Final Report

Analysis of Mississippi River Pore Water and Surface Water Samples near the 3M Cottage Grove Facility – September 2016 Sampling

Laboratory Request Number: ISO11-01-03-26

Method Requirement: 3M Method ETS-8-044.3

Report Date: Date of Last Signature

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ilac-MRA A2LA ACCREDITED Testing Cert. # 2052.01

The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Testing Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Exhibit 2455
State of Minnesota v. 3M Co.,
Court File No. 27-CV-10-28862

3M Environmental Laboratory

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Analytical Report ISO11-01-03-26

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1 Summary/Introduction

The 3M Environmental Laboratory prepared and analyzed samples collected by Integral Consulting personnel from the Mississippi River near the 3M Cottage Grove facility. Samples were collected September 7-10, 2016. Samples were returned to the 3M Environmental Laboratory on September 11, 2016 and analyzed for perfluorobutanoic acid (PFBA), perfluorooctanoic acid (PFOA), and perfluorooctane sulfonate (PFOS), under 3M Environmental Laboratory project number ISO11-01-03-26.

The 3M Environmental Laboratory prepared sample containers for fifteen sampling locations; twelve pore water (interstitial water; IW) locations and three surface water (SW) locations. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 100 mL. An additional five sample sets were included to be used as needed. Sample bottle sets consisted of a field sample, field sample duplicate, and a target analyte field matrix spike. All samples bottles included the addition of internal standards and surrogate recovery standards (SRSs) [$^{13}C_3$]-PFBA, [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS, which were added to the sample containers prior to being sent to the field for sample collection. Sample bottles reserved for target analyte field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection.

Two different sampling methods were used to collect the pore water samples; Trident, designated as T in the sample description and push point, designated as pp in the sample description. The push point sampling method was used by Weston and Anchor in the past. Integral Consulting replicated this sampling method at three of the nine locations to compare it to the Trident method, which hasn't been used at this site before. A brief description of the Trident and Push point sampling methods are provided in section 2.2.

Samples were prepared and analyzed using solvent dilution according to 3M Environmental Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Internal standards were used to aid in the quantitation of the samples

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.

Table 1. Sample Results Summary. [1]

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFOS Concentration (ng/mL)
Surface Water Locations				
ISO11-01-03-26-003	IW-14b-sw - Sample	<0.0250	0.0566	0.0260
ISO11-01-03-26-003-DUP	IW-14b-sw - Sample Duplicate	<0.0250	0.0500	<0.0232
	Average	**<0.0250** [2]	**0.0533**	**0.0260**
	%RPD Sample/Sample Dup	**NA**	**12**	**NA**
ISO11-01-03-26-009	IW-19-sw - Sample	<0.0250	<0.0240	<0.0232
ISO11-01-03-26-009-DUP	IW-19-sw - Sample Duplicate	<0.0250	<0.0240	<0.0232
	Average	**<0.0250** [2]	**<0.0240** [2]	**<0.0232** [2]
	%RPD Sample/Sample Dup	**NA**	**NA**	**NA**
ISO11-01-03-26-015	IW-25f-sw - Sample	<0.0250	<0.0240	<0.0232
ISO11-01-03-26-015-DUP	IW-25f-sw - Sample Duplicate	<0.0250	<0.0240	<0.0232
	Average	**<0.0250**	**<0.0240**	**<0.0232** [2]
	%RPD Sample/Sample Dup	**NA**	**NA**	**NA**
ISO11-01-03-26-018	IW-19-sw-dup - Sample	<0.0250	<0.0240	<0.0232
ISO11-01-03-26-018-DUP	IW-19-sw-dup - Sample Duplicate	<0.0250	<0.0240	<0.0232
	Average	**<0.0250**	**<0.0240**	**<0.0232**
	%RPD Sample/Sample Dup	**NA**	**NA**	**NA**
Interstitial Water (Pore Water)				
ISO11-01-03-26-001	IW-14b-pw-T - Sample	27.8	99.7	13.4
ISO11-01-03-26-001-DUP	IW-14b-pw-T - Sample Duplicate	28.2	98.9	12.2
	Average	**28.0** [3]	**99.3** [3]	**12.8** [3]
	%RPD Sample/Sample Dup	**1.4**	**0.81**	**9.4**
ISO11-01-03-26-002	IW-14b-pw-pp - Sample	**21.7** [4]	**94.0** [4]	**20.0** [4]
ISO11-01-03-26-004	IW-14-pw-T - Sample	32.7	66.0	9.46
ISO11-01-03-26-004-DUP	IW-14-pw-T - Sample Duplicate	33.9	64.6	7.93
	Average	**33.3** [3]	**65.3** [3]	**8.70** [3]
	%RPD Sample/Sample Dup	**3.6**	**2.1**	**18**
ISO11-01-03-26-005	IW-14f-pw-T - Sample	92.6	50.4	<0.0232
ISO11-01-03-26-005-DUP	IW-14f-pw-T - Sample Duplicate	94.8	44.4	<0.0232
	Average	**93.7** [3]	**47.4** [3]	**<0.0232**
	%RPD Sample/Sample Dup	**2.3**	**13**	**NA**

NA = Not Applicable

(1) All samples associated with the reported sampling location were analyzed using solvent dilution with internal standard calibration unless noted otherwise. The analytical data uncertainties for the reported results are as follows: PFBA ± 19%, PFOA ± 11%, and PFOS ± 13%.

(2) The field matrix spike sample for the location did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded, see section 4 of the report for additional information.

(3) All samples associated with the reported sampling location were analyzed using solvent dilution with external standard calibration unless noted otherwise. The analytical data uncertainties for the reported results are as follows: PFBA ± 34%, PFOA ± 20%, and PFOS ± 20%.

(4) Due to insufficient sample volume, a field duplicate and field matrix spike sample were not collected.

(5) Due to insufficient sample volume, a field duplicate was not collected; however a field matrix spike sample was collected.

(6) The RPD value did not meet method acceptance criteria of ≤20%.

