane sulfonamides	PFOSA	106	99	102	100	97	107
	NMeFOSA	103	95	97	98	93	105
	NEtFOSA	106	96	101	102	94	112
Perfluorooctane sulfonamidoacetic acid	NMeFOSAA	104	100	100	103	98	109
	NEtFOSAA	108	105	110	107	95	113
Perfluorooctane sulfonamidoethanols	NMeFOSE	105	102	106	102	99	109
	NEtFOSE	107	104	105	107	103	108
Per- and Polyfluoroether carboxylates	HFPO-DA	102	101	103	105	98	110
	ADONA	95	89	92	113	86	116
	PFMBA	103	98	96	99	93	103
	PFMPA	100	99	95	101	91	103
	NFDHA	85	96	100	98	78	121
Ether sulfonates	9Cl-PF3ONS	102	97	101	123	94	126
	11Cl-PF3OUdS	105	101	104	127	98	130
	PFEESA	106	98	100	96	90	111
Fluorotelomer carboxylates	3:3FTCA	91	87	88	92	86	93
	5:3FTCA	91	88	87	86	83	94
	7:3FTCA	94	90	92	87	84	98

Based on the ANOVA and the Dunnett's test, there were four (4) compounds with sufficient statistically differences to have determined holding times of zero (0) days; therefore, the percent difference between

the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 50 and 51).

Table 50. Statistical Difference for Sediment Sample Stored at -20 ºC, Compared to Day 0					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	Y	Y	N	0
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	Y	N	N	0
	NMeFOSA	Y	Y	N	0
	PFOSA	Y	N	Y	0
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	Y	28
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	Y	N	14
Ether sulfonates	11Cl-PF3OUdS	N	N	Y	28
	9Cl-PF3ONS	N	N	Y	28
	PFEESA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

All compounds had a chosen holding time of 90 days except for 11Cl-PF3OUdS and 9Cl-PF3ONS which have a holding time of 28 days. However, these percent differences were barely above 20% (21.2% and 20.8% respectively) for Day 90.

Table 51. Selected Holding Time Based on 20% Difference for Sediment Sample Stored at -20 ºC					
Chemical Group	**PFAS Acronym**	**Day 14**	**Day 28**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
Perfluoroalkyl carboxylates	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	Y	28
	9Cl-PF3ONS	N	N	Y	28
	PFEESA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

4.3 Sample Extract Stability

The stability of extracts stored at 4 ºC, at increasing time intervals, was investigated. Only the extracts from groundwater, soil, biosolids, and sediment samples were analyzed.

Groundwater Sample Extract

Groundwater sample extracts had recoveries within the 70 – 130% interim limits, except for one replicate for NFDHA analyzed on Day 90, which had a recovery of 152%.

Table 52. Mean Recoveries for Groundwater Extracts Stored at 4 ºC

Chemical Group	PFAS Acronym	Mean Recovery per Storage Day (%)				Recovery (%)	
		Day 7	Day 15	Day 29	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	102	103	102	102	102	103
	PFDA	109	103	108	107	100	112
	PFDoA	101	104	105	105	98	111
	PFHpA	105	103	103	106	100	107
	PFHxA	101	99	100	101	95	104
	PFNA	104	101	101	102	98	106
	PFOA	98	99	100	104	94	106
	PFPeA	109	106	107	110	105	112
	PFTeDA	101	103	100	102	98	104
	PFTrDA	99	101	98	96	95	101
	PFUnA	102	103	105	99	97	106
Perfluoroalkyl sulfonates	PFBS	108	107	103	109	100	112
	PFDS	97	100	99	98	95	101
	PFDoS	87	90	90	92	85	94
	PFHpS	96	97	97	97	94	99
	PFHxS	109	108	107	111	104	112
	PFNS	103	103	105	102	100	107
	PFOS	99	101	101	100	82	115
	PFPeS	93	92	95	95	88	101
Fluorotelomer sulfonates	4:2FTS	103	101	106	99	95	108
	6:2FTS	102	103	103	102	98	107
	8:2FTS	116	115	114	103	102	120
Perfluorooctane sulfonamides	NEtFOSA	99	98	101	101	92	105
	NMeFOSA	94	95	94	96	89	100
	PFOSA	107	107	108	107	103	112
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	102	99	102	99	96	104
	NMeFOSAA	99	96	102	99	95	108
Perfluorooctane sulfonamidoethanols	NEtFOSE	109	108	110	111	108	112
	NMeFOSE	107	109	106	107	104	110
Per- and Polyfluoroether carboxylates	ADONA	88	90	89	101	85	106
	HFPO-DA	102	102	107	102	98	110
	NFDHA	101	97	108	124	89	152
	PFMBA	105	107	104	108	101	109
	PFMPA	96	98	98	102	95	104
Ether sulfonates	11Cl-PF3OUdS	88	95	91	104	86	107
	9Cl-PF3ONS	93	92	93	108	90	111
	PFEESA	100	102	100	99	94	106
Fluorotelomer carboxylates	3:3FTCA	99	97	102	110	95	112
	5:3FTCA	93	92	97	98	91	101
	7:3FTCA	99	99	104	106	96	107

