



February 11, 2010

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8EHQ-0598-373
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CERTIFIED MAIL

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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: Sulfonate-based and Carboxylic-based
Fluorochemicals, Docket 8EHQ-0598-373 - Results from 2009 Analyses of Fish
from the Mississippi River

Dear Sir or Madam:

3M is submitting this notice to supplement previous submissions on sulfonyl and carboxylic based fluorochemicals (FCs), and more specifically our June 20, 2006 and November 9, 2009 submittals concerning fish data generated by the Minnesota Pollution Control Agency (MPCA). The November 9, 2009 submittal included an Excel file, provided to 3M by the MPCA, entitled "PFCs MissPool2 Fish all years.xls" and 3M Environmental Laboratory Report No. E09-0372, Fluorochemical Analysis of Fish Fillet Homogenates Collected from the Mississippi River by the Minnesota Pollution Control Agency May 2009 – Phase 1: PFOS.

The aforementioned Excel file included fish fillet data for 297 fish collected from Pool 2 of the Mississippi River in 2009 that were analyzed by the MPCA for C4-C12 perfluorocarboxylic acids; C4, C6 and C8 perfluorosulfonates; and perfluorooctanesulfonamide. The 3M Environmental Laboratory Report presented fish tissue analytical results for PFOS for 30 of the 297 fish samples that were split in order to obtain confirmatory data.

Enclosed with this correspondence is a second 3M report entitled "3M Environmental Laboratory Report No. E09-0372, Fluorochemical Analysis of Fish Fillet Homogenates Collected from the Mississippi River by the Minnesota Pollution Control Agency May 2009 – Phase 2: Perfluorocarboxylic Acids (C4-C12), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS) and Perfluorooctanesulfonamide (FOSA). This report presents additional corroborative analytical data for the previously referenced 30 fish for the analytes indicated. As noted in the November 9 correspondence, sample identification numbers in the Excel spreadsheet and the 3M Environmental Laboratory Report can be cross-referenced to compare split sample results.

While 3M does not believe that any of the referenced or submitted data taken alone or cumulatively meet the "substantial risk" reporting threshold, and while 3M was not responsible for the generation of this information (other than the 3M analytical data provided here), we nevertheless recognize the ongoing work by U.S. EPA to assess fluorochemical

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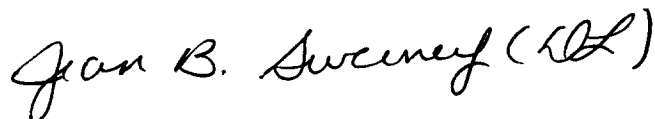
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U.S. EPA Document Processing Center
February 11, 2010
Page 2

exposure pathways. Therefore, we are placing these results in the 8(e) docket as a supplement to previous submissions.

If you have any questions, please do not hesitate to contact Deanna Luebker at (651) 737-1374 or djluebker@mmm.com.

Sincerely,

A handwritten signature in black ink that reads "Jean B. Sweeney" followed by a stylized set of initials in parentheses, likely "JBS".

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

Enclosure

Final Analytical Report

Perfluorochemical Analysis of Fish Fillet Homogenates
Collected from the Mississippi River by the Minnesota Pollution
Control Agency in May 2009 – Phase II: Perfluorocaryboxylic
Acids (C4-C12), Perfluorobutane sulfonate (PFBS),
Perfluorohexane sulfonate (PHFS) and
Perfluorooctanesulfonamide (FOSA)

Laboratory Request Number: E09-0372

Method Requirement: ETS 8-45.0

Testing Laboratory

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

Requester

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



Certificate #2052-01

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.

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 E09-0372

Perfluorochemical Analysis of Fish Fillet Homogenates Collected from the Mississippi River by the Minnesota Pollution Control Agency May 2009 – Phase II:
Perfluorocarboxylic Acids (PFCAs) C4-C12, PFBS, PFHS, and FOSA

Report Date: Upon Signing

1 Introduction

The Minnesota Pollution Control Agency (MPCA) submitted thirty fish fillet homogenates to the 3M Environmental Laboratory for perfluorochemical analysis under project number E09-0372. The thirty submitted samples represented an approximate ten percent split of the total samples collected during a May 2009 fish sampling campaign from Pool 2 of the Mississippi River. Six samples from five different species comprised the sample set. The fillet homogenates 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. Results for PFOS were provided in the Phase I report. This Phase II report provides the results from the other target analytes. The full chemical names, structures, and acronyms for each analyte are provided in Section 2.1.