Table 1 continued. Sample Results Summary. [1]

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFOS Concentration (ng/mL)
Interstitial Water (Pore Water)				
ISO11-01-03-26-006	IW-19b-pw-T - Sample	62.2	147	14.1
ISO11-01-03-26-006-DUP	IW-19b-pw-T - Sample Duplicate	61.7	150	11.5
	Average	**62.0** [3]	**149** [3]	**12.8** [3]
	%RPD Sample/Sample Dup	**0.81**	**2.0**	**20**
ISO11-01-03-26-007	IW-19b-pw-pp - Sample	**18.3** [5]	**47.9** [5]	**17.7** [5]
ISO11-01-03-26-008	IW-19-pw-T - Sample	89.3	121	37.4
ISO11-01-03-26-008-DUP	IW-19-pw-T - Sample Duplicate	91.3	124	36.9
	Average	**90.3** [3]	**123** [3]	**37.2** [3]
	%RPD Sample/Sample Dup	**2.2**	**2.4**	**1.3**
ISO11-01-03-26-010	IW-19f-pw-T - Sample	59.7	150	3.86
ISO11-01-03-26-010-DUP	IW-19f-pw-T - Sample Duplicate	54.8	152	3.78
	Average	**57.3** [3]	**151** [3]	**3.82** [3]
	%RPD Sample/Sample Dup	**8.6**	**1.3**	**2.1**
ISO11-01-03-26-011	IW-25b-pw-T - Sample	69.5	64.8	101
ISO11-01-03-26-011-DUP	IW-25b-pw-T - Sample Duplicate	66.6	68.3	109
	Average	**68.1** [3]	**66.6** [3]	**105** [3]
	%RPD Sample/Sample Dup	**4.3**	**5.3**	**7.6**
ISO11-01-03-26-012	IW-25b-pw-pp - Sample	73.9	62.3	78.3
ISO11-01-03-26-012-DUP	IW-25b-pw-pp - Sample Duplicate	72.0	61.9	83.3
	Average	**73.0** [3]	**62.1** [3]	**80.8** [3]
	%RPD Sample/Sample Dup	**2.6**	**0.64**	**6.2**
ISO11-01-03-26-013	IW-25-pw-T - Sample	27.6	36.5	0.104
ISO11-01-03-26-013-DUP	IW-25-pw-T - Sample Duplicate	27.4	30.9	0.108
	Average	**27.5** [3]	**33.7** [3]	**0.106**
	%RPD Sample/Sample Dup	**0.73**	**17**	**3.8**
ISO11-01-03-26-014	IW-25f-pw-T - Sample	21.0	20.3	3.61
ISO11-01-03-26-014-DUP	IW-25f-pw-T - Sample Duplicate	24.0	18.7	2.48
	Average	**22.5** [3]	**19.5** [3]	**3.05**
	%RPD Sample/Sample Dup	**13**	**8.2**	**37** [6]

NA = Not Applicable

(1) All samples associated with the reported sampling location were analyzed using solvent dilution with internal standard calibration unless noted otherwise. The analytical data uncertainties for the reported results are as follows: PFBA ± 19%, PFOA ± 11%, and PFOS ± 13%.

(2) The field matrix spike sample for the location did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded, see section 4 of the report for additional information.

(3) All samples associated with the reported sampling location were analyzed using solvent dilution with external standard calibration unless noted otherwise. The analytical data uncertainties for the reported results are as follows: PFBA ± 34%, PFOA ± 20%, and PFOS ± 20%.

(4) Due to insufficient sample volume, a field duplicate and field matrix spike sample were not collected.

(5) Due to insufficient sample volume, a field duplicate was not collected; however a field matrix spike sample was collected.

(6) The RPD value did not meet method acceptance criteria of ≤20%.

Table 1 continued. Sample Results Summary. [1]

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFOS Concentration (ng/mL)
Interstitial Water (Pore Water)				
ISO11-01-03-26-016	IW-25b-pw-T-dup - Sample	**68.9** [4]	**67.2** [4]	**105** [4]
Field Blanks (Trip and Equipment)				
ISO11-01-03-26-017	EB-pw-pp - Sample	<0.0250	<0.0240	<0.0232
ISO11-01-03-26-017-DUP	EB-pw-pp - Sample Duplicate	<0.0250	<0.0240	<0.0232
	Average	**<0.0250**	**<0.0240**	**<0.0232**
	%RPD Sample/Sample Dup	**NA**	**NA**	**NA**
ISO11-01-03-26-021	Travel Blank	<0.0250	<0.0240	<0.0232
ISO11-01-03-26-022	Equipment Blank	<0.0250	<0.0240	<0.0232

NA = Not Applicable

(1) All samples associated with the reported sampling location were analyzed using solvent dilution with internal standard calibration unless noted otherwise. The analytical data uncertainties for the reported results are as follows: PFBA ± 19%, PFOA ± 11%, and PFOS ± 13%.

(2) The field matrix spike sample for the location did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded, see section 4 of the report for additional information.

(3) All samples associated with the reported sampling location were analyzed using solvent dilution with external standard calibration unless noted otherwise. The analytical data uncertainties for the reported results are as follows: PFBA ± 34%, PFOA ± 20%, and PFOS ± 20%.

(4) Due to insufficient sample volume, a field duplicate and field matrix spike sample were not collected.

(5) Due to insufficient sample volume, a field duplicate was not collected; however a field matrix spike sample was collected.

(6) The RPD value did not meet method acceptance criteria of ≤20%.

2 Method Summary

2.1 Methods

Analysis for PFBA, PFOA, and PFOS was completed following 3M Environmental Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS".

Table 2. Target Analytes.

Target Analytes	Acronym	Reference Material Structure
Perfluorobutanoate (C4 Acid)	PFBA	Linear
Perfluorooctanoate (C8 Acid)	PFOA	Linear + Branched
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched

2.2 Sample Collection

Pore water (interstitial water) and surface water samples were collected in 125 mL Nalgene™ (high-density polyethylene) bottles prepared at the 3M Environmental Laboratory. For each sample set, a field sample, field sample duplicate, and field matrix spike container were provided. A set of laboratory prepared Trip Blank and Trip Blank field matrix spike samples were sent with the collection bottles. Sample bottles were received by the laboratory on September 11, 2016. Samples were stored refrigerated at the laboratory after receipt. Due to the limited volume of sample collected at locations IW-14b-pw-T (ISO11-01-03-26-002), IW-19b-pw-T (ISO11-01-03-26-006), IW-19b-pw-pp (ISO11-01-

03-26-007), and IW-25b-pw-T-dup (ISO11-01-03-26-016), not all the sample containers in the set were filled. A copy of the chain of custody form is included as an attachment to report for reference.