Based on the ANOVA and the Dunnett's test, there were four (4) compounds with sufficient statistically differences to have determined holding times of zero (0) or seven (7) days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 53 and 54).

Table 53. Statistical Difference for Groundwater Sample Extracts, Compared to Day 0

Chemical Group	PFAS Acronym	Day 7	Day 15	Day 29	Day 90	Determined holding time
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	Y	N	N	7
	PFUnA	N	N	N	Y	29
Perfluoroalkyl sulfonates	PFBS	Y	N	N	Y	29
	PFDS	N	N	N	N	90
	PFDoS	N	N	N	N	90
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	Y	N	15
	PFOS	N	N	N	N	90
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	Y	29
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	Y	Y	15
	NMeFOSE	N	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	Y	29
	HFPO-DA	N	N	N	N	90
	NFDHA	N	N	N	N	90
	PFMBA	Y	Y	Y	Y	0
	PFMPA	Y	Y	Y	Y	0
Ether sulfonates	11Cl-PF3OUdS	N	Y	N	Y	7
	9Cl-PF3ONS	N	N	N	Y	29
	PFEESA	N	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	Y	29
	5:3FTCA	N	N	N	N	90
	7:3FTCA	N	N	N	N	90

All compounds had a chosen holding time of 90 days except for 9Cl-PF3ONS, which had a holding time of 29 days. However, the percent difference for this compound was barely above 20% (21.5%) on Day 90.

Table 54. Selected Holding Time Based on 20% Difference for Groundwater Sample Extract

Chemical Group	PFAS Acronym	Day 7	Day 15	Day 29	Day 90	Chosen HT
Perfluoroalkyl carboxylates	PFBA	N	N	N	N	90
	PFDA	N	N	N	N	90
	PFDoA	N	N	N	N	90
	PFHpA	N	N	N	N	90
	PFHxA	N	N	N	N	90
	PFNA	N	N	N	N	90
	PFOA	N	N	N	N	90
	PFPeA	N	N	N	N	90
	PFTeDA	N	N	N	N	90
	PFTrDA	N	N	N	N	90
	PFUnA	N	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	N	90
	PFDS	N	N	N	N	90
	PFDoS	N	N	N	N	90
	PFHpS	N	N	N	N	90
	PFHxS	N	N	N	N	90
	PFNS	N	N	N	N	90
	PFOS	N	N	N	N	90
	PFPeS	N	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	N	90
	6:2FTS	N	N	N	N	90
	8:2FTS	N	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	N	90
	NMeFOSA	N	N	N	N	90
	PFOSA	N	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	N	90
	NMeFOSAA	N	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	N	90
	NMeFOSE	N	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	N	90
	9Cl-PF3ONS	N	N	N	Y	29
	PFEESA	N	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	N	90
	HFPO-DA	N	N	N	N	90
	NFDHA	N	N	N	N	90
	PFMBA	N	N	N	N	90
	PFMPA	N	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	N	90
	5:3FTCA	N	N	N	N	90
	7:3FTCA	N	N	N	N	90

Soil Sample Extracts

Soil sample extracts had percent recoveries within the interim limit for all 40 compounds (Table 55); however, the statistical analysis found sufficient differences between the values to determine holding times at less than 15 days for two compounds, PFOA and NEtFOSAA, while there were five compounds that with holding times of 29 days (Table 56). Percent differences were examined and the statistics compiled in Table 57.

