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8EHQ-80-373
AL-226-3892

3M

CERTIFIED MAIL

09 APR 15 AM 8:00

April 9, 2009

NO CBI

Document Processing Center
EPA East – Room 6428 Attn: Section 8(e)
Office of Pollution Prevention and Toxics, U.S. EPA
1200 Pennsylvania Avenue NW
Washington, DC 20460-0001

Re: TSCA 8(e) Substantial Risk Notice: Supplemental to Docket No. 8EHQ-0598-373; Sulfonate-based and Carboxylate-based Fluorochemicals

To whom it may concern:

3M is submitting this notice to supplement its previous submissions on sulfonate and carboxylate-based fluorochemicals.

3M recently conducted fluorochemical (FC) analysis of 33 fish fillet tissues of walleye, northern pike, sauger, bluegill, smallmouth bass, and black crappie as split samples collected by the Minnesota Pollution Control Agency (MPCA) from the Mississippi River.

Levels of PFOS quantified in all tissues were comparable to those reported by the MPCA (March 2009, http://www.pca.state.mn.us/publications/c-pfc1-01.pdf) and ranged from 28.5 to 382 ng/g. In addition, levels of perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUnA), and perfluorododecanoic acid (PFDoA) were quantified in most of the samples. PFDA levels ranged from 0.581-10.2 ng/g, PFUnA levels ranged from 0.288-5.81 ng/g, and PFDoA levels ranged from 0.271-3.38 ng/g.

The PFOS data have been finalized and are provided in the enclosed report. Data for the other analytes are currently being finalized, and will be forwarded to EPA upon completion of the reports.

While 3M does not believe that any of these data taken alone or cumulatively meet the "substantial risk" reporting threshold, we nevertheless recognize the ongoing work by U.S. EPA to assess FC exposure pathways. Therefore, we are placing these results in the 8(e) docket as a supplement to previous submissions.

If you have any questions or would like any additional information, please contact Deanna Luebker at (651) 737-1374 or djluebker@mmm.com.

Sincerely,

Jean B Sweeney (AL)

Jean B. Sweeney
Staff Vice President, 3M Environmental, Health and Safety Operations

Enclosure

Contains No CBI CONTAINS NO CBI

318508

Final Analytical Report

Perfluorochemical Analysis of Fish Fillet Homogenates collected from the Mississippi River by the Minnesota Pollution Control Agency Phase I: PFOS

Laboratory Request Number: E09-0053

Method Requirement: ETS 8-45.0

Testing Laboratory

3M EHS Operations
3M Environmental Laboratory
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Building 260-5N-17
Maplewood, MN 55144

Requester

Gary Hohenstein
3M EHS Operations
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Building 224-5W-03
Maplewood, MN 55144

ACCREDITED

The testing reported herein meet the requirements of ISO/IEC 17025-2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Certificate #2052-01. Testing that complies with this International Standard also operate in accordance with ISO 9001:2000.

Certificate #2052-01

3M Environmental Laboratory
3M Environmental Laboratory Manager: William K. Reagen, Ph.D.
3M Principal Analytical Investigator and Report Author: Michelle D. Malinsky, Ph.D.

Analytical Report E09-0053 Phase I

Perfluorochemical Analysis of Fish Fillet Homogenates collected from the Mississippi River by the Minnesota Pollution Control Agency Phase I: PFOS

April 1, 2009

1 Introduction

The Minnesota Pollution Control Agency (MPCA) submitted thirty-three fish fillet homogenates to the 3M Environmental Laboratory for perfluorochemical analysis under project number E09-0053. The samples were extracted and analyzed using method ETS 8-45.0 "Determination of Fluorochemicals via Protein Precipitation of Fish Tissues (Fillet or Whole Body) and Analysis by High Performance Liquid Chromatography with Tandem Mass Spectrometry". The target analytes for this study included perfluoroalkane carboxylic acids ranging from C4 to C12, perfluoroalkane sulfonates (C4, C6, and C8), and perfluorooctanesulfonamide. This phase I report summarizes the results for perfluorooctane sulfonate (PFOS) only. The results for the other target analytes will be issued in a phase II report.

The 3M Environmental Laboratory maintains A2LA accreditation to ISO/IEC 17025 for the specific tests/calibrations listed in A2LA Certificate #2052-01 and meets the relevant quality systems requirements of ISO 9001:1994. ETS 8-45.0 falls under the laboratory's current scope of accreditation. The data and final report were audited for compliance to laboratory's ISO/IEC17025 quality system.

A key for the sample descriptions is provided in Section 2.2.

Table 1 below summarizes the PFOS results. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.

ACCREDITED

The testing reported herein meet the requirements of ISO/IEC 17025-2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Certificate #2052-01. Testing that complies with this International Standard also operate in accordance with ISO 9001:2000.

Certificate #2052-01

Table 1. Sample Results Summary: PFOS

		PFOS
3M LIMS ID	*Sample Description*	*[1]Conc. (ng/g)*
E09-0053-001	MissR_pool2_WE_1	28.7
E09-0053-001 Dup		28.4
	Average	***28.5***
	%RPD	***1.0***
E09-0053-002	MissR_pool2_WE_2	45.9
E09-0053-002 Dup		43.4
	Average	***44.7***
	%RPD	***5.8***
E09-0053-003	MissR_pool2_WE_3	84.3
E09-0053-003 Dup		81.9
	Average	***83.1***
	%RPD	***2.9***
E09-0053-004	MissR_pool2_WE_4	51.0
E09-0053-004 Dup		43.0
	Average	***47.0***
	%RPD	***17***
E09-0053-005	MissR_pool2_SAG_1	41.1
E09-0053-005 Dup		38.6
	Average	***39.8***
	%RPD	***6.3***
E09-0053-006	MissR_pool2_SAG_2	43.7
E09-0053-006 Dup		43.4
	Average	***43.5***
	%RPD	***0.68***
E09-0053-007	MissR_pool2_SMB_1	57.5
E09-0053-007 Dup		50.4
	Average	***54.0***
	%RPD	***13***
E09-0053-008	MissR_pool2_SMB_2	Ø379
E09-0053-008 Dup		Ø384
	Average	***Ø382***
	%RPD	***1.4***
E09-0053-009	MissR_pool2_SMB_3	111
E09-0053-009 Dup		123
	Average	***117***
	%RPD	***10***
E09-0053-010	MissR_pool2_SMB_4	131
E09-0053-010 Dup		122
	Average	***127***
	%RPD	***7.1***

Table 1 Continued.

3M LIMS ID	Sample Description	PFOS (1)Conc. (ng/g)
E09-0053-011	MissR_pool2_SMB_5	141
E09-0053-011 Dup		150
	Average	***146***
	%RPD	***6.2***
E09-0053-012	MissR_pool2_BKS_1	55.5
E09-0053-012 Dup		54.5
	Average	***55.0***
	%RPD	***1.8***
E09-0053-013	MissR_pool2_BGS_1	155
E09-0053-013 Dup		168
	Average	***161***
	%RPD	***7.8***
E09-0053-014	MissR_pool2_BGS_2	110
E09-0053-014 Dup		100
	Average	***105***
	%RPD	***9.4***
E09-0053-015	MissR_pool2_BGS_3	188
E09-0053-015 Dup		167
	Average	***177***
	%RPD	***11***
E09-0053-016	MissR_pool2_BGS_4	(2)331
E09-0053-016 Dup		(2)340
	Average	(2)***336***
	%RPD	***2.7***
E09-0053-017	MissR_pool2_BGS_5	123
E09-0053-017 Dup		120
	Average	***122***
	%RPD	***2.0***
E09-0053-018	MissR_pool3_WE_1	97.4
E09-0053-018 Dup		95.5
	Average	***96.4***
	%RPD	***2.0***
E09-0053-019	MissR_pool3_WE_2	78.3
E09-0053-019 Dup		78.6
	Average	***78.5***
	%RPD	***0.37***
E09-0053-020	MissR_pool3_WE_3	97.4
E09-0053-020 Dup		82.3
	Average	***89.9***
	%RPD	***17***
E09-0053-021	MissR_pool3_WE_4	49.4
E09-0053-021 Dup		52.3
	Average	***50.8***
	%RPD	***5.7***

Table 1 Continued.

3M LIMS ID	Sample Description	PFOS [1] Conc. (ng/g)
E09-0053-022	MissR_pool3_WE_5	64.2
E09-0053-022 Dup		68.1
	Average	***66.2***
	%RPD	***5.8***
E09-0053-023	MissR_pool3_NOP_1	49.8
E09-0053-023 Dup		51.2
	Average	***50.5***
	%RPD	***2.8***
E09-0053-024	MissR_pool3_SMB_1	29.5
E09-0053-024 Dup		29.9
	Average	***29.7***
	%RPD	***1.1***
E09-0053-025	MissR_pool3_SMB_2	103
E09-0053-025 Dup		108
	Average	***105***
	%RPD	***4.9***
E09-0053-026	MissR_pool3_SMB_3	48.8
E09-0053-026 Dup		48.3
	Average	***48.5***
	%RPD	***1.0***
E09-0053-027	MissR_pool3_SMB_4	63.0
E09-0053-027 Dup		66.9
	Average	***64.9***
	%RPD	***6.0***
E09-0053-028	MissR_pool3_SMB_5	55.0
E09-0053-028 Dup		53.3
	Average	***54.2***
	%RPD	***3.1***
E09-0053-029	MissR_pool3_BGS_1	76.7
E09-0053-029 Dup		72.8
	Average	***74.7***
	%RPD	***5.3***
E09-0053-030	MissR_pool3_BGS_2	160
E09-0053-030 Dup		165
	Average	***163***
	%RPD	***2.6***
E09-0053-031	MissR_pool3_BGS_3	138
E09-0053-031 Dup		128
	Average	***133***
	%RPD	***7.6***
E09-0053-032	MissR_pool3_BGS_4	223
E09-0053-032 Dup		219
	Average	***221***
	%RPD	***1.7***

Table 1 Continued.