The summarized results have been broken out into four separate tables:

- Table 1: perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), and perfluorohexanoic acid (PFHxA).
- Table 2: perfluoroheptanoic acid (PFHpA), perfluorooctanoic acid (PFOA), and perfluorononanoic acid (PFNA).
- Table 3: perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUnA), and perfluorododecanoic acid (PFDoA).
- Table 4: perfluorobutane sulfonate (PFBS), perfluorohexane sulfonate (PFHS), and perfluorooctanesulfonamide (FOSA).

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



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: PFCAs (C4-C6).

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA Conc (ng/g)	⁽¹⁾ PFPeA Conc (ng/g)	⁽¹⁾ PFHxA Conc (ng/g)
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	<0.275	<0.105	<0.265
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	<0.282	<0.108	<0.272
	Average			%RPD	<0.278	<0.106	<0.269
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	NA	NA	NA
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	<0.258	0.124	0.341
	Average			%RPD	<0.253	0.191	<0.244
E09-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	0.2468	<0.255	0.158	⁽²⁾ 0.341
E09-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			0.2328	NA	⁽⁴⁾ 3	NA
	Average			%RPD	<0.267	<0.102	0.312
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2364	<0.284	0.147	<0.274
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	<0.275	⁽³⁾ 0.147	⁽³⁾ 0.312
	Average			%RPD	NA	NA	NA
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	0.299	0.253	<0.270
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	0.271	0.113	<0.254
	Average			%RPD	0.285	0.183	<0.262
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	10	⁽⁴⁾ 76	NA
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2482	0.939	<0.0939	1.21
	Average			%RPD	0.979	<0.0970	1.05
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	0.959	<0.0987	1.13
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	4.2	NA	14
	Average			%RPD	<0.250	<0.0957	0.318
					<0.266	<0.102	0.293
					<0.258	<0.0987	0.306
					NA	NA	8.3
					<0.273	<0.105	<0.264
					<0.258	<0.0985	<0.249
					<0.265	<0.102	<0.256
					NA	NA	NA

Table 1 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA Conc (ng/g)	⁽¹⁾ PFPeA Conc (ng/g)	⁽¹⁾ PFHxA Conc (ng/g)
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	<0.253	<0.0869	<0.245
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	<0.263	<0.101	<0.254
	Average				<0.258	<0.0988	<0.249
	%RPD				NA	NA	NA
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	<0.278	<0.106	<0.268
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2699	<0.245	<0.0936	<0.236
	Average				<0.261	<0.0999	<0.252
	%RPD				NA	NA	NA
E09-0372-010	Axys ID- L12932-4, Client ID- 090096	Smallmouth Bass	Section 2	0.2622	<0.252	<0.0863	<0.243
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090096 Dup			0.2561	<0.258	<0.0886	<0.249
	Average				<0.255	<0.0974	<0.246
	%RPD				NA	NA	NA
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.2430	0.350	0.217	⁽³⁾ NR
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	0.441	0.270	⁽³⁾ NR
	Average				0.395	0.243	⁽³⁾ NR
	%RPD				⁽⁴⁾ 23	⁽⁴⁾ 22	NA
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	0.891	0.420	⁽³⁾ NR
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	0.605	0.175	⁽³⁾ NR
	Average				0.748	0.298	⁽³⁾ NR
	%RPD				⁽⁴⁾ 38	⁽⁴⁾ 83	NA
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	0.538	0.259	⁽³⁾ NR
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2599	0.922	0.509	⁽³⁾ NR
	Average				0.730	0.384	⁽³⁾ NR
	%RPD				⁽⁴⁾ 53	⁽⁴⁾ 65	NA
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	<0.265	<0.101	<0.504
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	<0.272	<0.104	<0.518
	Average				<0.269	<0.103	<0.511
	%RPD				NA	NA	NA