Table 55. Mean Recoveries for Soil Extracts Stored at 4 ºC

		Mean Recovery per Storage Day (%)			Recovery (%)	
Chemical Group	**PFAS Acronym**	**Day 15**	**Day 29**	**Day 90**	**Min**	**Max**
Perfluoroalkyl carboxylates	PFBA	103	104	102	101	104
	PFDA	106	101	105	99	110
	PFDoA	102	99	99	95	104
	PFHpA	101	105	105	101	107
	PFHxA	100	99	98	94	102
	PFNA	103	103	106	97	107
	PFOA	97	102	97	94	106
	PFPeA	106	105	109	103	111
	PFTeDA	98	100	100	96	102
	PFTrDA	108	107	105	104	109
	PFUnA	104	101	104	96	108
Perfluoroalkyl sulfonates	PFBS	105	101	106	100	109
	PFDS	102	101	101	99	106
	PFDoS	87	87	87	82	89
	PFHpS	101	99	99	97	103
	PFHxS	109	110	109	108	111
	PFNS	109	111	105	103	111
	PFOS	107	109	109	102	112
	PFPeS	104	104	100	99	109
Fluorotelomer sulfonates	4:2FTS	100	104	101	97	110
	6:2FTS	102	107	102	92	120
	8:2FTS	116	115	111	110	118
Perfluorooctane sulfonamides	NEtFOSA	113	109	109	105	114
	NMeFOSA	107	102	105	101	108
	PFOSA	105	106	106	103	110
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	105	106	112	103	113
	NMeFOSAA	105	107	105	101	111
Perfluorooctane sulfonamidoethanols	NEtFOSE	108	108	110	106	111
	NMeFOSE	108	107	108	106	109
Per- and Polyfluoroether carboxylates	ADONA	95	103	109	93	111
	HFPO-DA	106	106	101	98	110
	NFDHA	114	90	94	79	119
	PFMBA	100	101	101	99	105
	PFMPA	102	99	101	98	103
Ether sulfonates	11Cl-PF3OUdS	105	113	123	103	127
	9Cl-PF3ONS	102	111	121	99	124
	PFEESA	101	100	99	96	105
Fluorotelomer carboxylates	3:3FTCA	97	96	96	94	99
	5:3FTCA	97	99	92	89	102
	7:3FTCA	105	107	106	103	110

Table 56. Statistical Difference for Soil Sample Extracts, Compared to Day 0					
Chemical Group	**PFAS Acronym**	**Day 15**	**Day 29**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	Y	N	15
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	Y	Y	N	0
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	Y	29
	NMeFOSE	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	Y	29
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	Y	29
	9Cl-PF3ONS	N	N	Y	29
	PFEESA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	Y	29
	7:3FTCA	N	N	N	90

All compounds had a chosen holding time of 90 days, based on a 20% difference from Day 0.

Table 57. Selected Holding Time Based on 20% Difference for Soil Sample Extract					
Chemical Group	**PFAS Acronym**	**Day 15**	**Day 29**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	90
	9Cl-PF3ONS	N	N	N	90
	PFEESA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

Biosolids Sample Extracts

Percent recovery for biosolids sample extract, shown in Table 58, had recoveries within 70 – 130% with the following exceptions. PFDoS had low recoveries across the board. The sample had low recovery beginning on Day 0 and remained at low recovery until Day 90, with an average of 61% and a range of 57 – 64% until Day 90. Compound NFDHA had a maximum recovery of 135% for one replicate on Day 29.