3M LIMS ID	Sample Description	PFOS (1) Conc. (ng/g)
E09-0053-033	MissR_pool3_BGS_5	126
E09-0053-033 Dup		133
	Average	***129***
	%RPD	***5.7***

(1) Unless otherwise specified, the recovery of associated laboratory matrix spike (LMS) was within 100±30% which meets ETS 8-45.0 method acceptance criteria. Sample results are considered accurate to within the overall analytical method uncertainty 100±15% (PFOS). See Section 4.9 *Determination of Analytical Method Uncertainty* for more information. Sample results are reported to three significant figures and %RPD to two significant figures. Additional significant figures were used to calculate the average, %RPD, and %recovery. Values in the raw data may vary slightly due to rounding.

(2) Sample results were reported from a 1:10 post-extraction dilution.

2. Study and Sample Information

2.1 Target Analytes.

Table 2 below provides pertinent information regarding the target analytes, internal standards, and surrogate compounds investigated in this study.

Table 2. Target Analyte Summary.

Compound Name	Synonym or Acronym	Analytical Purpose	Formula	Reference Standard Source
Perfluorobutanoic acid	PFBA (C4 Acid)	Target	C_3F_7COOH	Aldrich
Perfluoropentanoic acid	PFPeA (C5 Acid)	Target	C_4F_9COOH	Alfa Aesar
Perfluorohexanoic acid	PFHxA (C6 Acid)	Target	$C_5F_{11}COOH$	Oakwood Products
Perfluoroheptanoic acid	PFHpA (C7 Acid)	Target	$C_6F_{13}COOH$	Aldrich
Perfluorooctanoic acid	PFOA (C8 Acid)	Target	$C_7F_{15}COOH$	L-PFOA (linear) – Wellington Labs
			$[C_7F_{15}COO^-][NH_4^+]$	ECF PFOA (branched) 3M Production Lot 332
Perfluorononanoic acid	PFNA (C9 Acid)	Target	$C_8F_{17}COOH$	Oakwood Products
Perfluorodecanoic acid	PFDA (C10 Acid)	Target	$C_9F_{19}COOH$	Oakwood Products
Perfluoroundecanoic acid	PFUnA (C11 Acid)	Target	$C_{10}F_{21}COOH$	Oakwood Products
Perfluorododecanoic acid	PFDoA (C12 Acid)	Target	$C_{11}F_{23}COOH$	Oakwood Products
Perfluorobutane sulfonate	PFBS (C4 Sulfonate)	Target	$[C_4F_9SO_3^-][K^+]$	3M Production Lot 2
Perfluorohexane sulfonate	PFHS (C6 Sulfonate)	Target	$[C_6F_{13}SO_3^-][Na^+]$	L-PFHS (linear)- Wellington Labs
Perfluorooctane sulfonate	PFOS (C8 Sulfonate)	Target	$[C_8F_{17}SO_3^-][K^+]$	L-PFOS (linear)- Wellington Labs
			$[C_8F_{17}SO_3^-][K^+]$	ECF PFOS (branched) 3M Production Lot 217

Table 2 Continued.

Compound Name	*Synonym or Acronym*	*Analytical Purpose*	*Formula*	*Reference Standard Source*
Perfluorooctanesulfonamide	FOSA (C8 Sulfonamide)	Target	$C_8F_{17}SO_2NH_2$	3M (L-15709)
$^{13}C_4$-Perfluorobutanoic acid	[1,2,3,4-$^{13}C_4$]PFBA	IS	$^{13}CF_3(^{13}CF_2)_2{}^{13}COOH$	Wellington Labs
$^{13}C_2$-Perfluorohexanoic acid	[1,2 -$^{13}C_2$]PFHxA	IS	$CF_3(CF_2)_3(^{13}CF_2)^{13}COOH$	Wellington Labs
$^{13}C_4$-Perfluorooctanoic acid	[1,2,3,4-$^{13}C_4$]PFOA	IS	$CF_3(CF_2)_3(^{13}CF_2)_3{}^{13}COOH$	Wellington Labs
$^{13}C_5$-Perfluorononanoic acid	[1,2,3,4,5-$^{13}C_5$]PFNA	IS	$CF_3(CF_2)_3(^{13}CF_2)_4{}^{13}COOH$	Wellington Labs
$^{13}C_2$-Perfluorodecanoic acid	[1,2 -$^{13}C_2$]PFDA	IS	$CF_3(CF_2)_7(^{13}CF_2)^{13}COOH$	Wellington Labs
$^{13}C_2$-Perfluoroundecanoic acid	[1,2 -$^{13}C_2$]PFUnA	IS	$CF_3(CF_2)_8(^{13}CF_2)^{13}COOH$	Wellington Labs
$^{13}C_2$-Perfluorododecanoic acid	[1,2 -$^{13}C_2$]PFDoA	IS	$CF_3(CF_2)_9(^{13}CF_2)^{13}COOH$	Wellington Labs
$^{18}O_2$-Ammonium Perfluorobutane sulfonate	[$^{18}O_2$]PFBS	IS	$[C_4F_9S^{18}O_2O^-][NH_4^+]$	RTI International
$^{18}O_2$-Ammonium Perfluorohexane sulfonate	[$^{18}O_2$]PFHS	IS	$[C_6F_{13}S^{18}O_2O^-][NH_4^+]$	Wellington Labs
$^{13}C_4$-Sodium Perfluorooctane sulfonate	[1,2,3,4-$^{13}C_4$]PFOS	IS	$[CF_3(CF_2)_3(^{13}CF_2)_3{}^{13}CF_2SO_3^-][Na^+]$	Wellington Labs
$^{13}C_2$-Perfluorooctanoic acid	[1,2-$^{13}C_2$]PFOA	Surrogate	$CF_3(CF_2)_5(^{13}CF_2)^{13}COOH$	Perkin Elmer
$^{18}O_2$-Ammonium Perfluorooctane sulfonate	[$^{18}O_2$]PFOS	Surrogate	$[C_8F_{17}S^{18}O_2O^-][NH_4^+]$	RTI International

2.2 Sample Descriptions.

The table below summarizes the abbreviations provided by the MPCA used to identify the fish species collected for this study. No information was provided regarding the location of sample collection within Pool 2 or Pool 3 of the Mississippi River.

Table 3. Sample Description Key.

Species	*Abbreviation*
Bluegill	BGS
Walleye	WE
Black Crappie	BKS
Sauger	SAG
Smallmouth Bass	SMB
Northern Pike	NOP

2.3 Sample Collection and Receipt.

The chain of custody provided by the MPCA indicated that the samples were collected on June 5 and June 9, 2008. On January 27, 2009, the 3M Environmental Laboratory received the homogenized fillet samples in Nalgene bottles on dry ice. The samples were logged into the 3M Environmental Laboratory LIMS system under project E09-0053 and placed in frozen storage. Information provided by the MPCA indicated that the samples were homogenized with the skin on and scales removed; however, no specifics regarding the homogenization procedure were given.

It should be noted that these fish samples have been previously handled and analyzed at another laboratory under contract by the MPCA. It is unknown how these samples have been handled during analysis and storage. It is possible that some, or all, of these samples may possibly have been compromised during the previous handling, analysis and storage. No assumptions are being made to the integrity of these samples.

3M Environmental Laboratory personnel removed the homogenized fish tissue from the sample container by cutting the bottle away from the frozen sample mass using a clean utility knife. The frozen sample mass was then placed in a plastic bag and returned to frozen storage before the tissue was homogenized before extraction. Based on the sample consistency in the Nalgene bottles, it appeared that the sample homogenates had thawed at some point before they arrived at the 3M Environmental Laboratory. (Homogenized tissue had refrozen into a solid mass that could not be broken up while frozen in the container.)

3 Methods - Analytical and Preparatory

3.1 Tissue Preparation

Each submitted sample and bluegill fillet control tissue (TN08-0220-1/1) purchased from Osage Catfisheries, Inc. (Osage Beach, MO) were homogenized frozen using dry ice.

Depending on the sample size, frozen fish tissue was added to a pre-chilled stainless steel bowl of either a Robot Coupe RSI 2Y-1 or a RSI 23B vertical batch processor and homogenized with dry ice until a fine powder consistency was reached. After homogenization, the ground fish tissue was transferred to a polyethylene bag which was placed in the freezer at -20°C unsealed overnight to allow the residual carbon dioxide to sublime. After sublimation, the bag was sealed and homogenates were kept frozen.

3.2 Sample Extraction

Polyethylene centrifuge tubes (50 mL) were chilled in a cooler with dry ice for approximately thirty minutes prior to weighing homogenate aliquots to prevent the tissue powder from thawing and congealing during the weighing process. A 0.5 g sub-sample of the fish tissue homogenate was accurately weighed in a pre-chilled 50 mL polyethylene centrifuge tube. Tissue aliquots were then spiked with internal standard (IS) and surrogates to produce an approximate concentration of 1 ng/g. Samples designated as lab matrix spikes or matrix-matched calibration standards were additionally spiked with the target and surrogate analytes at appropriate levels to produce the desired tissue concentrations. Samples intended for laboratory QC (matrix blanks and laboratory control matrix spikes) were spiked with surrogates at 1 ng/g and the other target analytes at appropriate levels. After spiking, the tissue homogenates were allowed to sit for 30 minutes before 5 mL of acetonitrile was added. The samples were then thoroughly homogenized with the acetonitrile using an Omni Prep multi-place homogenizer with disposable plastic probes (15,000 rpm for 2 minutes). The acetonitrile/tissue extracts were then placed in a freezer at -20°C for at least one hour. Upon removal from the freezer, sample extracts were centrifuged at a targeted temperature of -5°C for 20 minutes at 3000 rpm. If any delays occurred, samples were returned to the -20°C freezer and then re-centrifuged.

After centrifugation, 1 mL of clarified supernatant was transferred to a 2 mL autovial spiked with 10 µL of 10% formic acid. Autovials were then capped and vortex mixed.