Table 1 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA Conc (ng/g)	⁽¹⁾ PFPeA Conc (ng/g)	⁽¹⁾ PFHxA Conc (ng/g)
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	<0.262	<0.100	<0.498
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	<0.265	<0.101	<0.504
	Average			Average	<0.263	<0.101	<0.501
	%RPD				NA	NA	NA
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.2570	<0.257	<0.0982	<0.488
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	<0.271	<0.104	<0.515
	Average			Average	<0.264	<0.101	<0.502
	%RPD				NA	NA	NA
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	<0.270	<0.103	<0.514
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	<0.259	<0.0992	<0.493
	Average			Average	<0.265	<0.101	<0.504
	%RPD				NA	NA	NA
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	<0.250	<0.0958	<0.475
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	<0.271	<0.104	<0.516
	Average			Average	<0.261	<0.0997	<0.496
	%RPD				NA	NA	NA
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	<0.257	<0.0883	<0.489
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	<0.267	<0.102	<0.507
	Average			Average	<0.262	<0.100	<0.498
	%RPD				NA	NA	NA
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	<0.278	<0.106	<0.529
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	<0.263	<0.101	<0.500
	Average			Average	<0.271	<0.104	<0.515
	%RPD				NA	NA	NA
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	<0.278	<0.106	<0.268
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	<0.256	<0.0981	<0.248
	Average			Average	<0.267	<0.102	<0.258
	%RPD				NA	NA	NA

Table 1 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA Conc (ng/g)	⁽³⁾ PFPeA Conc (ng/g)	⁽¹⁾ PFHxA Conc (ng/g)
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	<0.267	<0.102	<0.258
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	<0.248	<0.0960	<0.240
				Average %RPD	<0.258	<0.0987	<0.249
					NA	NA	NA
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	<0.281	<0.108	<0.272
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	<0.260	0.132	<0.251
				Average %RPD	<0.271	^(a) 0.132	<0.262
					NA	NA	NA
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	<0.277	0.157	<0.267
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	<0.272	<0.104	<0.262
				Average %RPD	<0.274	^(a) 0.157	<0.265
					NA	NA	NA
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	<0.282	<0.108	<0.273
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	<0.256	<0.0978	<0.247
				Average %RPD	<0.269	<0.103	<0.260
					NA	NA	NA
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	<0.280	0.124	<0.271
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2495	<0.265	<0.101	<0.256
				Average %RPD	<0.272	^(a) 0.124	<0.263
					NA	NA	NA
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	<0.253	0.216	<0.244
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	<0.256	0.261	<0.247
				Average %RPD	<0.254	0.238	<0.246
					NA	19	NA
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	0.532	<0.103	<0.260
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	0.414	<0.100	<0.253
				Average %RPD	0.473	<0.101	<0.256
					^(a) 25	NA	NA

Table 1 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA Conc (ng/g)	⁽¹⁾ PFPeA Conc (ng/g)	⁽¹⁾ PFHxA Conc (ng/g)
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	0.738	<0.104	<0.263
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	0.724	<0.102	0.340
				Average	0.731	<0.103	⁽²⁾ 0.340
				%RPD	1.9	NA	NA
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	<0.259	0.124	<0.251
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	<0.256	<0.0980	<0.247
				Average	<0.258	⁽³⁾ 0.124	<0.249
				%RPD	NA	NA	NA

(1) Unless otherwise specified, the recovery of the 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±13% (PFBA), 100±48% (PFPeA), and 100±39% (PFHxA). 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. PFBA and PFPeA results were generated using a solvent (unextracted) curve for quantitation. PFHxA results were quantitated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

(2) One sample result was above the limit of quantitation (LOQ) and the other replicate was below the LOQ. The average reflects only the value above the LOQ. A relative percent difference (%RPD) was not calculated.

(3) NR = not reported. The percent recovery of the associated laboratory matrix spike did not meet method criteria of 100±30%. The sample results are not reported because the accuracy could not be assessed.

(4) The %RPD exceeded 20%.

Table 2. Sample Results Summary: PFCAs (C7-C9).