Table 58. Mean Recoveries for Biosolids Extracts Stored at 4 ºC						
		Mean Recovery per Storage Day (%)			Recovery (%)	
Chemical Group	**PFAS Acronym**	**Day 15**	**Day 29**	**Day 90**	**Min**	**Max**
Perfluoroalkyl carboxylates	PFBA	103	103	103	102	105
	PFDA	104	108	109	102	112
	PFDoA	100	103	104	96	111
	PFHpA	105	104	103	101	107
	PFHxA	104	102	102	99	108
	PFNA	103	103	105	101	108
	PFOA	101	98	102	97	104
	PFPeA	107	106	112	104	114
	PFTeDA	101	103	102	100	106
	PFTrDA	99	100	103	97	104
	PFUnA	105	107	104	103	112
Perfluoroalkyl sulfonates	PFBS	107	102	107	99	113
	PFDS	90	91	90	88	94
	PFDoS	59	62	61	57	64
	PFHpS	96	96	95	94	99
	PFHxS	111	113	111	107	117
	PFNS	102	110	103	100	112
	PFOS	104	106	105	102	109
	PFPeS	102	106	103	101	108
Fluorotelomer sulfonates	4:2FTS	101	102	102	98	107
	6:2FTS	107	105	100	96	111
	8:2FTS	120	121	118	116	124
Perfluorooctane sulfonamides	NEtFOSA	113	109	110	107	119
	NMeFOSA	107	106	105	103	109
	PFOSA	107	105	106	105	109
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	109	110	107	105	113
	NMeFOSAA	104	104	106	101	108
Perfluorooctane sulfonamidoethanols	NEtFOSE	114	115	116	113	119
	NMeFOSE	106	105	107	104	108
Per- and Polyfluoroether carboxylates	ADONA	96	94	112	91	115
	HFPO-DA	103	105	106	96	111
	NFDHA	116	113	115	81	135
	PFMBA	102	103	101	99	107
	PFMPA	101	98	102	96	104
Ether sulfonates	11Cl-PF3OUdS	96	98	115	89	120
	9Cl-PF3ONS	104	106	123	99	127
	PFEESA	106	102	100	98	108
Fluorotelomer carboxylates	3:3FTCA	95	95	96	92	97
	5:3FTCA	97	96	90	87	100
	7:3FTCA	104	101	100	98	108

Based on the ANOVA and the Dunnett's test, there were two (2) compounds with sufficient statistically differences to have determined holding times of zero (0); therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 59 and 60).

Table 59. Statistical Difference for Biosolids Sample Extracts, Compared to Day 0					
Chemical Group	**PFAS Acronym**	**Day 15**	**Day 29**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	Y	29
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	Y	N	Y	0
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	Y	N	N	0
	NMeFOSE	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	Y	29
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	Y	29
	9Cl-PF3ONS	N	N	Y	29
	PFEESA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

Based on a percent difference of 20%, all the compounds have a chosen holding time of 90 days.

Table 60. Selected Holding Time Based on 20% Difference for Biosolids Extract					
Chemical Group	**PFAS Acronym**	**Day 15**	**Day 29**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	90
	9Cl-PF3ONS	N	N	N	90
	PFEESA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

Sediment Sample Extracts

Percent recovery for sediment sample extract, shown in Table 61, had recoveries within 70 – 130% with the following exception. NFDHA had recoveries above 130% for one replicate on Day 29 and two replicates on Day 90.

Table 61. Mean Recoveries for Sediment Extracts Stored at 4 ºC						
	PFAS	Mean Recovery per Storage Day (%)			Recovery (%)	
Chemical Group	Acronym	Day 15	Day 29	Day 90	Min	Max
Perfluoroalkyl carboxylates	PFBA	103	102	102	102	104
	PFDA	102	103	105	99	108
	PFDoA	99	99	103	93	107
	PFHpA	101	103	99	97	104
	PFHxA	100	101	103	97	106
	PFNA	103	102	100	99	107
	PFOA	100	99	103	98	103
	PFPeA	107	106	109	103	110
	PFTeDA	100	101	101	98	104
	PFTrDA	102	100	102	99	105
	PFUnA	102	103	100	96	107
Perfluoroalkyl sulfonates	PFBS	104	100	106	98	110
	PFDS	99	100	101	95	107
	PFDoS	97	95	100	94	102
	PFHpS	98	96	97	94	101
	PFHxS	116	111	110	108	120
	PFNS	107	105	107	100	111
	PFOS	110	110	113	106	115
	PFPeS	103	101	102	98	108
Fluorotelomer sulfonates	4:2FTS	101	107	103	98	109
	6:2FTS	105	104	106	101	108
	8:2FTS	117	116	108	104	121
Perfluorooctane sulfonamides	NEtFOSA	103	106	104	95	109
	NMeFOSA	105	103	104	100	107
	PFOSA	105	105	105	102	107
Perfluorooctane sulfonamidoacetic acid	NEtFOSAA	108	109	110	106	114
	NMeFOSAA	105	106	108	103	113
Perfluorooctane sulfonamidoethanols	NEtFOSE	108	108	110	107	110
	NMeFOSE	105	110	106	104	112
Per- and Polyfluoroether carboxylates	ADONA	92	99	107	89	109
	HFPO-DA	102	108	102	99	113
	NFDHA	102	108	129	82	138
	PFMBA	101	97	100	95	106
	PFMPA	102	97	101	96	103
Ether sulfonates	11Cl-PF3OUdS	104	112	123	103	126
	9Cl-PF3ONS	99	111	120	95	125
	PFEESA	103	99	102	97	105
Fluorotelomer carboxylates	3:3FTCA	91	91	92	88	93
	5:3FTCA	91	89	87	85	94
	7:3FTCA	98	97	96	94	100