Samples were extracted in three separate batches. For each submitted sample, a sample, sample duplicate, and laboratory matrix spike (LMS) were prepared. Two samples were re-extracted in a fourth batch as the associated LMSs prepared the first time were not high enough for resultant endogenous concentration. Each preparation batch consisted of the following calibration and quality control elements:

- a nine point extracted matrix calibration curve using the purchased bluegill fillet control tissue
- triplicate lab control spikes of the bluegill fillet control tissue at three levels (0.05 ng/g, 1.5 ng/g, and 8 ng/g)
- triplicate lab control spikes of 3M electrochemical fluorination (ECF) PFOA and PFOS at 5 ng/g
- duplicate control matrix blanks with and without internal standards and surrogates
- duplicate method (aqueous) blanks with and without internal standards and surrogates

The table below summarizes the preparation dates and samples prepared in each batch.

Table 4. Sample Preparation Summary.

Batch Number	*Preparation Dates*	*Samples*
Batch 1	2/18/2009 – 2/20/2009	E09-0053-001 – E09-0053-011
Batch 2	2/19/2009 – 2/23/2009	E09-0053-012 – E09-0053-022
Batch 3	2/24/2009 – 2/25/2009	E09-0053-023 – E09-0053-033
Batch 4	3/5/2009 – 3/6/2009	E09-0053-008 & E09-0053-016 (reprep)

3.3 Analysis

Analysis for the suite of PFCs listed above was performed using high performance liquid chromatography-tandem mass spectrometry (HPLC/MS/MS). For this investigation, samples were analyzed using an Agilent 1100 series (Palo Alto, CA) HPLC system interfaced to either a PE SCIEX API 4000 triple quadrupole or SCIEX API 4000 Q-Trap mass spectrometer (Foster City, CA). Both instruments were equipped with a SCIEX Turbo V ion-spray interface operating in the negative ion MS/MS mode using multiple reaction monitoring (MRM). Analyst™ 1.4.2 software was used for all data collection and reduction. Table 5 lists the MRM transitions monitored for each analyte. A Thermo Scientific PRISM RP guard column (2.1mm × 50 mm; 5µ particle size) was placed in-line after the purge valve and before the sample injection port to trap any PFC contaminants coming from the HPLC instrument and/or the mobile phases. This sufficiently separated the elution of the "system" PFC peaks from those present in the sample extracts.

Table 5. MRM Transition Summary.

Compound	Analyte Description	[1]MRM Transition(s)	Dwell Time (ms)
PFBA (C4 Acid)	Target	213>169	100
PFPeA (C5 Acid)	Target	263>219	100
PFHxA (C6 Acid)	Target	313>269	100
		313>119	100
PFHpA (C7 Acid)	Target	363>319	50
		363>169	50
PFOA (C8 Acid)	Target	413>369	50
		413>219	50
		413>169	50
PFNA (C9 Acid)	Target	463>419	50
		463>219	50
		463>169	50
PFDA (C10 Acid)	Target	513>469	50
		513>269	50
		513>219	50
PFUnA (C11 Acid)	Target	563>519	50
		563>269	50
		563>219	50
PFDoA (C12 Acid)	Target	613>569	50
		613>319	50
		613>169	50
PFBS (C4 Sulfonate)	Target	299>80	50
		299>99	50
PFHS (C6 Sulfonate)	Target	399>80	50
		399>99	50
PFOS (C8 Sulfonate)	Target	499>80	50
		499>99	50
		499>130	50
FOSA (C8 Sulfonamide)	Target	498>78	50
[1,2,3,4 -$^{13}C_4$]PFBA	IS for PFBA and PFPeA	217>172	100
[1,2 -$^{13}C_2$]PFHxA	IS for PFHxA	315>270	100
[1,2,3,4-$^{13}C_4$]PFOA	IS for PFHpA and PFOA	417>372	50
[1,2,3,4,5-$^{13}C_5$]PFNA	IS for PFNA	468>423	50
[1,2 -$^{13}C_2$]PFDA	IS for PFDA	515>470	50
[1,2 -$^{13}C_2$]PFUnA	IS for PFUnA	565>520	50
[1,2 -$^{13}C_2$]PFDoA	IS for PFDoA	615>570	50
[$^{18}O_2$]PFBS	IS for PFBS	303>84	50
[$^{18}O_2$]PFHS	IS for PFHS	403>84	50
[1,2,3,4- $^{13}C_4$]PFOS	IS for PFOS, FOSA	503>80	50
[1,2 - $^{13}C_2$]PFOA	Surrogate (Acids)	415>370	50
[$^{18}O_2$]PFOS	Surrogate(Sulfonates FOSA)	503>84	50

(1) Individual transitions were summed for each analyte to produce a "total ion chromatogram" (TIC). The TICs were used for quantitation.

Analysis of the small acids (C4 through C6) was performed using a Thermo Scientific PRISM RP column (2.1mm × 50 mm; 5 µ particle size) held at 30°C with the mobile phase system consisting of 5 mM ammonium acetate with 0.01% acetic acid in water (A) and methanol (B). The gradient used to elute the analytes of interest is presented in Table 6. The flow rate was held at 300 µL/min throughout the run and a 20 µL injection volume was used. The outlet of the analytical column was directed to a column switching valve where the first three minutes of the run were diverted to waste. After three minutes, the valve was switched to direct the effluent to the mass spectrometer for analysis.

The large acids (C7 through C12), the sulfonates, and FOSA were analyzed using a Thermo Scientific Betasil™ C18 analytical column (2.1mm × 100 mm; 5µ particle size) held at 30°C with the mobile phase system consisting of 2 mM ammonium acetate in water (A) and acetonitrile (B). The gradient used is also provided in Table 6. The flow rate was held at 400 µL/min throughout the run and a 25 µL injection volume was used. Samples were injected onto a Waters (Milford, MA) Oasis® HLB on-line extraction column (20 mm × 3.0 mm, 25 µ particle size) with the outlet directed to a column switching valve where the first five minutes were diverted to waste. After five minutes, the valve was switched and the effluent was directed to the analytical column for separation of the target analytes and analysis by the mass spectrometer.

Table 6. LC Gradient Parameters.

Small Acids: C4-C6				*Large Acids: C7-C12; Sulfonates, FOSA*			
Step	*Total Time (min)*	*%A*	*%B*	*Step*	*Total Time (min)*	*%A*	*%B*
0	0.0	90	10	0	0.0	97	3.0
1	3.0	90	10	1	3.0	97	3.0
2	3.5	30	70	2	3.5	70	30
3	9.0	5.0	95	3	13.5	40	60
4	15.0	5.0	95	4	15.5	40	60
5	15.1	90	10	5	16.0	10	90
6	19.0	90	10	6	18.0	10	90
				7	18.3	97	3.0
				8	21.0	97	3.0
A	5 mM ammonium acetate with 0.01% acetic acid (aq)			A	2 mM ammonium acetate (aq)		
B	Methanol			B	Acetonitrile		

To manage the large number of transitions required for the large acids, sulfonates, and FOSA, a multiperiod method was constructed. The first period (time=0 minutes to 10.6 minutes) collected the transitions for PFBS, PFHpA, PFOA, [1,2 - $^{13}C_2$]PFOA, PFHS, PFNA and their respective ISs. The second period (time=10.5 min to 21.0 minutes) collected the transitions for PFDA, PFUnA, PFOS, [$^{18}O_2$]PFOS, PFDoA, and FOSA and their respective ISs. For analytes where more than one transition was acquired, a total ion chromatogram (TIC) which summed the respective transitions for that analyte was used to analyze the data.

4 Data Analysis

4.1 Calibration

Extracted *matrix-matched calibration standards and QCs were prepared by spiking known amounts of target analytes, surrogates, and internal standards into individual 0.5 g aliquots of bluegill fillet control

tissue (TN08-0202-1/1) homogenate. Each spiked fish aliquot was then extracted using the procedures outlined in Section 3.2. A total of nine spiked standards ranging from 0.25 ng/g to 25 ng/g (nominal) were prepared. Additionally, an unextracted acetonitrile solvent curve ranging from 0.025 ng/mL to 50 ng/mL was also prepared. All sample results for PFOS and [$^{18}O_2$]PFOS were quantitated against the solvent curve as sample extract concentrations for PFOS exceeded the upper calibration range of the extracted curve. To demonstrate equivalency, the extracted curve and the laboratory control QC samples were quantitated using the solvent curve. Once the concentrations were corrected for the endogenous level of PFOS in the control tissue, PFOS recoveries of the calibration curve points were within 100±14%. The recoveries of the extracted curve points quantitated against the solvent curve points are provided in the Supplemental Information Section 11.1.

A quadratic, 1/x or 1/x^2 weighted calibration curve was used to fit the data for PFOS and [$^{18}O_2$]PFOS. Internal standard quantitation was used. The data was not forced through zero during the fitting process. The accuracy of each curve point was verified by calculating the concentration using the area count ratio of the target analyte to the internal standard. Each solvent calibration standard used to generate the final calibration curve met the method calibration accuracy requirement of 100±25%, except the LOQ standard (100±30%). The coefficients of determination (r^2) were greater than 0.990 for all analytes. As required by ETS 8-45, a minimum of six calibration points were used to generate the final calibration curve.

For PFHS, PFOS, and PFOA, the reference standard used for calibration and lab control spikes was comprised predominantly of the linear isomer.

It is noted that the purchased control bluegill tissue matrix differs in composition from the study bluegill samples in that the purchased bluegill fillet homogenate does not contain the skin of the fillet, only the "meat".

*As fish matrix can vary greatly from species to species, the control matrix used to generate the extracted calibration curve and laboratory control QC samples is considered to be matrix-matched for the bluegill samples only. The laboratory control matrix should be considered as a "surrogate" control matrix for the other species investigated in this study.

4.2 Limit of Quantitation (LOQ)

The LOQ as defined in ETS 8-45 is the lowest non-zero calibration standard in the curve in which the area counts of the target analyte are at least twice those of the matrix blank(s). Since the solvent curve was used for quantitation, the aqueous method blanks were used to establish the LOQ so that the curve could be used to quantitate the endogenous levels of PFOS in the control matrix. The limit of quantitation for PFOS varied from extraction date and instrument batch. The PFOS LOQ ranged from 0.00251 ng/mL to 0.00995 ng/mL which corresponds to approximately 0.025 ng/g to 0.1 ng/g in fish tissue once the conversion is made.