Two different sampling methods were used to collect the pore water samples; Trident and Push point. The push point sampling method was used by Weston and Anchor in the past. Integral Consulting replicated this sampling method at three of the nine locations to compare it to the Trident method, which hasn't been used at this site before. Below is a brief description of the two sampling methods.

Trident:
The Trident probe is a direct-push, integrated temperature sensor, conductivity sensor, grain-size sensor and porewater sampler developed to screen sites for areas where groundwater may be discharging to a surface water body. Differences in observed conductivity and temperature indicate areas where groundwater discharge is occurring. The integral porewater sampler can be used to rapidly confirm the presence of groundwater constituents and map the subsurface distribution of contaminants of concern.

Push point:
The water-sampling probe allows interstitial waters to be extracted from the sediment at selected depths up to about 90 cm below the sediment water interface. Porewater is collected by a low-flow peristaltic pump extraction through a small-diameter, stainless steel probe. The probes consist of a length of 9.5 mm diameter stainless steel tubing fitted with a solid, removable point. On the side of the tube near the tip there is a sample port consisting of a hole covered by a small mesh size stainless steel screen. The porewater sampler can also be configured with a secondary screen with a sand-pack to provide a pre-filter for the sampling and to minimize clogging of the sampler. This secondary screen is installed over the outside of the probe, and the void between the probe and the screen is packed with pre-cleaned sand. Multiple probes can be used to together to further increase surface area, enhance sampling rate, and minimize potential clogging.

2.3 Sample Preparation

All samples were initially prepared on 9/15/16 and analyzed on 9/16/16 for all analytes by diluting with methanol. Samples were diluted 10-fold by removing a 0.5 mL aliquot of the well mixed sample and diluting it with 4.5 mL of methanol. Diluted samples and LCSs were fortified with 0.01 mL of a solution containing surrogate recovery standard (SRSs) [$^{13}C_3$]-PFBA, [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS spiked at a nominal concentration of 1 ng/mL. The laboratory control samples were prepared and analyzed in the same manner as the samples. Prepared samples were analyzed by external standard calibration.

Samples that were at or below the limit of quantitation when analyzed using the 10-fold dilution were re-prepared on 10/4/16 by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL) prior to diluting in the same manner as the samples. The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. Prepared samples were analyzed on 10/5/16 by internal standard calibration.

2.4 Analysis

All samples and quality control samples were analyzed for PFBA, PFOA, PFOS, SRSs, and ISs using high performance liquid chromatography/ tandem mass spectrometry (HPLC/MS/MS). Detailed instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the raw data hard copies placed in the final data packet, and are briefly described below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used

for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

Table 3. Instrument Parameters.

Instrument Name	ETS Jonas	ETS DaVinci
Analysis Dates	9/16/16 – External Calibration	10/5/16 – Internal and External Calibration
Analytical Method	ETS-8-044.3	ETS-8-044.3
Liquid Chromatograph	Agilent 1200	Agilent 1260
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5μ	Betasil C18 (2.1 mm X 100 mm), 5μ
Injection Volume	2 or 5 μL	10 μL
Mass Spectrometer	Applied Biosystems API 5000	Applied Biosystems API 6500
Ion Source	Turbo Spray	Turbo Spray
Electrode	Turbo ion electrode	Turbo ion electrode
Polarity	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Conditions.

ETS-8-044.3				
Step Number	Total Time (min)	Flow Rate (μL/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions.

Analyte	Mass Transition Q1/Q3	Internal Standard (1)	Mass Transition Q1/Q3
PFBA	213/169	[$^{13}C_4$]-PFBA	217/172
PFOA	413/369	[$^{13}C_8$]-PFOA	421/376
	413/219		
	413/169		
PFOS	499/99	[$^{13}C_8$]-PFOS	507/80
	499/80		
	499/130		
[$^{13}C_3$]-PFBA	216/172	[$^{13}C_4$]-PFBA	217/172
[$^{13}C_4$]-PFOA	417/372	[$^{13}C_8$]-PFOA	421/376
[$^{13}C_4$]-PFOS	503/80	[$^{13}C_8$]-PFOS	507/80

Dwell time was 20 or 50 msec for each transition. The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standards were not used for the quantitation of samples analyzed on 9/16/16 and 10/5/16 for location ISO11-01-03-26-013 (IW-25-pw-T).

3 Analytical Results

3.1 Calibration

Internal Standard Analysis (10/5/16) - Samples were quantitated for all analytes against an internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 methanol: laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. Ten standards ranging from 0.02 ng/mL to 10 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

External Standard Analysis (9/16/16 and 10/5/16 for ISO11-01-03-26-013) - Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution into 50 mL of 90:10 methanol: laboratory Milli-Q™ water. Twelve standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers. Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes in each analysis.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria of less than or equal to 5% relative standard deviation (RSD) for peak area/ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes in each analysis.

3.3 Limit of Quantitation (LOQ)

The LOQ for each analysis is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts/ratio are at least twice those of the appropriate blanks. The LOQ for all analytes can be found in Table 6.

Table 6. Limit of Quantitation (LOQ).

Analyte	9/16/16 External Standard Calibration Analysis LOQ, ng/mL [1]	10/5/16 Internal Standard Calibration Analysis LOQ, ng/mL [2]	10/5/16 External Standard Calibration Analysis LOQ, ng/mL [2]
PFBA	5.00	0.0250	NA
PFOA	0.479	0.0240	NA
PFOS	0.200	0.0232	0.0232

NA = Not Applicable
(1) A dilution factor of 10 was applied to the LOQ.
(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of each analytical sequence, continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported sample results were bracketed by CCVs that met method criteria of 100% ± 25%.

3.5 Blanks

Four types of blanks were prepared and analyzed with the samples: solvent blanks, method (procedural) blanks, field/trip blanks, and equipment rinseate blanks for the water samples. Each blank result was reviewed and used to evaluate method performance. Procedural blank results were reviewed according to the method and used to evaluate method performance to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared and analyzed in triplicate. The LCS samples were prepared by spiking known amounts of the analyte into 10 mL of laboratory reagent water or 1 mL of laboratory Milli-Q™ water to produce the desired concentration. The LCSs were diluted with methanol in the same manner as the samples. All LCS results were used to determine overall method uncertainty in Section 3.7.

The method acceptance criteria states that the average recovery of LCS be 100% ± 20% with a RSD ≤20%, when evaluated independently at each concentration level. All LCSs met acceptance criteria with the following exceptions:

9/16/16 Analysis: The low set of LCSs for PFBA had an average recovery of 134%.