Based on the ANOVA and the Dunnett's test, there were five (5) compounds with sufficient statistically differences to have determined holding times of 15 days; therefore, the percent difference between the average recovery for each day of the study were compared to the average recovery for the analytes on Day 0 (Tables 62 and 63).

Table 62. Statistical Difference for Sediment Sample Extracts, Compared to Day 0					
Chemical Group	**PFAS Acronym**	**Day 15**	**Day 29**	**Day 90**	**Determined holding time**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	Y	N	15
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	Y	29
	NMeFOSE	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	Y	29
	HFPO-DA	N	Y	N	15
	NFDHA	N	N	N	90
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	Y	Y	15
	9Cl-PF3ONS	N	Y	Y	15
	PFEESA	N	Y	N	15
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

All compounds had a chosen holding time of 90 days except for NFDHA which has a chosen holding time of 28 days.

Table 63. Selected Holding Time Based on 20% Difference for Sediment Sample Extract					
Chemical Group	**PFAS Acronym**	**Day 15**	**Day 29**	**Day 90**	**Chosen HT**
Perfluoroalkyl carboxylates	PFBA	N	N	N	90
	PFDA	N	N	N	90
	PFDoA	N	N	N	90
	PFHpA	N	N	N	90
	PFHxA	N	N	N	90
	PFNA	N	N	N	90
	PFOA	N	N	N	90
	PFPeA	N	N	N	90
	PFTeDA	N	N	N	90
	PFTrDA	N	N	N	90
	PFUnA	N	N	N	90
Perfluoroalkyl sulfonates	PFBS	N	N	N	90
	PFDS	N	N	N	90
	PFDoS	N	N	N	90
	PFHpS	N	N	N	90
	PFHxS	N	N	N	90
	PFNS	N	N	N	90
	PFOS	N	N	N	90
	PFPeS	N	N	N	90
Fluorotelomer sulfonates	4:2FTS	N	N	N	90
	6:2FTS	N	N	N	90
	8:2FTS	N	N	N	90
Perfluorooctane sulfonamides	NEtFOSA	N	N	N	90
	NMeFOSA	N	N	N	90
	PFOSA	N	N	N	90
Perfluorooctane sulfonamidoacetic acids	NEtFOSAA	N	N	N	90
	NMeFOSAA	N	N	N	90
Perfluorooctane sulfonamidoethanols	NEtFOSE	N	N	N	90
	NMeFOSE	N	N	N	90
Ether sulfonates	11Cl-PF3OUdS	N	N	N	90
	9Cl-PF3ONS	N	N	N	90
	PFEESA	N	N	N	90
Per- and Polyfluoroether carboxylates	ADONA	N	N	N	90
	HFPO-DA	N	N	N	90
	NFDHA	N	N	Y	28
	PFMBA	N	N	N	90
	PFMPA	N	N	N	90
Fluorotelomer carboxylates	3:3FTCA	N	N	N	90
	5:3FTCA	N	N	N	90
	7:3FTCA	N	N	N	90

Section 5.0 Conclusions

The extent of degradation of a contaminant in a sample is dependent on matrix, concentration, preservation technique, class, and measurement variability. The results were compiled by matrix, although not all aqueous samples were analyzed for the same number of analytes. Since there were 29 or 40 compounds analyzed across the two data sets, the results were further compiled by chemical class instead of designating a holding time for each individual compound. A tally was made of the numbers of analytes that "survived" on each day of the study (e.g., the mean did not differ by more than 20% from the mean on Day 0) for each chemical group/treatment/day combination. In some instances, the percent difference marginally exceeded the 20% criteria in a small minority of analytes. This slight margin exceedance was not considered adequate grounds to change the holding time for those analytes. Many organic analyses typically have accuracies of ±30% or more; therefore, the ±20% mean difference criteria was considered a conservative baseline.