4.3 System Suitability

Four replicate injections of the solvent calibration standard were analyzed at the beginning of the analytical sequence to demonstrate overall system suitability. Method criteria states that system suitability injections shall produce a RSD of less than 7% for the ratio of target analyte area counts to internal standard area counts and an RSD of less than 2% for the retention time. Method acceptance criteria were met for both PFOS and [$^{18}O_2$]PFOS.

4.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response from initial calibration curve was still in control. CCV injections produced recoveries of PFOS and [$^{18}O_2$]PFOS within 100%±25%, which met method criteria.

4.5 Blanks

Five types of blanks were prepared and analyzed with the samples: matrix blanks (bluegill filet control tissue: two with IS and surrogate, two without IS and surrogate), aqueous method blanks (two with IS and surrogate, two without IS and surrogate), acidified acetonitrile solvent blanks, acetonitrile blanks with internal standards and surrogates, and acetonitrile solvent blanks (unmodified). Each blank result was reviewed and used to evaluate method performance to determine the LOQ for each analyte. Surrogate recoveries of spiked blanks are provided in the Supplemental Information Section 11.2.

4.6 Lab Control Spikes (LCSs)

Triplicate lab control spikes of the purchased bluegill fillet control matrix at three different levels were prepared each extraction day to determine analytical method uncertainty (See Section 4.9). For PFHS, PFOS, and PFOA, the standard reference material used for the LCS spikes was the linear isomer, which was the same used for the calibration curves. Separate 3M electrochemical fluorination (ECF) spikes of branched PFOS and PFOA were prepared to evaluate any potential bias from quantitation against a linear standard. Lab control spikes were prepared to evaluate method accuracy and precision and were used to determine analytical method uncertainty (See Section 4.9). The [$^{18}O_2$]PFOS surrogate was spiked at a consistent 1 ng/g (0.1 ng/mL) for all LCS samples. For PFOS, the spiked concentration was corrected for the average PFOS concentration determined for the control matrix in the same extraction batch. Lab control spike recoveries are provided in the Supplemental Information (Section 11.3). For both PFOS and [$^{18}O_2$]PFOS, all replicates (N=48) except one, produced recoveries within method acceptance criteria 100%±25%.

4.7 Sample Concentration Calculation

The solvent calibration curve used for quantitation provided extract concentration in units of ng/mL. The following equation was used to convert the extract concentration to sample tissue concentration in units of ng/g.

$$\text{Tissue Concentration}\left(\frac{ng}{g}\right) = \frac{\text{Extract Concentration}\left(\frac{ng}{mL}\right) * \text{ExtractionVolume(mL)}}{\text{ExtractedMass (g)}}$$

Extraction Volume = 5 mL
Extracted Mass ~0.5 g

4.8 Lab Matrix Spikes (LMSs)

For each submitted sample, a separate laboratory matrix spike was prepared by extracting a separate aliquot of the fillet tissue that was spiked with the target analytes. For each LMS, all target analytes were spiked at a nominal concentration of 2 ng/g except PFOS, which was spiked at a total concentration of approximately 150 ng/g. The recovery of the LMS was calculated using the following equation.

$$\text{LMS \%Recovery} = \frac{\left(\text{Extract Conc.}\left(\frac{ng}{mL}\right) * \text{ExtractionVolume(mL)}\right) - \left(\text{Average Sample Conc.}\left(\frac{ng}{g}\right) * \text{LMS Sample Mass (g)}\right)}{\text{Spike Amount (ng)}} * 100\%$$

Although ETS 8-45.0 does not provide specific criteria for the lab matrix spike concentrations, the 3M Environmental Laboratory uses a general guideline that lab matrix spike concentration should be at least half the endogenous concentration before the spike is considered "appropriate" for the given matrix. For PFOS, the LMS was 76.0 ng (~150 ng/g tissue concentration) except for samples E09-0053-008 and -016 where the LMS was 250 ng (~500 ng/g tissue concentration). The [$^{18}O_2$]PFOS surrogate was spiked at 0.502 ng (~1 ng/g tissue concentration).

4.9 Analytical Uncertainty

ETS 8-45.0 states that the analytical method uncertainty will be determined by statistical evaluation of the individual analyte recovery in LCSs. Current project QC results, as well as historical values, are used in the evaluation until a maximum of fifty points (minimum of twenty, if possible) are included. For ETS 8-45.0, the historical LCSs are categorized by tissue type: whole-body or fillet unless there is strong evidence suggesting that a species-specific matrix effect is present and that results from these species should be considered separately. The standard deviation of the collective recovery results (in %) is calculated. The expanded uncertainty is then calculated by multiplying the standard deviation by a factor of two, which corresponds to the 95% confidence limit. Table 7 below provides the analytical uncertainty for PFOS and the [$^{18}O_2$]PFOS surrogate.

Table 7. Analytical Method Uncertainty

	PFOS	*[$^{18}O_2$]PFOS Surrogate*
Average % Recovery	105	108
Standard Deviation	7.6	7.1
2*Standard Deviation	15	14
N	50 (2 Rainbow Trout fillets, 48 Bluegill fillets)	48 (Bluegill fillet only)
Analytical Uncertainty	***±15%***	***±14%***

5. Data Summary and Discussion

Table 8 below provides the sample and lab matrix spike results for PFOS and the [$^{18}O_2$]PFOS surrogate quantitated using an unextracted, solvent curve. The extracted curve prepared in bluegill fillet control matrix was demonstrated to be equivalent to the solvent curve for both PFOS and the [$^{18}O_2$]PFOS surrogate over the approximate range of 0.25 ng/g to 25 ng/g. (See Supplemental Information Section 11.1 for additional information.) As most of the study samples were greater than 25 ng/g for PFOS, the solvent curve, which went up to 50 ng/mL or ~500 ng/g was used for quantitation. All samples produced PFOS LMS recoveries within method acceptance criteria of 100±30%. E09-0053-008 and -016 were re-prepared in a separate extraction batch with a LMS of 250 ng (~500 ng/g). The initial analyses for these two samples indicated that the original LMS concentration of ~150 ng/g was less than half the endogenous concentration and therefore was not considered an appropriate level for assessing sample quantitation accuracy. Results for these two samples from the initial analysis were not reported. Instances of the surrogate recovery exceeding 100±30% have been flagged in Table 8; however, ETS 8-45.0 specifies no acceptance criteria for surrogate recoveries as the intent of the surrogate is to evaluate systematic biases or interferences that may be present As the PFOS LMS recoveries were all within 100±30% and the overall number of surrogate recoveries exceeding 100±30% were few, all results were considered reportable and accurate to within the stated analytical method uncertainty of 100±15%.

The recoveries of the 3M ECF LCSs prepared with each extraction batch (N=12 for the study) ranged from 97.5% to 130% with a calculated average of 110% (8.4% RSD). The results of the ECF QC spikes demonstrated that there is no discernible bias when PFOS ECF isomers are quantitated against the predominantly linear reference standard. Therefore, the reported PFOS values are considered accurate to within the stated method uncertainty regardless of the degree of branched PFOS isomers present in the sample extracts.

The PFOS sample concentrations presented in Table 1 and Table 8 were reported from the total ion chromatogram (TIC) where the instrument software summed the three individual transitions (499>80,

499>99, 499>130). As the 499>80 transition has been well documented to produce PFOS matrix interferences in biological tissue studies[1], the three individual transitions were evaluated separately. to verify that the area ratios observed in the standards were conserved in the samples. For Batches 1, 2, and 3, the combined average area percentages of 499>80, 499>99, and 499>130 in the solvent calibration curve points were 76.5% (3.4% RSD), 21.1% (13% RSD), and 2.36% (42%RSD), respectively. The 499>99 and 499>130 transitions are significantly weaker in intensity when compared to the 499>80 transition. The higher variabilities observed for these two transitions in the solvent curve was largely due to very weak signals in the calibration standards less than 0.5 ng/mL in concentration. Calibration standards of 0.5 ng/mL and higher produced %RSD of approximately 8% for these two transitions. Figure 1 and Figure 2 display the average area percentages observed for each transition in the study samples. Here, the plotted average and %RSD is the statistical calculations for sample, sample duplicate, and associated LMS. For viewing simplicity, the %RSD for 499>130 was excluded from the graphs as it was often larger than the area average and produced error bars that extended into the negative y-axis region making the graph difficult to read. From the figures, it is clear that the overall transition ratios observed in the solvent standard are conserved in the extracted samples and QC.

Table 8. Sample and Lab Matrix Spike Results: PFOS and [$^{18}O_2$]PFOS surrogate.