The batch LCS recovery results were reviewed when evaluating the analytical data uncertainty in section 3.7 of the report. The following calculations were used to generate data in Table 7 for laboratory control spikes.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%$$

$$\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%$$

Table 7. Laboratory Control Spike Recovery.

ETS-8-044.3 External Calibration Analyzed 9/16/16	PFBA			PFOA (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160915-1	9.90	13.5	136	9.90	11.0	111
LCS-160915-2	9.90	12.7	128	9.90	11.0	111
LCS-160915-3	9.90	13.1	132	9.90	11.1	113
Average ± %RSD	**134% ± 3.0% (1)**			**112% ± 1.8%**		
LCS-160915-4	99.0	114	115	99.0	109	110
LCS-160915-5	99.0	110	111	99.0	109	110
LCS-160915-6	99.0	105	106	99.0	106	107
Average ± %RSD	**111% ± 4.1%**			**109% ± 1.6%**		
LCS-160915-7	498	477	95.9	498	463	93.0
LCS-160915-8	498	489	98.1	498	467	93.8
LCS-160915-9	498	510	102	498	494	99.1
Average ± %RSD	**98.7% ± 3.1%**			**95.3% ± 3.5%**		

ETS-8-044.3 External Calibration Analyzed 9/16/16	PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160915-1	9.90	9.65	97.4
LCS-160915-2	9.90	9.46	95.5
LCS-160915-3	9.90	9.90	100
Average ± %RSD	**97.6% ± 2.3%**		
LCS-160915-4	99.0	99.4	100
LCS-160915-5	99.0	101	102
LCS-160915-6	99.0	97.6	98.5
Average ± %RSD	**100% ± 1.8%**		
LCS-160915-7	498	468	94.1
LCS-160915-8	498	485	97.3
LCS-160915-9	498	507	102
Average ± %RSD	**97.8% ± 4.1%**		

(1) The average recovery did not meet acceptance criteria of 100 ± 30%.
(2) LCS was spiked post dilution, but reported with the dilution factor applied.

Table 7 continued. Laboratory Control Spike Recovery.

ETS-8-044.3 External Calibration Analyzed 9/16/16	$[^{13}C_3]$-PFBA (2)			$[^{13}C_4]$-PFOA (2)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160915-1	0.988	1.05	106	0.988	1.05	106
LCS-160915-2	0.988	1.05	107	0.988	1.05	107
LCS-160915-3	0.988	0.972	98.4	0.988	0.972	98.4
Average ± %RSD	**104% ± 4.5%**			**104% ± 4.5%**		
LCS-160915-4	39.8	42.2	106	39.8	42.2	106
LCS-160915-5	39.8	43.3	109	39.8	43.3	109
LCS-160915-6	39.8	42.3	106	39.8	42.3	106
Average ± %RSD	**107% ± 1.6%**			**107% ± 1.6%**		

ETS-8-044.3 External Calibration Analyzed 9/16/16	[13C4]-PFOS (2)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160915-1	0.947	1.02	108
LCS-160915-2	0.947	1.07	113
LCS-160915-3	0.947	1.01	107
Average ± %RSD	**109% ± 2.9%**		
LCS-160915-4	38.1	41.9	110
LCS-160915-5	38.1	44.1	116
LCS-160915-6	38.1	42.7	112
Average ± %RSD	**113% ± 2.7%**		

(1) The average recovery did not meet acceptance criteria of 100 ± 30%.

(2) LCS was spiked post dilution, but reported with the dilution factor applied.

Table 7 continued. Laboratory Control Spike Recovery.

ETS-8-044.1 Internal Calibration Analyzed 10/5/16	PFBA			PFOA (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161004-1	0.198	0.162	81.6	0.190	0.159	83.9
LCS-161004-2	0.198	0.146	73.5	0.190	0.170	89.7
LCS-161004-3	0.198	0.160	80.6	0.190	0.159	83.7
Average ± %RSD	**81.1% ± 1.2%**			**83.8% ± 0.24%**		
LCS-161004-4	1.98	2.03	102	1.90	1.73	91.2
LCS-161004-5	1.98	1.99	101	1.90	1.80	94.6
LCS-161004-6	1.98	2.02	102	1.90	1.81	95.3
Average ± %RSD	**102% ± 0.57%**			**93.7% ± 2.3%**		
LCS-161004-7	39.6	35.6	89.9	38.0	32.4	85.4
LCS-161004-8	39.6	35.6	89.9	38.0	32.8	86.4
LCS-161004-9	39.6	35.8	90.5	38.0	33.3	87.6
Average ± %RSD	**90.1% ± 0.38%**			**86.5% ± 1.3%**		

ETS-8-044.1 Internal Calibration Analyzed 10/5/16	PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161004-1	0.184	0.155	84.3
LCS-161004-2	0.184	0.160	86.8
LCS-161004-3	0.184	0.167	90.7
Average ± %RSD	**87.3% ± 3.7%**		
LCS-161004-4	1.84	1.76	95.8
LCS-161004-5	1.84	1.70	92.5
LCS-161004-6	1.84	1.80	98.0
Average ± %RSD	**95.4% ± 2.9%**		
LCS-161004-7	36.7	32.0	87.2
LCS-161004-8	36.7	34.2	93.2
LCS-161004-9	36.7	33.4	90.9
Average ± %RSD	**90.4% ± 3.3%**		

(1) The average recovery did not meet acceptance criteria of 100 ± 30%.

(2) LCS was spiked post dilution, but reported with the dilution factor applied.

Table 7 continued. Laboratory Control Spike Recovery.

ETS-8-044.3 Internal Calibration Analyzed 10/5/16	$[^{13}C_3]$-PFBA			$[^{13}C_4]$-PFOA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161004-1	0.197	0.184	93.6	0.198	0.169	85.3
LCS-161004-2	0.197	0.182	92.2	0.198	0.170	85.9
LCS-161004-3	0.197	0.179	90.8	0.198	0.173	87.3
Average ± %RSD	**92.2% ± 1.5%**			**86.2% ± 1.2%**		
LCS-161004-4	1.97	1.87	94.7	1.98	1.84	92.7
LCS-161004-5	1.97	1.90	96.4	1.98	1.81	91.6
LCS-161004-6	1.97	1.86	94.4	1.98	1.87	94.5
Average ± %RSD	**95.2% ± 1.1%**			**92.9% ± 1.6%**		

ETS-8-044.3 Internal Calibration Analyzed 10/5/16	$[^{13}C_4]$-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161004-1	0.189	0.166	87.6
LCS-161004-2	0.189	0.182	96.2
LCS-161004-3	0.189	0.162	85.5
Average ± %RSD	**89.8% ± 6.3%**		
LCS-161004-4	1.89	1.71	90.6
LCS-161004-5	1.89	1.68	89.1
LCS-161004-6	1.89	1.74	91.8
Average ± %RSD	**90.5% ± 1.5%**		

(1) The average recovery did not meet acceptance criteria of 100 ± 30%.