Table 64 shows the holding time that was determined for aqueous samples for each PFAS chemical group and the percentage of observations in each day that had a percent difference greater than 20% when compared to Day 0. The results show that holding time is improved by freezing the samples at a temperature of -20 ºC, with some of the compounds increasing in stability by up to 90 days. When aqueous samples are stored at 4 ºC, the perfluorooctane sulfonamide ethanols and perfluorooctane sulfonamidoacetic acids did not show consistent results at 7 days; therefore , these compounds should be analyzed as soon as possible after collection; however, freezing the samples allows these compounds to be stored for a longer period of time, allowing for greater laboratory flexibility. When the samples are frozen, the perfluoroalkyl sulfonates can be expected to be stable for analysis up to 14 days, and fluorotelomer sulfonates, perfluoroalkyl carboxylates, and perfluorooctane sulfonamides can be expected to be stable up to 28 days. These observations had a percent difference below ±25%. It is important to note that fluorotelomer sulfonates remain stable up to 28 days regardless of the temperature used for preservation. After analyzing all the variables, the best and most stable length of storage for aqueous samples is 90 days when stored at -20 ºC. Aqueous samples may be stored at 4 ºC for up to 28 days; however, the user is advised that at this storage temperature there is evidence of the transformation of precursors, as discussed above, when the sample is held beyond 7 days.

Table 64. Holding Time for Aqueous Matrices per Chemical Group

Chemical Group	Total # of Observations	# of Times HT was Observed			
		7 days	14 days	28 days	90 days
Samples Stored at 4 °C					
Ether sulfonates	3	0	0	1	2
Fluorotelomer carboxylates	3	0	0	1	2
Fluorotelomer sulfonates	15	0	1	0	14
Per- and Polyfluoroether carboxylates	5	0	0	1	4
Perfluoroalkyl carboxylates	55	2	0	0	53
Perfluoroalkyl sulfonates	40	0	5	1	34
Perfluorooctane sulfonamidoethanols	10	1	0	3	4
Perfluorooctane sulfonamides	15	4	1	1	9
Perfluorooctane sulfonamidoacetic acids	10	1	1	3	4
Samples Stored at -20 °C					
Ether sulfonates	3	0	0	0	3
Fluorotelomer carboxylates	3	0	0	0	3
Fluorotelomer sulfonates	15	0	0	1	14
Per- and Polyfluoroether carboxylates	5	0	0	0	5
Perfluoroalkyl carboxylates	55	0	0	1	54
Perfluoroalkyl sulfonates	40	0	7	1	32

Table 64. Holding Time for Aqueous Matrices per Chemical Group					
	Total # of Observations	# of Times HT was Observed			
Chemical Group		7 days	14 days	28 days	90 days
Perfluorooctane sulfonamidoethanols	10	0	0	0	10
Perfluorooctane sulfonamides	15	0	0	1	14
Perfluorooctane sulfonamidoacetic acids	10	0	0	0	10

Table 65 shows the holding time that was determined for biosolids for each PFAS chemical group and the percentage of observations in each day that had a percent difference greater than 20% when compared to Day 0. The results show that all the compounds are stable up to 90 days when biosolids samples are stored at 4 ºC. If the samples are stored at -20 ºC, then the holding time will be 90 days. However, when the biosolids are frozen, PFDoS would have the holding time decrease to 14 days. Because microbiological activity in biosolids samples at 0 - 6 ºC may lead to production of gases which may cause the sample to be expelled from the container when it is opened, as well as producing noxious odors, EPA strongly recommends that samples be frozen if they need to be stored for more than a few days before extraction.