			PFOS			[$^{18}O_2$]PFOS		
3M LIMS ID	*Sample Description*	*Mass (g)*	*Conc. (ng/mL)*	*(1) Conc. (ng/g)*	*%Rec*	*Conc. (ng/mL)*	*Conc. (ng/g)*	*%Rec*
E09-0053-001	MissR_pool2_WE_1	0.4799	2.75	28.7	NA	0.102	1.06	102
E09-0053-001 Dup		0.4884	2.77	28.4	NA	0.108	1.11	108
E09-0053-001 LMS		0.5032	19.9	198	112	0.126	1.25	126
		Average		*28.5*		*Average %Recovery*		*112*
		%RPD		*1.0*			*%RSD*	*11*
E09-0053-002	MissR_pool2_WE_2	0.5049	4.64	45.9	NA	0.107	1.06	107
E09-0053-002 Dup		0.5212	4.52	43.4	NA	0.104	1.00	104
E09-0053-002 LMS		0.5008	18.9	189	95.0	0.122	1.22	122
		Average		*44.7*		*Average %Recovery*		*111*
		%RPD		*5.8*			*%RSD*	*8.7*
E09-0053-003	MissR_pool2_WE_3	0.5202	8.77	84.3	NA	0.108	1.04	108
E09-0053-003 Dup		0.5056	8.28	81.9	NA	0.112	1.11	112
E09-0053-003 LMS		0.5159	24.4	236	104	0.133	1.29	(2)133
		Average		*83.1*		*Average %Recovery*		*117*
		%RPD		*2.9*			*%RSD*	*11*
E09-0053-004	MissR_pool2_WE_4	0.4822	4.92	51.0	NA	0.114	1.18	114
E09-0053-004 Dup		0.4974	4.28	43.0	NA	0.105	1.06	105
E09-0053-004 LMS		0.5163	20.5	199	103	0.115	1.11	115
		Average		*47.0*		*Average %Recovery*		*111*
		%RPD		*17*			*%RSD*	*4.9*
E09-0053-005	MissR_pool2_SAG_1	0.4871	4.00	41.1	NA	0.101	1.04	101
E09-0053-005 Dup		0.4954	3.82	38.6	NA	0.106	1.07	106
E09-0053-005 LMS		0.4781	18.4	192	96.1	0.107	1.12	107
		Average		*39.8*		*Average %Recovery*		*104*
		%RPD		*6.3*			*%RSD*	*3.1*
E09-0053-006	MissR_pool2_SAG_2	0.4808	4.20	43.7	NA	0.0999	1.04	100
E09-0053-006 Dup		0.5129	4.45	43.4	NA	0.114	1.11	114
E09-0053-006 LMS		0.4891	18.3	187	92.4	0.111	1.13	111
		Average		*43.5*		*Average %Recovery*		*108*
		%RPD		*0.68*			*%RSD*	*6.9*
E09-0053-007	MissR_pool2_SMB_1	0.5287	6.08	57.5	NA	0.105	0.99	105
E09-0053-007 Dup		0.5257	5.30	50.4	NA	0.109	1.04	109
E09-0053-007 LMS		0.4943	22.9	232	116	0.116	1.17	116
		Average		*54.0*		*Average %Recovery*		*110*
		%RPD		*13*			*%RSD*	*5.1*
E09-0053-008	MissR_pool2_SMB_2	0.4785	(3)36.3	(3)379	NA	(3)0.130	(3)1.36	(2)130
E09-0053-008 Dup		0.5163	(3)39.7	(3)384	NA	(3)0.121	(3)1.17	121
E09-0053-008 LMS		0.5012	(3)86.6	(3)854	(3)96.6	(3)0.143	(3)1.43	(2)142
		Average		*(3)382*		*Average %Recovery*		*(2)131*
		%RPD		*1.4*			*%RSD*	*8.4*

Table 8 Continued.

			PFOS			$[^{18}O_2]$PFOS		
3M LIMS ID	Sample Description	Mass (g)	Conc. (ng/mL)	[1]Conc. (ng/g)	%Rec	Conc. (ng/mL)	Conc. (ng/g)	%Rec
E09-0053-009	MissR_pool2_SMB_3	0.4963	11.0	111	NA	0.106	1.07	106
E09-0053-009 Dup		0.4975	12.2	123	NA	0.116	1.17	116
E09-0053-009 LMS		0.4784	26.8	280	103	0.116	1.21	116
		Average		***117***		*Average %Recovery*		***112***
		%RPD		***10***			*%RSD*	***5.1***
E09-0053-010	MissR_pool2_SMB_4	0.5107	13.4	131	NA	0.119	1.17	119
E09-0053-010 Dup		0.4952	12.1	122	NA	0.102	1.03	102
E09-0053-010 LMS		0.4839	26.6	275	94.4	0.124	1.28	124
		Average		***127***		*Average %Recovery*		***115***
		%RPD		***7.1***			*%RSD*	***10***
E09-0053-011	MissR_pool2_SMB_5	0.4926	13.9	141	NA	0.114	1.16	114
E09-0053-011 Dup		0.4861	14.6	150	NA	0.120	1.23	120
E09-0053-011 LMS		0.5148	27.6	268	83.0	0.122	1.18	122
		Average		***146***		*Average %Recovery*		***118***
		%RPD		***6.2***			*%RSD*	***3.5***
E09-0053-012	MissR_pool2_BKS_1	0.4749	5.27	55.5	NA	0.0995	1.05	99.1
E09-0053-012 Dup		0.5146	5.61	54.5	NA	0.105	1.02	105
E09-0053-012 LMS		0.5028	22.5	224	112	0.116	1.15	116
		Average		***55.0***		*Average %Recovery*		***106***
		%RPD		***1.8***			*%RSD*	***7.9***
E09-0053-013	MissR_pool2_BGS_1	0.5058	15.7	155	NA	0.110	1.09	110
E09-0053-013 Dup		0.4887	16.4	168	NA	0.105	1.07	105
E09-0053-013 LMS		0.5147	35.3	343	123	0.108	1.05	108
		Average		***161***		*Average %Recovery*		***107***
		%RPD		***7.8***			*%RSD*	***2.3***
E09-0053-014	MissR_pool2_BGS_2	0.5237	11.5	110	NA	0.123	1.17	123
E09-0053-014 Dup		0.4809	9.61	100	NA	0.109	1.13	109
E09-0053-014 LMS		0.5201	26.0	250	99.3	0.113	1.09	113
		Average		***105***		*Average %Recovery*		***115***
		%RPD		***9.4***			*%RSD*	***6.3***
E09-0053-015	MissR_pool2_BGS_3	0.5118	19.2	188	NA	0.108	1.06	108
E09-0053-015 Dup		0.5022	16.8	167	NA	0.112	1.12	112
E09-0053-015 LMS		0.5098	37.4	367	127	0.117	1.15	117
		Average		***177***		*Average %Recovery*		***112***
		%RPD		***11***			*%RSD*	***4.0***
E09-0053-016	MissR_pool2_BGS_4	0.5024	[3]33.3	[3]331	NA	[3]0.122	[3]1.21	122
E09-0053-016 Dup		0.5096	[3]34.7	[3]340	NA	[3]0.118	[3]1.16	118
E09-0053-016 LMS		0.5124	[3]83.7	[3]817	[3]98.5	[3]0.163	[3]1.59	[2]162
		Average		***[2]336***		*Average %Recovery*		***[2]134***
		%RPD		***2.7***			*%RSD*	***19***

Table 8 Continued.

			PFOS			$[^{13}O_2]$PFOS		
3M LIMS ID	*Sample Description*	*Mass (g)*	*Conc. (ng/mL)*	*(1) Conc. (ng/g)*	*%Rec*	*Conc. (ng/mL)*	*Conc. (ng/g)*	*%Rec*
E09-0053-017	MissR_pool2_BGS_5	0.5005	12.3	123	NA	0.116	1.16	116
E09-0053-017 Dup		0.5146	12.4	120	NA	0.104	1.01	104
E09-0053-017 LMS		0.5052	28.0	277	103	0.118	1.17	118
		Average		***122***		*Average %Recovery*		***112***
		%RPD		***2.0***			*%RSD*	***6.7***
E09-0053-018	MissR_pool3_WE_1	0.5021	9.78	97.4	NA	0.102	1.02	102
E09-0053-018 Dup		0.5091	9.72	95.5	NA	0.102	1.00	102
E09-0053-018 LMS		0.5168	27.1	262	113	0.117	1.13	117
		Average		***96.4***		*Average %Recovery*		***107***
		%RPD		***2.0***			*%RSD*	***8.1***
E09-0053-019	MissR_pool3_WE_2	0.5233	8.20	78.3	NA	0.115	1.10	115
E09-0053-019 Dup		0.5150	8.10	78.6	NA	0.105	1.02	105
E09-0053-019 LMS		0.4892	24.9	254	113	0.119	1.22	119
		Average		***78.5***		*Average %Recovery*		***113***
		%RPD		***0.37***			*%RSD*	***6.4***
E09-0053-020	MissR_pool3_WE_3	0.5075	9.89	97.4	NA	0.130	1.28	130
E09-0053-020 Dup		0.5040	8.30	82.3	NA	0.106	1.05	106
E09-0053-020 LMS		0.5172	28.7	277	128	0.122	1.18	122
		Average		***89.9***		*Average %Recovery*		***119***
		%RPD		***17***			*%RSD*	***10***
E09-0053-021	MissR_pool3_WE_4	0.5093	5.03	49.4	NA	0.110	1.08	110
E09-0053-021 Dup		0.5144	5.38	52.3	NA	0.100	0.972	100
E09-0053-021 LMS		0.5168	21.0	203	104	0.105	1.02	105
		Average		***50.8***		*Average %Recovery*		***105***
		%RPD		***5.7***			*%RSD*	***4.8***
E09-0053-022	MissR_pool3_WE_5	0.5152	6.62	64.2	NA	0.100	0.970	100
E09-0053-022 Dup		0.4847	6.60	68.1	NA	0.105	1.08	105
E09-0053-022 LMS		0.4779	23.7	248	114	0.123	1.29	123
		Average		***66.2***		*Average %Recovery*		***109***
		%RPD		***5.8***			*%RSD*	***11***
E09-0053-023	MissR_pool3_NOP_1	0.4741	4.72	49.8	NA	0.108	1.14	108
E09-0053-023 Dup		0.5087	5.21	51.2	NA	0.108	1.06	108
E09-0053-023 LMS		0.5024	21.7	216	109	0.132	1.31	(2)132
		Average		***50.5***		*Average %Recovery*		***116***
		%RPD		***2.8***			*%RSD*	***12***
E09-0053-024	MissR_pool3_SMB_1	0.4791	2.83	29.5	NA	0.110	1.15	110
E09-0053-024 Dup		0.5276	3.15	29.9	NA	0.102	0.967	102
E09-0053-024 LMS		0.5115	18.2	178	100	0.114	1.11	114
		Average		***29.7***		*Average %Recovery*		***108***
		%RPD		***1.1***			*%RSD*	***5.6***

Table 8 Continued.