(2) LCS was spiked post dilution, but reported with the dilution factor applied.

Table 7 continued. Laboratory Control Spike Recovery.

ETS-8-044.1 External Calibration Analyzed 10/5/16	PFOS (Linear + Branched)			[$^{13}C_4$]-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161004-1	0.184	0.163	88.6	0.189	0.177	93.4
LCS-161004-2	0.184	0.155	84.2	0.189	0.179	94.5
LCS-161004-3	0.184	0.161	87.7	0.189	0.158	83.7
Average ± %RSD	**86.8% ± 2.7%**			**90.5% ± 6.6%**		
LCS-161004-4	1.84	1.83	99.6	1.89	1.84	97.5
LCS-161004-5	1.84	1.87	102	1.89	1.92	102
LCS-161004-6	1.84	1.83	99.7	1.89	1.82	96.5
Average ± %RSD	**100% ± 1.4%**			**98.7% ± 3.0%**		
LCS-161004-7	36.7	33.5	91.4			
LCS-161004-8	36.7	35.2	95.8			
LCS-161004-9	36.7	38.5	105			
Average ± %RSD	**97.4% ± 7.1%**					

(1) The average recovery did not meet acceptance criteria of 100 ± 30%

(2) LCS was spiked post dilution, but reported with the dilution factor applied.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.3. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The analytical method uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%. When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in Table 8 below.

- The analytical method uncertainty when calculated by ETS-12-012.3 for PFBA by external standard calibration was ± 16%; however, based on the average recovery for the low level LCSs analyzed on 9/16/16, the analytical data uncertainty was expanded to ± 34%.

Table 8. Analytical Method Uncertainty

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty (%)
PFBA	Internal	9.51	± 19
PFOA	Internal	5.67	± 11
PFOS	Internal	6.35	± 13
PFBA	External	NA	± 34
PFOA	External	10.2	± 20
PFOS	External	9.90	± 20

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spikes (FMS) were prepared for each location with the spike level selected based on the results from the last sampling of these locations in 2014. FMSs were generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. FMS recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the extraction and analysis of the analytes of interest. FMS concentrations must be at least 50% of the sample concentration to be considered an appropriate spike level. The reference standards for PFOA and PFOS in the field matrix spiking solution consisted of linear and branched isomers. Table 9 lists the locations and spiking levels for which a target analyte FMS was prepared.

In addition, field matrix spikes for this project consisted of stable isotope surrogate recovery standard spikes (SRSs) of [$^{13}C_3$]-PFBA, [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to all sample bottles prior to sample collection. The [$^{13}C_3$]-labeled PFBA was selected to represent PFBA the [$^{13}C_4$]-labeled PFOA was selected to represent PFOA, and the [$^{13}C_4$]-labeled PFOS was selected to represent PFOS. Following sample analysis, it was suspected that the surrogate recovery standards were not spiked as intended; therefore, no surrogate recovery standard results were used to assess sample recovery.

The following calculation was used to generate the field matrix spike recovery in Section 4 of the report:

$$\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%$$

Table 9. Field Matrix Spike Levels.

Sampling location	PFBA (ng/mL)	PFOA (ng/mL)	PFOS (ng/mL)
IW-14b-sw, IW-19-sw, IW-25f-sw	0.100	0.100	0.100
IW-14-pw	2.00	2.00	2.00
IW-14f-pw	5.00	5.00	5.00
IW-25f-pw, Sets 1 – 5	10.0	10.0	10.0
IW-25-pw	25.0	25.0	25.0
IW-14b-pw, IW-19b-pw, IW-19-pw, IW-19f-pw	50.0	50.0	50.0
IW-25b-pw	200	200	200
Trip Blank	50.0	50.0	50.0
IW-19b-pw (1)	379	379	379

(1) Due to the limited volume of sample collected, the field matrix spike concentration was adjusted for the final fill volume of 13.2 mL.

4 Data Summary and Discussion

The tables below summarize the sample results and target analyte field matrix spike recoveries for the sampling locations as well as the Trip Blank. Results and values are average rounded to three significant figures according to EPA rounding rules. Because of rounding, values may vary slightly from those listed in the raw data. Field matrix spike meeting the method acceptance criteria of ± 30%, demonstrate that the method is appropriate for the given matrix and their respective quantitative ranges. Because of rounding, values may vary slightly from those listed in the raw data. Field matrix spikes and surrogate recoveries meeting the method acceptance criteria of ±30%, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. The field matrix spike concentration was selected based on the expected concentration of PFOA and/or PFOS, based on the results from the last sampling of these locations in 2014. As a result the spike level, at times, exceeded the recommended upper limit of 10 times the analyte concentration. In these instances the FMS recovery was reported and flagged as above 10 times the sample concentration. All surrogate recovery standards and field matrix spike recoveries met method acceptance criteria with the following exceptions.

IW-14b-pw-T: Due to the limited volume of sample collected, the field duplicate and field matrix spike sample containers were not filled. The surrogate recovery standards, which met method acceptance criteria, were used to assess method accuracy.

IW-14-sw: The FMS recovery for PFBA was 143%. The method uncertainty has been expanded to ±43% for PFBA.

IW-19b-pw-T: Due to the limited volume of sample collected, the field matrix spike sample container was not filled. The field sample and field sample duplicate had a RPD of 22% for PFOS. The surrogate recovery standards, which met method acceptance criteria, were used to assess method accuracy.

IW-19b-pw-pp: Due to the limited volume of sample collected, the field duplicate sample container was not filled. The field matrix spike container was under filled and the target analyte spike concentration adjusted accordingly. The resulting spike concentration was not appropriate as compared to the sample concentration for PFBA and PFOS, but did meet method acceptance criteria. The results have been flagged accordingly.