Table 65. Holding Time for Biosolids Matrices per Chemical Group				
	Total # of Observations	# of Times HT was Observed		
Chemical Group		14 days	28 days	90 days
Samples Stored at 4 ºC				
Ether sulfonates	3	0	0	3
Fluorotelomer carboxylates	3	0	0	3
Fluorotelomer sulfonates	3	0	0	3
Per- and Polyfluoroether carboxylates	5	0	0	5
Perfluoroalkyl carboxylates	11	0	0	11
Perfluoroalkyl sulfonates	8	0	0	8
Perfluorooctane sulfonamidoethanols	2	0	0	2
Perfluorooctane sulfonamides	3	0	0	3
Perfluorooctane sulfonamidoacetic acids	2	0	0	2
Samples Stored at -20 ºC				
Ether sulfonates	3	0	0	3
Fluorotelomer carboxylates	3	0	0	3
Fluorotelomer sulfonates	3	0	0	3
Per- and Polyfluoroether carboxylates	5	0	0	5
Perfluoroalkyl carboxylates	11	0	0	11
Perfluoroalkyl sulfonates	8	1	0	7
Perfluorooctane sulfonamidoethanols	2	0	0	2
Perfluorooctane sulfonamides	3	0	0	3
Perfluorooctane sulfonamidoacetic acids	2	0	0	2

Table 66 shows the holding times that were determined for solid samples for each PFAS chemical group based on a percent difference of 20%, when compared to Day 0. The results show that most of the compounds are stable up to 90 days when the samples are stored at 4 °C, except for ADONA (per- and polyfluoroether carboxylates) and ether sulfonates, which were only marginally over a 20% difference at 28 days. When the sample is stored at -20 ºC, ADONA remained stable up to 90 days, but the ether sulfonates maintained their stability of 28 days. NFDHA is unstable at either storage temperature. For this compound, storage Day 14 was significantly different from Day 0. Since the study did not analyze the samples at any other day between storage Day 0 and 14, it is impossible to know if NFDHA would be stable at a shorter storage period between Day 0 and Day 14 (e.g., 7 days); therefore, the holding time for this compound has been determined to be 0 days. This holding time would not change even if the percent

difference were increased to 30% since the calculated difference is > 40%. The observations for the ether sulfonates in samples stored at 4 ºC had percent differences < 25% while the percent difference for these compounds when the sample was stored at -20 ºC, were < 21%. For solid samples, the best holding time would be 90 days when stored at either 4 ºC or -20 ºC, with the exception of NFDHA, which must be analyzed as soon as possible from time of collection if it is a target analyte of concern.

Table 66. Holding Time for Solid Matrices per Chemical Group

Chemical Group	Total # of Observations	# of Times HT was Observed		
		14 days	28 days	90 days
Samples Stored at 4 ºC				
Ether sulfonates	6	0	2	4
Fluorotelomer carboxylates	6	0	0	6
Fluorotelomer sulfonates	6	0	0	6
Per- and Polyfluoroether carboxylates	10	0	1	8
Perfluoroalkyl carboxylates	22	0	0	22
Perfluoroalkyl sulfonates	16	0	0	16
Perfluorooctane sulfonamidoethanols	4	0	0	4
Perfluorooctane sulfonamides	6	0	0	6
Perfluorooctane sulfonamidoacetic acids	4	0	0	4
Samples Stored at -20 ºC				
Ether sulfonates	6	0	2	4
Fluorotelomer carboxylates	6	0	0	6
Fluorotelomer sulfonates	6	0	0	6
Per- and Polyfluoroether carboxylates	10	0	0	9
Perfluoroalkyl carboxylates	22	0	0	22
Perfluoroalkyl sulfonates	16	0	0	16
Perfluorooctane sulfonamidoethanols	4	0	0	4
Perfluorooctane sulfonamides	6	0	0	6
Perfluorooctane sulfonamidoacetic acids	4	0	0	4

Table 67 shows the holding time that was determined for the sample extracts for all three types of matrices for each PFAS group in each day that had a percent difference greater than 20% when compared to Day 0. The extracts were stored at 4 ºC and no other storage temperature was studied. For all matrices most of the compounds are stable for up to 90 days with the exception of NFDHA in solids and the ether sulfonates in aqueous samples. Therefore, the holding time for sample extracts has been set to 90 days except for NFDHA in solids and ether sulfonates in aqueous samples, which will have a holding time of 28 days.