			PFOS			$[^{18}O_2]$PFOS		
3M LIMS ID	*Sample Description*	*Mass (g)*	*Conc. (ng/mL)*	*(1) Conc. (ng/g)*	*%Rec*	*Conc. (ng/mL)*	*Conc. (ng/g)*	*%Rec*
E09-0053-025	MissR_pool3_SMB_2	0.4868	10.0	103	NA	0.105	1.08	105
E09-0053-025 Dup		0.5146	11.1	108	NA	0.125	1.21	125
E09-0053-025 LMS		0.5212	25.9	248	98.2	0.122	1.17	122
		Average		*105*		*Average %Recovery*		*117*
		%RPD		*4.9*			*%RSD*	*9.2*
E09-0053-026	MissR_pool3_SMB_3	0.5004	4.88	48.8	NA	0.108	1.08	108
E09-0053-026 Dup		0.4942	4.77	48.3	NA	0.105	1.06	105
E09-0053-026 LMS		0.4928	20.0	203	100	0.12	1.22	120
		Average		*48.5*		*Average %Recovery*		*111*
		%RPD		*1.0*			*%RSD*	*7.2*
E09-0053-027	MissR_pool3_SMB_4	0.4802	6.05	63.0	NA	0.112	1.17	112
E09-0053-027 Dup		0.4978	6.66	66.9	NA	0.115	1.16	115
E09-0053-027 LMS		0.5202	20.2	194	88.5	0.118	1.13	118
		Average		*64.9*		*Average %Recovery*		*115*
		%RPD		*6.0*			*%RSD*	*2.6*
E09-0053-028	MissR_pool3_SMB_5	0.5170	5.69	55.0	NA	0.108	1.04	108
E09-0053-028 Dup		0.5016	5.35	53.3	NA	0.114	1.14	114
E09-0053-028 LMS		0.5015	20.6	205	100	0.12	1.20	120
		Average		*54.2*		*Average %Recovery*		*114*
		%RPD		*3.1*			*%RSD*	*5.3*
E09-0053-029	MissR_pool3_BGS_1	0.4922	7.55	76.7	NA	0.113	1.15	113
E09-0053-029 Dup		0.5236	7.62	72.8	NA	0.0988	0.943	98.4
E09-0053-029 LMS		0.5289	21.3	201	88.2	0.112	1.06	112
		Average		*74.7*		*Average %Recovery*		*108*
		%RPD		*5.3*			*%RSD*	*7.3*
E09-0053-030	MissR_pool3_BGS_2	0.5234	16.8	160	NA	0.104	0.994	104
E09-0053-030 Dup		0.5010	16.5	165	NA	0.115	1.15	115
E09-0053-030 LMS		0.4780	28.3	296	84.0	0.122	1.28	122
		Average		*163*		*Average %Recovery*		*113*
		%RPD		*2.6*			*%RSD*	*8.0*
E09-0053-031	MissR_pool3_BGS_3	0.4868	13.4	138	NA	0.119	1.22	119
E09-0053-031 Dup		0.5055	12.9	128	NA	0.105	1.04	105
E09-0053-031 LMS		0.4815	25.8	268	85.8	0.122	1.27	122
		Average		*133*		*Average %Recovery*		*115*
		%RPD		*7.6*			*%RSD*	*7.9*
E09-0053-032	MissR_pool3_BGS_4	0.5164	23.0	223	NA	0.105	1.02	105
E09-0053-032 Dup		0.4841	21.2	219	NA	0.115	1.19	115
E09-0053-032 LMS		0.5015	34.5	344	81.3	0.124	1.24	124
		Average		*221*		*Average %Recovery*		*114*
		%RPD		*1.7*			*%RSD*	*8.3*

Table 8 Continued.

			PFOS			$[^{18}O_2]$PFOS		
3M LIMS ID	*Sample Description*	*Mass (g)*	*Conc. (ng/mL)*	*(1) Conc. (ng/g)*	*%Rec*	*Conc. (ng/mL)*	*Conc. (ng/g)*	*%Rec*
E09-0053-033	MissR_pool3_BGS_5	0.5092	12.8	126	NA	0.117	1.15	117
E09-0053-033 Dup		0.5071	13.5	133	NA	0.121	1.19	121
E09-0053-033 LMS		0.4998	26.0	260	86.0	0.120	1.20	120
		Average		*129*		*Average %Recovery*		*119*
		%RPD		*5.7*			*%RSD*	*1.7*

(1) Unless otherwise specified, the recovery of associated laboratory matrix spike (LMS) was within 100±30% which meets ETS 8-45.0 method acceptance criteria. Sample results are considered accurate to within the overall analytical method uncertainty 100±15% (PFOS). See Section 4.9 *Determination of Analytical Method Uncertainty* for more information. Sample results are reported to three significant figures and %RPD to two significant figures. Additional significant figures were used to calculate the average, %RPD, and %recovery. Values in the raw data may vary slightly due to rounding.

(2) Surrogate recovery exceeded 100±30%

(3) Reported concentrations for these samples are from a 1:10 post-extraction dilution of the final extract. These two samples were re-prepared and analyzed in Batch 4 with a ~500 ng/g LMS. The results from the original analysis were not reported as the LMS concentration (~150 ng/g) was less than half the endogenous sample concentration and consequently, the accuracy of the sample concentrations could not be assessed.

Figure 1. Area Percentages of Individual PFOS Transitions:

Samples -001 through -018.

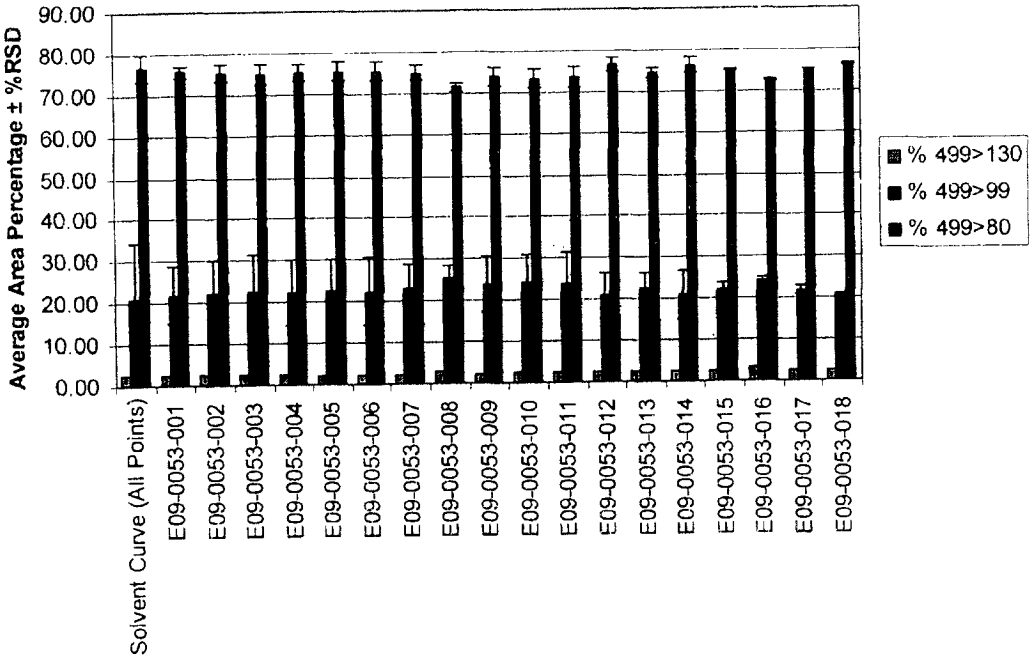
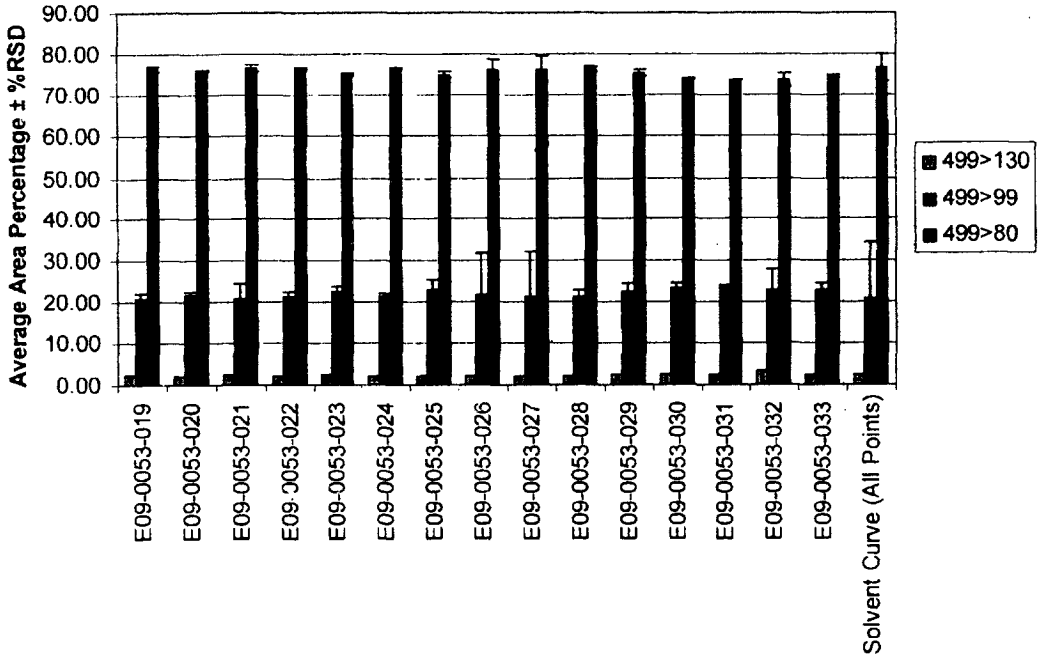


Figure 2. Area Percentages of Individual PFOS Transitions:

Samples -019 through -033.



6 Conclusions

The PFOS sample results presented in Table 1 and Table 8 are considered accurate within the overall method uncertainty of 100±15%. When compared to the solvent standards used for quanitation, area count ratios of the individual PFOS transitions (499>80, 499>99, and 499>130) were conserved in extracted samples. The accuracy (% recovery) and precision (%RSD) of 3M ECF (branched) PFOS laboratory control spikes quantitated against the predominantly linear reference standard demonstrated that the isomers produce no discernable quantitative bias.