IW-19-pw-T: The field duplicate sample had a SRS recovery of 135% for both [$^{13}C_3$]-PFBA and [$^{13}C_4$]-PFOS; however, the average recovery for the sample set for [$^{13}C_3$]-PFBA and [$^{13}C_4$]-PFOS met method acceptance criteria and no adjustment was made to the data uncertainty.

IW-19-sw: The FMS recovery for PFBA was 138%, for PFOA 136% and for PFOS 138%. The method uncertainty has been expanded for PFBA to ±38%, for PFOA to ±36%, and for PFOS to ±38%.

IW-25b-pw-T-dup: Due to the limited volume of sample collected, the field duplicate and field matrix spike sample containers were not filled. The surrogate recovery standards, which met method acceptance criteria, were used to assess method accuracy.

Table 10. IW-14b-pw-T

		PFBA		PFOA		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-001	IW-14b-pw-T - Sample	27.8	NA	99.7	NA	13.4	NA
ISO11-01-03-26-001-DUP	IW-14b-pw-T - Sample Duplicate	28.2	NA	98.9	NA	12.2	NA
ISO11-01-03-26-001-FMS	IW-14b-pw-T - FMS	83.3	111	145	91.4	62.6	99.6
Average Concentration (ng/mL) ± %RPD		**28.0 ng/mL ± 1.4%**		**99.3 ng/mL ± 0.81%**		**12.8 ng/mL ± 9.4%**	

NA = Not Applicable
Samples were analyzed by external standard calibration.

Table 11. IW-14b-sw

		PFBA		PFOA		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-003	IW-14b-sw - Sample	<0.0250	NA	0.0566	NA	0.0260	NA
ISO11-01-03-26-003-DUP	IW-14b-sw - Sample Duplicate	<0.0250	NA	0.0500	NA	<0.0232	NA
ISO11-01-03-26-003-FMS	IW-14b-sw - FMS	0.143	143 (1)	0.173	120	0.144	118
Average Concentration (ng/mL) ± %RPD		**<0.0250 ng/mL (2)**		**0.0533 ng/mL ± 12%**		**0.0260 ng/mL**	

NA = Not Applicable
Samples were analyzed by internal standard calibration.
(1) The field matrix spike did not meet method acceptance criteria of 100 ± 30%.
(2) The method uncertainty has been expanded for PFBA to ± 43%.

Table 12. IW-14-pw-T

		PFBA		PFOA		PFOS	
3M LIMS ID	**Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO11-01-03-26-004	IW-14-pw-T - Sample	32.7	NA	66.0	NA	9.46	NA
ISO11-01-03-26-004-DUP	IW-14-pw-T - Sample Duplicate	33.9	NA	64.6	NA	7.93	NA
ISO11-01-03-26-004-FMS	IW-14-pw-T - FMS	33.4	NC	65.3	NC	11.0	NC
Average Concentration (ng/mL) ± %RPD		**33.3 ng/mL ± 3.6%**		**65.3 ng/mL ± 2.1%**		**8.70 ng/mL ± 18%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
Samples were analyzed by external standard calibration.

Table 13. IW-14f-pw-T

		PFBA (1)		PFOA (1)		PFOS (2)	
3M LIMS ID	**Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO11-01-03-26-005	IW-14f-pw-T - Sample	92.6	NA	50.4	NA	<0.0232	NA
ISO11-01-03-26-005-DUP	IW-14f-pw-T - Sample Duplicate	94.8	NA	44.4	NA	<0.0232	NA
ISO11-01-03-26-005-FMS	IW-14f-pw-T - FMS	106	NC	57.2	NC	5.86 (3)	117
Average Concentration (ng/mL) ± %RPD		**93.7 ng/mL ± 2.3%**		**47.4 ng/mL ± 13%**		**<0.0232 ng/mL**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples were analyzed by external standard calibration.
(2) Samples were analyzed by internal standard calibration.
(3) FMS spike concentration was greater than 10X the endogenous sample concentration.

Table 14. IW-19b-pw-pp

	PFBA	PFOA	PFOS

3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-007	IW-19b-pw-pp - Sample	18.3	NA	47.9	NA	17.7	NA
ISO11-01-03-26-0007-FMS	IW-19b-pw-pp - FMS	398 (1)	100	413	96.4	374 (1)	94.1
Concentration (ng/mL)		**18.3 ng/mL**		**47.9 ng/mL**		**17.7 ng/mL**	

NA = Not Applicable
Samples were analyzed by external standard calibration.
(1) FMS spike concentration was greater than 10X the endogenous sample concentration.

Table 15. IW-19-pw-T

		PFBA		PFOA		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-008	IW-19-pw-T - Sample	89.3	NA	121	NA	37.4	NA
ISO11-01-03-26-008-DUP	IW-19-pw-T - Sample Duplicate	91.3	NA	124	NA	36.9	NA
ISO11-01-03-26-008-FMS	IW-19-pw-T - FMS	137	93.4	174	103	88.1	102
Average Concentration (ng/mL) ± %RPD		**90.3 ng/mL ± 2.2%**		**123 ng/mL ± 2.4%**		**37.2 ng/mL ± 1.3%**	

NA = Not Applicable
Samples were analyzed by external standard calibration.

Table 16. IW-19-sw

	PFBA	PFOA	PFOS

3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-009	IW-19-sw - Sample	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-009-DUP	IW-19-sw - Sample Duplicate	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-009-FMS	IW-19-sw - FMS	0.138	138 (1)	0.136	136 (1)	0.138	138 (1)
Average Concentration (ng/mL) ± %RPD		**<0.0500 ng/mL (2)**		**<0.0240 ng/mL (2)**		**<0.0232 ng/mL (2)**	

NA = Not Applicable
Samples were analyzed by internal standard calibration.
(1) The field matrix spike did not meet method acceptance criteria of 100 ± 30%.
(2) The method uncertainty has been expanded for PFBA to ± 38%, for PFOA to ± 36%, and for PFOS to ± 38%.