Table 67. Holding Time for Sample Extracts per Chemical Group

Chemical Group	Total # of Observations	# of Times HT was Observed			
		7 days	15 days	29 days	90 days
Aqueous Sample Extracts					
Ether sulfonates	3	0	0	1	2
Fluorotelomer carboxylates	3	0	0	0	3
Fluorotelomer sulfonates	3	0	0	0	3
Per- and Polyfluoroether carboxylates	5	0	0	0	5
Perfluoroalkyl carboxylates	11	0	0	0	11
Perfluoroalkyl sulfonates	8	0	0	0	8
Perfluorooctane sulfonamidoethanols	2	0	0	0	2
Perfluorooctane sulfonamides	3	0	0	0	3
Perfluorooctane sulfonamidoacetic acids	2	0	0	0	2

Table 67. Holding Time for Sample Extracts per Chemical Group					
Chemical Group	Total # of Observations	# of Times HT was Observed			
		7 days	15 days	29 days	90 days
Biosolids Sample Extracts					
Ether sulfonates	3	-	0	0	3
Fluorotelomer carboxylates	3	-	0	0	3
Fluorotelomer sulfonates	3	-	0	0	3
Per- and Polyfluoroether carboxylates	5	-	0	0	5
Perfluoroalkyl carboxylates	11	-	0	0	11
Perfluoroalkyl sulfonates	8	-	0	0	8
Perfluorooctane sulfonamidoethanols	2	-	0	0	2
Perfluorooctane sulfonamides	3	-	0	0	3
Perfluorooctane sulfonamidoacetic acids	2	-	0	0	2
Solid Samples Extracts					
Ether sulfonates	6	-	0	0	6
Fluorotelomer carboxylates	6	-	0	0	6
Fluorotelomer sulfonates	6	-	0	0	6
Per- and Polyfluoroether carboxylates	10	-	0	1	9
Perfluoroalkyl carboxylates	22	-	0	0	22
Perfluoroalkyl sulfonates	16	-	0	0	16
Perfluorooctane sulfonamidoethanols	4	-	0	0	4
Perfluorooctane sulfonamides	6	-	0	0	6
Perfluorooctane sulfonamidoacetic acids	4	-	0	0	4

References

1 Interstate Technology Regulatory Council (ITRC), PFAS Fact Sheets, https://pfas-1.itrcweb.org/fact-sheets/

2 ASTM 2018. D4841-88(2018), Standard for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents, ASTM International, West Conshohocken, PA, 2017, www.astm.org

3 Woudneh, Million B., Bharat Chandramouli, Coreen Hamilton, Richard Grace, 2019, "Effects of Sample Storage on the Quantitative Determination of 29 PFAS: Observation of Analyte Interconversions during Storage," Environmental Science and Technology 53(21): 12576-12585

Appendix A Background Results for Aqueous and Solid Matrices for the 2018 and 2020 Studies							
	Aqueous Samples (ng/L)				Solid Samples (µg/kg)		
Compounds	**River Water**	**Effluent 1**	**Effluent 2**	**Groundwater**	**Sediment**	**Soil**	**Biosolids**
PFBA				18.2			
PFPeA			1.3	22.2	0.03	0.53	0.41
PFHxA	0.73	1.2	1.5	19.6	0.02	0.45	1.1
PFHpA				7.5		0.35	
PFOA	0.52	1.3	1.1	23.0		1.9	0.52
PFNA				2.1		0.61	
PFDA				0.56		2.4	0.73
PFUnA						0.24	
PFDoA						0.65	
PFTrDA						0.08	0.55
PFTeDA						0.18	0.24
PFBS				20.6			0.38
PFPeS				8.1			0.05
PFHxS				48.1		0.18	0.07
PFHpS				1.3			
PFOS	0.42	0.73	0.56	124		1.1	1.3
PFNS							
PFDS						0.18	
PFDoS							
4:2FTS							
6:2FTS					0.15	2.3	7.7
8:2FTS							
PFOSA				5.6			
NMeFOSA							
NEtFOSA						0.02	
NMeFOSAA							1.3
NEtFOSAA						0.06	0.54
MeFOSE							
NEtFOSE							
HFPO-DA							
ADONA							
9Cl-PF3ONS							
11Cl-PF3OUdS							
3:3 FTCA							
5:3 FTCA							9.9
7:3 FTCA							
PFEESA							
PFMBA							
PFMPA							
NFDHA							