Results for remaining target analytes will be in a phase II report.

7 References

[1] J. P. Benskin, M. Bataineh, J.W. Martin, Simultaneous Characterization of Perfluoroalkyl Carboxylate, Sulfonate, and Sulfonamide Isomers by Liquid Chromatography – Tandem Mass Spectrometry, Anal. Chem. 79 (2007) 6455-6464.

8 Data/Sample Retention

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

9 Analytical Personnel

9.1 3M Environmental Laboratory

Michelle D. Malinsky, Ph.D Research Specialist

9.2 Pace Lab Ops

Jonathan Steege Lab Analyst II

10 Signatures

Michelle D. Malinsky, Ph.D, 3M Research Specialist, Principal Analytical Investigator — April 1, 2009 (Date)

William K. Reagen, Ph.D., Environmental Laboratory Management — 1 April 2009 (Date)

Cliffton B. Jacoby, Ph.D., 3M Technical Reviewer — 1 April 2009 (Date)

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

QAI Representative — 4-1-09 (Date)

11 Supplemental Information

11.1 Solvent/Matrix-Matched Extracted Calibration Curve Equivalency

Extracted Matrix Curve, Prep Batch 1, Analysis Date: 02/20/2009			PFOS			$[^{18}O_2]$PFOS		
Sample ID	Sample Decription	Extracted Mass (g)	(1) Corrected Standard Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Standard Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090220 BG fillet Extracted Curve Point 1	0.25 ng/g curve point	0.4903	0.0829	0.0720	86.9	0.0253	0.0195	77 1
090220 BG fillet Extracted Curve Point 2	0.35 ng/g curve point	0.4918	0.0929	0.0926	100	0.0353	0.0365	103
090220 BG fillet Extracted Curve Point 3	0.5 ng/g curve point	0.5022	0.111	0.106	95.6	0.0522	0.0551	106
090220 BG fillet Extracted Curve Point 4	1 ng/g curve point	0.5201	0.161	0.154	95.8	0.100	0.107	107
090220 BG fillet Extracted Curve Point 5	2.5 ng/g curve point	0.5024	0.310	0.319	103	0.253	0.256	101
090220 BG fillet Extracted Curve Point 6	5 ng/g curve point	0.5039	0.576	0.605	105	0.522	0.591	113
090220 BG fillet Extracted Curve Point 7	10 ng/g curve point	0.5088	1.05	1.16	110	1.00	1.18	118
090220 BG fillet Extracted Curve Point 8	15 ng/g curve point	0.4853	1.57	1.60	102	1.53	1.69	111
090220 BG fillet Extracted Curve Point 9	25 ng/g curve point	0.4873	2.57	2.71	106	2.53	2.53	99.9

(1) Average endogenous concentration as determined by quantitation of the matrix blanks using solvent curve quantitation = 0.589 ng/g (RPD= 0.57%).

Extracted Matrix Curve, Prep Batch 2, Analysis Date: 02/23/2009			PFOS			$[^{18}O_2]$PFOS		
Sample ID	Sample Decription	Extracted Mass (g)	(1) Corrected Standard Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Standard Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090223 BG fillet Extracted Curve Point 1	0.25 ng/g curve point	0.4791	0.0744	0.0830	112	0.0253	0.0238	94.0
090223 BG fillet Extracted Curve Point 2	0.35 ng/g curve point	0.4878	0.0852	0.0894	105	0.0353	0.0359	102
090223 BG fillet Extracted Curve Point 3	0.5 ng/g curve point	0.5148	0.105	0.105	100	0.0522	0.0487	93.2
090223 BG fillet Extracted Curve Point 4	1 ng/g curve point	0.5036	0.151	0.152	100	0.100	0.0952	95.2
090223 BG fillet Extracted Curve Point 5	2.5 ng/g curve point	0.5135	0.304	0.297	97.7	0.253	0.237	93.6
090223 BG fillet Extracted Curve Point 6	5 ng/g curve point	0.4782	0.566	0.622	110	0.522	0.568	109
090223 BG fillet Extracted Curve Point 7	10 ng/g curve point	0.5116	1.05	1.15	110	1.00	1.00	100
090223 BG fillet Extracted Curve Point 8	15 ng/g curve point	0.4986	1.56	1.75	112	1.53	1 59	104
090223 BG fillet Extracted Curve Point 9	25 ng/g curve point	0.5233	2.56	2.79	109	2.53	2.37	93.6

(1) Average endogenous concentration as determined by quantitation of the matrix blanks by solvent curve quantitation = 0.515 ng/g (RPD = 1.7%).

Extracted Matrix Curve, Prep Batch 3, Analysis Date: 02/25/2009			PFOS			$[^{13}O_2]$PFOS		
Sample ID	Sample Decription	Extracted Mass (g)	(1)Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090225 BG fillet Extracted Curve Point 1	0.25 ng/g curve point	0.5253	0.0847	0.0817	96.5	0.0253	0.0216	85.2
090225 BG fillet Extracted Curve Point 2	0.35 ng/g curve point	0.5141	0.0933	0.102	109	0.0353	0.0348	98.6
090225 BG fillet Extracted Curve Point 3	0.5 ng/g curve point	0.4962	0.108	0.113	105	0.0522	0.0557	107
090225 BG fillet Extracted Curve Point 4	1 ng/g curve point	0.5119	0.158	0.159	101	0.100	0.106	106
090225 BG fillet Extracted Curve Point 5	2.5 ng/g curve point	0.5159	0.309	0.332	107	0.253	0.267	105
090225 BG fillet Extracted Curve Point 6	5 ng/g curve point	0.5231	0.576	0.608	106	0.522	0.583	112
090225 BG fillet Extracted Curve Point 7	10 ng/g curve point	0.5265	1.05	1.14	108	1.00	1.00	100
090225 BG fillet Extracted Curve Point 8	15 ng/g curve point	0.4783	1.56	1.78	114	1.53	1.60	105
090225 BG fillet Extracted Curve Point 9	25 ng/g curve point	0.4785	2.56	2.90	113	2.53	2.41	95.1

(1) Average endogenous concentration as determined by quantitation of the matrix blanks by solvent curve quantitation = 0.567 ng/g (RPD = 2.8%).

Extracted Matrix Curve, Prep Batch 4, Analysis Date: 03/06/2009			PFOS			$[^{13}O_2]$PFOS		
Sample ID	Sample Decription	Extracted Mass (g)	(1)Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090306 BG fillet Extracted Curve Point 1	0.25 ng/g curve point	0.5067	0.0807	0.0859	106	0.0253	0.0223	88.3
090306 BG fillet Extracted Curve Point 2	0.35 ng/g curve point	0.4898	0.0887	0.0905	102	0.0353	0.0326	92.3
090306 BG fillet Extracted Curve Point 3	0.5 ng/g curve point	0.4852	0.105	0.111	106	0.0522	0.0587	112
090306 BG fillet Extracted Curve Point 4	1 ng/g curve point	0.5124	0.156	0.152	97.6	0.100	0.100	100
090306 BG fillet Extracted Curve Point 5	2.5 ng/g curve point	0.4998	0.306	0.298	97.4	0.253	0.257	102
090306 BG fillet Extracted Curve Point 6	5 ng/g curve point	0.4934	0.571	0.66	116	0.522	0.575	110
090306 BG fillet Extracted Curve Point 7	10 ng/g curve point	0.4948	1.05	1.13	108	1.00	1.02	102
090306 BG fillet Extracted Curve Point 8	15 ng/g curve point	0.5037	1.57	1.74	111	1.53	1.64	107
090306 BG fillet Extracted Curve Point 9	25 ng/g curve point	0.5182	2.57	2.87	112	2.53	2.67	106

(1) Average endogenous concentration as determined by quantitation of the matrix blanks by solvent curve quantitation = 0.548 ng/g (RPD = 5.0%).

11.2 Surrogate Recoveries in Spiked Blanks.

Table 9. Surrogate Recoveries in Spiked Blanks.

Prep Batch 1, Analysis Date: 02/20/2009		*[$^{18}O_2$]PFOS*			
Sample ID	*Sample Description*	*Area Counts*	*Spike Conc. (ng/mL)*	*Calc. Conc. (ng/mL)*	*%Rec*
Matrix Blank-1 (with IS/surr) 090220	Method Blank (BG fillet)	66130	0.100	0.109	109
Matrix Blank-2 (with IS/surr) 090220	Method Blank (BG fillet)	62887	0.100	0.107	107
Aqueous Blank-1 (with IS/surr) 090220	Aqueous Blank	69115	0.100	0.116	116
Aqueous Blank-2 (with IS/surr) 090220	Aqueous Blank	64501	0.100	0.0991	99.1
ACN Blank with IS/surr	[1]08 007-62	158329	NA	N/A	NA
ACN Blank with IS/surr	[1]08 007-62	147907	NA	NA	NA
Prep Batch 2, Analysis Date: 02/23/2009		*[$^{18}O_2$]PFOS*			
Sample ID	Sample Description	*Area Counts*	*Spike Conc. (ng/mL)*	*Calc. Conc. (ng/mL)*	*%Rec*
Matrix Blank-1 (with IS/surr) 090223	Method Blank (BG fillet)	68808	0.100	0.100	100
Matrix Blank-2 (with IS/surr) 090223	Method Blank (BG fillet)	69717	0.100	0.103	103
Aqueous Blank-1 (with IS/surr) 090223	Aqueous Blank	67533	0.100	0.116	116
Aqueous Blank-2 (with IS/surr) 090223	Aqueous Blank	56872	0.100	0.0908	90.8
ACN Blank with IS/surr	[1]08 007-62	135129	NA	N/A	NA
ACN Blank with IS/surr	[1]08 007-62	135207	NA	NA	NA
Prep Batch 3, Analysis Date: 02/25/2009		*[$^{18}O_2$]PFOS*			
Sample ID	*Sample Description*	*Area Counts*	*Spike Conc. (ng/mL)*	*Calc. Conc. (ng/mL)*	*%Rec*
Matrix Blank-1 (with IS/surr) 090225	Method Blank (BG fillet)	71175	0.100	0.113	113
Matrix Blank-2 (with IS/surr) 090225	Method Blank (BG fillet)	73840	0.100	0.109	109
Aqueous Blank-1 (with IS/surr) 090225	Aqueous Blank	65016	0.100	0.107	107
Aqueous Blank-2 (with IS/surr) 090225	Aqueous Blank	64033	0.100	0.102	102
ACN Blank with IS/surr	[1]08 007-62	153699	NA	NA	NA
ACN Blank with IS/surr	[1]08 007-62	151127	NA	NA	NA

Table 9 Continued.