Table 17. IW-19f-pw-T

		PFBA		PFOA		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-010	IW-19f-pw-T - Sample	59.7	NA	150	NA	3.86	NA
ISO11-01-03-26-010-DUP	IW-19f-pw-T - Sample Duplicate	54.8	NA	152	NA	3.78	NA
ISO11-01-03-26-010-FMS	IW-19f-pw-T - FMS	105	95.5	180	NC	52.8 (1)	98.0
Average Concentration (ng/mL) ± %RPD		**57.3 ng/mL ± 8.6%**		**151 ng/mL ± 1.3%**		**3.82 ng/mL ± 2.1%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
Samples were analyzed by external standard calibration.
(1) FMS spike concentration was greater than 10X the endogenous sample concentration

Table 18. IW-25b-pw-T

	PFBA	PFOA	PFOS

3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-011	IW-25b-pw-T - Sample	69.5	NA	64.8	NA	101	NA
ISO11-01-03-26-011-DUP	IW-25b-pw-T - Sample Duplicate	66.6	NA	68.3	NA	109	NA
ISO11-01-03-26-011-FMS	IW-25b-pw-T - FMS	285	108	279	106	337	116
Average Concentration (ng/mL) ± %RPD		**68.1 ng/mL± 4.3%**		**66.6 ng/mL ± 5.3%**		**105 ng/mL ± 7.6%**	

NA = Not Applicable
Samples were analyzed by external standard calibration.

Table 19. IW-25b-pw-pp

		PFBA		PFOA		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-012	IW-25b-pw-pp - Sample	73.9	NA	62.3	NA	78.3	NA
ISO11-01-03-26-012-DUP	IW-25b-pw-pp - Sample Duplicate	72.0	NA	61.9	NA	83.3	NA
ISO11-01-03-26-012-FMS	IW-25b-pw-pp - FMS	282	105	260	99.0	272	95.6
Average Concentration (ng/mL) ± %RPD		**73.0 ng/mL± 2.6%**		**62.1 ng/mL ± 0.64%**		**80.8 ng/mL ± 6.2%**	

NA = Not Applicable
Samples were analyzed by external standard calibration.

Table 20. IW-25-pw-T

	PFBA	PFOA	PFOS

3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-013	IW-25-pw-T - Sample	27.6	NA	36.5	NA	0.104	NA
ISO11-01-03-26-013-DUP	IW-25-pw-T - Sample Duplicate	27.4	NA	30.9	NA	0.108	NA
ISO11-01-03-26-013-FMS	IW-25-pw-T - FMS	51.7	96.8	60.4	107	25.2	100
Average Concentration (ng/mL) ± %RPD		**27.5 ng/mL± 0.73%**		**33.7 ng/mL ± 17%**		**0.106 ng/mL ± 3.8%**	

NA = Not Applicable
(1) Samples were analyzed by external standard calibration.
(2) Samples were analyzed by internal standard calibration.

Table 21. IW-25f-pw-T

		PFBA [1]		PFOA [1]		PFOS [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-014	IW-25f-pw-T - Sample	21.0	NA	20.3	NA	3.61	NA
ISO11-01-03-26-014-DUP	IW-25f-pw-T - Sample Duplicate	24.0	NA	18.7	NA	2.48	NA
ISO11-01-03-26-014-FMS	IW-25f-pw-T - FMS	32.3	98.0	30.6	111	15.0	120
Average Concentration (ng/mL) ± %RPD		**22.5 ng/mL± 13%**		**19.5 ng/mL ± 8.2%**		**3.05 ng/mL ± 37% [3]**	

NA = Not Applicable
(1) Samples were analyzed by external standard calibration.
(2) Samples were analyzed by internal standard calibration.
(3) The sample / sample duplicate did not meet method acceptance criteria of ≤20%.

Table 22. IW-25f-sw

	PFBA	PFOA	PFOS

3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-015	IW-25f-sw - Sample	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-015-DUP	IW-25f-sw - Sample Duplicate	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-015-FMS	IW-25f-sw - FMS	0.108	108	0.120	120	0.137	137 (1)
Average Concentration (ng/mL) ± %RPD		**<0.0250 ng/mL**		**<0.0240 ng/mL**		**<0.0232 ng/mL (2)**	

NA = Not Applicable
Samples were analyzed by internal standard calibration.
(1) The field matrix spike did not meet method acceptance criteria of 100 ± 30%.
(2) The method uncertainty has been expanded for PFOS to ± 37%.

Table 23. Extra Bottle Set 1; EB-pw-pp (Equipment Blank for push point)

		PFBA		PFOA		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-017	EB-pw-pp - Sample	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-017-DUP	EB-pw-pp - Sample Duplicate	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-017-FMS	EB-pw-pp - FMS	11.4 (1)	114	10.8 (1)	108	11.6 (1)	116
Average Concentration (ng/mL) ± %RPD		**<0.0250 ng/mL**		**<0.0240 ng/mL**		**<0.0232 ng/mL**	

NA = Not Applicable
Samples were analyzed by internal standard calibration.
(1) FMS spike concentration was greater than 10X the endogenous sample concentration.

Table 24. Extra Bottle Set 2; IW-19-sw-dup

	PFBA	PFOA	PFOS

3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-018	IW-19-sw-dup - Sample	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-018-DUP	IW-19-sw-dup - Sample Duplicate	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-018-FMS	IW-19-sw-dup - FMS	11.1 (1)	111	11.4 (1)	114	11.1 (1)	111
Average Concentration (ng/mL) ± %RPD		**<0.0250 ng/mL**		**<0.0240 ng/mL**		**<0.0232 ng/mL**	

NA = Not Applicable
Samples were analyzed by internal standard calibration.
(1) FMS spike concentration was greater than 10X the endogenous sample concentration.

Table 25. Travel Blank and Equipment Blank (1)

		PFBA		PFOA		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-26-021	Travel Blank	<0.0250	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-26-021-FMS	Travel Blank FMS	50.4 (2)	101	53.3 (2)	107	50.9 (2)	102
ISO11-01-03-26-022	Equipment Blank	<0.0250	NA	<0.0240	NA	<0.0232	NA

NA = Not Applicable
(1) Samples were analyzed by internal standard calibration unless noted otherwise.
(2) Samples were analyzed by external standard calibration.