Prep Batch 4, Analysis Date: 03/06/2009		*$[^{18}O_2]$PFOS*			
Sample ID	*Sample Description*	*Area Counts*	*Spike Conc. (ng/mL)*	*Calc. Conc. (ng/mL)*	*%Rec*
Matrix Blank-1 (with IS/surr) 090306	Method Blank (BG fillet)	93301	0.100	0.101	101
Matrix Blank-2 (with IS/surr) 090306	Method Blank (BG fillet)	93408	0.100	0.108	108
Aqueous Blank-1 (with IS/surr) 090306	Aqueous Blank	90119	0.100	0.105	105
Aqueous Blank-2 (with IS/surr) 090306	Aqueous Blank	87716	0.100	0.102	102
ACN Blank with IS/surr	(1)08 007-62	198454	NA	NA	NA
ACN Blank with IS/surr	(1)08 007-62	190408	NA	NA	NA

(1) Standard 08-007-62 was prepared using the same acetonitrile used for extraction procedure; however, the IS/surrogate was misprepared. Essentially, the surrogate was spiked twice, and the IS was not spiked. Therefore, no concentrations and recoveries could be calculated.

11.3 Lab Control Spike Results.

Table 10. LCS Results Prep Batch 1

Prep Batch 1, Analysis Date: 02/20/2009			*PFOS*			*$[^{18}O_2]$PFOS*		
Sample ID	*Sample Decription*	*Extracted Mass (g)*	*(1) Corrected Spike Conc. (ng/mL)*	*Calc. Conc. (ng/mL)*	*% Recovery*	*Spike Conc. (ng/mL)*	*Calc. Conc. (ng/mL)*	*% Recovery*
090220 LCS Low 1	0.5 ng/g LCS	0.4851	0.109	0.108	99.2	0.100	0.114	114
090220 LCS Low 2	0.5 ng/g LCS	0.4893	0.109	0.105	96.0	0.100	0.108	108
090220 LCS Low 3	0.5 ng/g LCS	0.4981	0.110	0.106	96.0	0.100	0.106	106
090220 LCS Mid 1	1.5 ng/g LCS	0.4791	0.207	0.200	96.4	0.100	0.105	105
090220 LCS Mid 2	1.5 ng/g LCS	0.5101	0.211	0.220	104	0.100	0.104	104
090220 LCS Mid 3	1.5 ng/g LCS	0.5028	0.210	0.205	97.5	0.100	0.108	108
090220 LCS High 1	8 ng/g LCS	0.5202	0.857	0.831	96.9	0.100	0.103	103
090220 LCS High 2	8 ng/g LCS	0.4921	0.854	0.832	97.4	0.100	0.0906	90.6
090220 LCS High 3	8 ng/g LCS	0.4811	0.853	0.940	110	0.100	0.116	116
ECF LCS High 1	5 ng/g ECF LCS	0.4748	0.554	0.556	100	0.100	0.101	101
ECF LCS High 2	5 ng/g ECF LCS	0.5239	0.560	0.546	97.5	0.100	0.0974	97.4
ECF LCS High 3	5 ng/g ECF LCS	0.5132	0.558	0.728	130	0.100	0.116	116
Average ± %RSD			*102%±10%*			*106%±7.1%*		

(1) Spiked sample concentration corrected for the average endogenous concentration as determined by quantitation of the batch's matrix blanks using solvent curve quantitation. Endogenous PFOS = 0.589 ng/g (RPD= 0.57%).

Table 11. LCS Results Prep Batch 2

Prep Batch 2, Analysis Date: 02/23/2009			PFOS			$[^{18}O_2]$PFOS		
Sample ID	Sample Decription	Extracted Mass (g)	(1) Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090223 LCS Low 1	0.5 ng/g LCS	0.5299	0.105	0.106	101	0.100	0.0948	94.8
090223 LCS Low 2	0.5 ng/g LCS	0.5109	0.103	0.116	112	0.100	0.113	113
090223 LCS Low 3	0.5 ng/g LCS	0.5079	0.103	0.103	99.9	0.100	0.102	102
090223 LCS Mid 1	1.5 ng/g LCS	0.4975	0.201	0.212	105	0.100	0.113	113
090223 LCS Mid 2	1.5 ng/g LCS	0.5199	0.204	0.217	107	0.100	0.111	111
090223 LCS Mid 3	1.5 ng/g LCS	0.5044	0.202	0.213	105	0.100	0.108	108
090223 LCS High 1	8 ng/g LCS	0.4844	0.845	0.806	95.4	0.100	0.0953	95.3
090223 LCS High 2	8 ng/g LCS	0.5215	0.849	0.833	98.1	0.100	0.103	103
090223 LCS High 3	8 ng/g LCS	0.5188	0.848	0.895	105	0.100	0.0986	98.6
ECF LCS High 1	5 ng/g ECF LCS	0.5019	0.549	0.558	102	0.100	0.105	105
ECF LCS High 2	5 ng/g ECF LCS	0.5017	0.549	0.553	101	0.100	0.0985	98.5
ECF LCS High 3	5 ng/g ECF LCS	0.5009	0.549	0.635	116	0.100	0.110	110
Average ± %RSD			104%±5.6%			104%±6.4%		

(1) Spiked sample concentration corrected for the average endogenous concentration as determined by quantitation of the batch's matrix blanks using solvent curve quantitation. Endogenous PFOS = 0.515 ng/g (RPD= 1.7%).

Table 12. LCS Results Prep Batch 3

Prep Batch 3, Analysis Date: 02/25/2009			*PFOS*			*$[^{13}O_2]$PFOS*		
Sample ID	*Sample Decription*	*Extracted Mass (g)*	*(1) Corrected Spike Conc. (ng/mL)*	*Calc. Conc. (ng/mL)*	*% Recovery*	*Spike Conc. (ng/mL)*	*Calc. Conc. (ng/mL)*	*% Recovery*
090225 LCS Low 1	0.5 ng/g LCS	0.5271	0.111	0.114	102	0.100	0.112	112
090225 LCS Low 2	0.5 ng/g LCS	0.4908	0.107	0.113	105	0.100	0.106	106
090225 LCS Low 3	0.5 ng/g LCS	0.4871	0.107	0.105	98.2	0.100	0.110	110
090225 LCS Mid 1	1.5 ng/g LCS	0.4926	0.207	0.238	115	0.100	0.134	134
090225 LCS Mid 2	1.5 ng/g LCS	0.5105	0.209	0.251	120	0.100	0.115	115
090225 LCS Mid 3	1.5 ng/g LCS	0.4982	0.207	0.210	101	0.100	0.111	111
090225 LCS High 1	8 ng/g LCS	0.4814	0.851	0.842	99.0	0.100	0.100	100
090225 LCS High 2	8 ng/g LCS	0.5146	0.854	0.928	109	0.100	0.114	114
090225 LCS High 3	8 ng/g LCS	0.5257	0.856	0.871	102	0.100	0.105	105
ECF LCS High 1	5 ng/g ECF LCS	0.4944	0.554	0.635	115	0.100	0.114	114
ECF LCS High 2	5 ng/g ECF LCS	0.5284	0.558	0.601	108	0.100	0.104	104
ECF LCS High 3	5 ng/g ECF LCS	0.5157	0.556	0.599	108	0.100	0.112	112
Average ± %RSD			*107%±6.5%*			*111%±7.6%*		

(1) Spiked sample concentration corrected for the average endogenous concentration as determined by quantitation of the batch's matrix blanks using solvent curve quantitation. Endogenous PFOS = 0.567 ng/g (RPD= 2.8%).

Table 13. LCS Results Prep Batch 4.

Prep Batch 4, Analysis Date: 03/06/2009			PFOS			$[^{18}O_2]$PFOS		
Sample ID	Sample Decription	Extracted Mass (g)	(1) Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090306 LCS Low 1	0.5 ng/g LCS	0.5095	0.108	0.110	102	0.100	0.106	106
090306 LCS Low 2	0.5 ng/g LCS	0.4898	0.105	0.105	100	0.100	0.109	109
090306 LCS Low 3	0.5 ng/g LCS	0.5065	0.107	0.104	97.0	0.100	0.106	106
090306 LCS Mid 1	1.5 ng/g LCS	0.5000	0.206	0.217	105	0.100	0.110	110
090306 LCS Mid 2	1.5 ng/g LCS	0.5211	0.208	0.223	107	0.100	0.109	109
090306 LCS Mid 3	1.5 ng/g LCS	0.5079	0.207	0.205	99.2	0.100	0.108	108
090306 LCS High 1	8 ng/g LCS	0.5028	0.851	0.910	107	0.100	0.113	113
090306 LCS High 2	8 ng/g LCS	0.4853	0.849	0.956	113	0.100	0.111	114
090306 LCS High 3	8 ng/g LCS	0.5079	0.852	0.943	111	0.100	0.104	104
ECF LCS High 1	5 ng/g ECF LCS	0.5028	0.553	0.636	115	0.100	0.113	113
ECF LCS High 2	5 ng/g ECF LCS	0.5189	0.555	0.636	115	0.100	0.109	109
ECF LCS High 3	5 ng/g ECF LCS	0.5299	0.556	0.627	113	0.100	0.111	111
Average ± %RSD			107%±5.9%			109%±2.5%		

(1) Spiked sample concentration corrected for the average endogenous concentration as determined by quantitation of the batch's matrix blanks using solvent curve quantitation. Endogenous PFOS = 0.548 ng/g (RPD= 5.0%).