Table 26. Surrogate Recovery (1)

3M LIMS ID	Description	[$^{13}C_3$]-PFBA %Recovery	[$^{13}C_4$]-PFOA %Recovery	[$^{13}C_4$]-PFOS %Recovery
ISO11-01-03-26-001	IW-14b-pw-T - Sample	119	115	125
ISO11-01-03-26-001-DUP	IW-14b-pw-T - Sample Duplicate	107	100	110
ISO11-01-03-26-001-FMS	IW-14b-pw-T - FMS	112	103	115
ISO11-01-03-26-002	IW-14b-pw-pp - Sample	95.9	95.	102
ISO11-01-03-26-003	IW-14b-sw - Sample	97.6 (2)	101 (2)	96.8 (2)
ISO11-01-03-26-003-DUP	IW-14b-sw - Sample Duplicate	111 (2)	102 (2)	97.9 (2)
ISO11-01-03-26-003-FMS	IW-14b-sw - FMS	101 (2)	106 (2)	106 (2)
ISO11-01-03-26-004	IW-14-pw-T - Sample	107	107	113
ISO11-01-03-26-004-DUP	IW-14-pw-T - Sample Duplicate	111	106	115
ISO11-01-03-26-004-FMS	IW-14-pw-T - FMS	112	108	110
ISO11-01-03-26-005	IW-14f-pw-T - Sample	109	104	99.0 (2)
ISO11-01-03-26-005-DUP	IW-14f-pw-T - Sample Duplicate	111	109	108 (2)
ISO11-01-03-26-005-FMS	IW-14f-pw-T - FMS	114	109	118 (2)
ISO11-01-03-26-006	IW-19b-pw-T - Sample	115	111	102 (2)
ISO11-01-03-26-006-DUP	IW-19b-pw-T - Sample Duplicate	113	109	102 (2)
ISO11-01-03-26-007	IW-19b-pw-pp - Sample	115	108	114
ISO11-01-03-26-0007-FMS	IW-19b-pw-pp - FMS	79.9	78.9	81.7
ISO11-01-03-26-008	IW-19-pw-T - Sample	115	110	116
ISO11-01-03-26-008-DUP	IW-19-pw-T - Sample Duplicate	135 (3)	124	135 (3)
ISO11-01-03-26-008-FMS	IW-19-pw-T - FMS	114	107	117
ISO11-01-03-26-009	IW-19-sw - Sample	99.7 (2)	102 (2)	99.0 (2)
ISO11-01-03-26-009-DUP	IW-19-sw - Sample Duplicate	96.0 (2)	99.1 (2)	91.5 (2)
ISO11-01-03-26-009-FMS	IW-19-sw - FMS	102 (2)	101 (2)	109 (2)
ISO11-01-03-26-010	IW-19f-pw-T - Sample	113	109	114
ISO11-01-03-26-010-DUP	IW-19f-pw-T - Sample Duplicate	113	109	116
ISO11-01-03-26-010-FMS	IW-19f-pw-T - FMS	114	112	120
ISO11-01-03-26-011	IW-25b-pw-T - Sample	117	110	121
ISO11-01-03-26-011-DUP	IW-25b-pw-T - Sample Duplicate	117	111	116
ISO11-01-03-26-011-FMS	IW-25b-pw-T - FMS	107	104	110
ISO11-01-03-26-012	IW-25b-pw-pp - Sample	124	120	126
ISO11-01-03-26-012-DUP	IW-25b-pw-pp - Sample Duplicate	118	112	120
ISO11-01-03-26-012-FMS	IW-25b-pw-pp - FMS	111	99.5	113
ISO11-01-03-26-013	IW-25-pw-T - Sample	106	106	99.1 (2)
ISO11-01-03-26-013-DUP	IW-25-pw-T - Sample Duplicate	105	100	95.3 (2)
ISO11-01-03-26-013-FMS	IW-25-pw-T - FMS	105	108	97.9 (2)
ISO11-01-03-26-014	IW-25f-pw-T - Sample	103	98.5	106 (2)
ISO11-01-03-26-014-DUP	IW-25f-pw-T - Sample Duplicate	98.0	94.5	101 (2)
ISO11-01-03-26-014-FMS	IW-25f-pw-T - FMS	113	106	106 (2)

(1) Samples contained surrogate prior to additional surrogate being added during sample preparation and analyzed by external standard calibration, unless noted otherwise.
(2) Samples analyzed by internal standard calibration.
(3) The SRS did not meet method acceptance criteria of 100 ± 30%; however the average recovery of the SRS for the sample set was within method acceptance criteria.

Table 26 continued. Surrogate Recovery [1]

3M LIMS ID	Description	[$^{13}C_3$]-PFBA %Recovery	[$^{13}C_4$]-PFOA %Recovery	[$^{13}C_4$]-PFOS %Recovery
ISO11-01-03-26-015	IW-25f-sw - Sample	94.9 [2]	99.6 [2]	102 [2]
ISO11-01-03-26-015-DUP	IW-25f-sw - Sample Duplicate	96.0 [2]	98.3 [2]	100 [2]
ISO11-01-03-26-015-FMS	IW-25f-sw - FMS	93.8 [2]	101 [2]	93.8 [2]
ISO11-01-03-26-016	IW-25b-pw-T-dup	113	113	111
ISO11-01-03-26-017	EB-pw-pp - Sample	85.8 [2]	89.5 [2]	87.7 [2]
ISO11-01-03-26-017-DUP	EB-pw-pp - Sample Duplicate	86.9 [2]	99.8 [2]	101 [2]
ISO11-01-03-26-017-FMS	EB-pw-pp - FMS	113 [2]	105 [2]	109 [2]
ISO11-01-03-26-018	IW-19-sw-dup - Sample	106 [2]	101 [2]	97.1 [2]
ISO11-01-03-26-018-DUP	IW-19-sw-dup - Sample Duplicate	102 [2]	94.2 [2]	104 [2]
ISO11-01-03-26-018-FMS	IW-19-sw-dup - FMS	96.7	108 [2]	93.3 [2]
ISO11-01-03-26-021	Travel Blank	97.6 [2]	99.1 [2]	95.9 [2]
ISO11-01-03-26-021-FMS	Travel Blank FMS	115	110	110
ISO11-01-03-26-022	Equipment Blank	100 [2]	98.0 [2]	98.6 [2]

(1) Samples contained surrogate prior to additional surrogate being added during sample preparation and analyzed by external standard calibration, unless noted otherwise.
(2) Samples analyzed by internal standard calibration.
(3) The SRS did not meet method acceptance criteria of 100 ± 30%; however the average recovery of the SRS for the sample set was within method acceptance criteria.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike and lab matrix spike recoveries (SRS addition during sample preparation) demonstrated that the analytical method was appropriate for the given sample matrix. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10- 26 of this report.

6 Data/Sample Retention

All remaining samples and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Attachment

Chain of custody form

8 Signatures

Susan Wolf, 3M Principal Analytical Investigator

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative

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