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	^(b) PFHpA Conc (ng/g)	^(b) PFOA Conc (ng/g)	^(b) PFNA Conc (ng/g)
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	<0.260	<0.273	0.351
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	<0.267	<0.279	0.382
	Average	%RPD			<0.263	<0.276	0.366
					NA	NA	8.5
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	<0.245	<0.256	<0.256
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	<0.239	<0.251	<0.251
	Average	%RPD			<0.242	<0.254	<0.254
					NA	NA	NA
E09-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	0.2468	<0.253	<0.265	<0.265
E09-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			0.2328	<0.268	<0.281	<0.281
	Average	%RPD			<0.261	<0.273	<0.273
					NA	NA	NA
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2364	<0.264	<0.277	0.435
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	<0.249	0.262	0.459
	Average	%RPD			<0.257	^(a) 0.262	0.447
					NA	NA	5.4
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	<0.232	1.14	<0.243
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	<0.240	1.20	<0.252
	Average	%RPD			<0.236	1.17	<0.248
					NA	4.9	NA
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	<0.237	<0.248	<0.248
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2482	<0.252	<0.264	<0.264
	Average	%RPD			<0.244	<0.256	<0.256
					NA	NA	NA
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	<0.259	<0.271	<0.271
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	<0.244	<0.256	<0.256
	Average	%RPD			<0.251	<0.263	<0.263
					NA	NA	NA

Table 2 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	(1)PFHpA Conc (ng/g)	(1)PFOA Conc (ng/g)	(1)PFNA Conc (ng/g)
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	<0.240	<0.251	<0.251
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	<0.249	<0.261	<0.261
				Average	<0.245	<0.256	<0.256
				%RPD	NA	NA	NA
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	<0.263	<0.276	<0.276
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2698	<0.232	<0.243	<0.243
				Average	<0.247	<0.259	<0.259
				%RPD	NA	NA	NA
E09-0372-010	Axys ID- L12932-4, Client ID- 090096	Smallmouth Bass	Section 2	0.2622	<0.238	<0.250	<0.250
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090096 Dup			0.2561	<0.244	<0.256	<0.256
				Average	<0.241	<0.263	<0.263
				%RPD	NA	NA	NA
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.2430	<0.257	<0.270	<0.270
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	<0.245	<0.257	<0.257
				Average	<0.251	<0.263	<0.263
				%RPD	NA	NA	NA
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	<0.236	<0.248	<0.248
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	<0.240	<0.252	<0.252
				Average	<0.238	<0.250	<0.250
				%RPD	NA	NA	NA
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	<0.257	<0.269	0.282
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2599	<0.240	<0.252	0.295
				Average	<0.249	<0.261	0.288
				%RPD	NA	NA	4.8
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	<0.251	<0.263	<0.263
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	<0.258	<0.270	<0.270
				Average	<0.255	<0.267	<0.267
				%RPD	NA	NA	NA

Table 2 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA Conc (ng/g)	⁽¹⁾ PFOA Conc (ng/g)	⁽¹⁾ PFNA Conc (ng/g)
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	<0.248	<0.260	0.387
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	<0.251	<0.263	0.341
			Average		<0.250	<0.261	0.364
			%RPD		NA	NA	13
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.2570	<0.243	<0.255	<0.255
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	<0.257	<0.269	<0.269
			Average		<0.250	<0.262	<0.262
			%RPD		NA	NA	NA
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	<0.256	<0.268	0.275
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	<0.246	<0.257	0.298
			Average		<0.251	<0.263	0.287
			%RPD		NA	NA	7.8
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	<0.237	<0.248	0.504
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	<0.257	<0.269	0.542
			Average		<0.247	<0.259	0.523
			%RPD		NA	NA	7.2
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	<0.243	<0.255	0.526
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	<0.253	<0.265	0.516
			Average		<0.248	<0.260	0.521
			%RPD		NA	NA	1.9
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	<0.263	<0.276	0.364
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	<0.249	<0.261	0.337
			Average		<0.256	<0.269	0.350
			%RPD		NA	NA	7.7
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	<0.263	<0.276	0.617
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	<0.243	<0.254	0.616
			Average		<0.253	<0.265	0.616
			%RPD		NA	NA	0.17

Table 2 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA Conc (ng/g)	⁽¹⁾ PFOA Conc (ng/g)	⁽¹⁾ PFNA Conc (ng/g)
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	<0.253	<0.265	0.400
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	<0.235	<0.247	0.285
				Average %RPD	<0.244	<0.256	0.343
					NA	NA	^(b) 34
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	<0.267	<0.279	0.289
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	<0.246	<0.258	0.265
				Average %RPD	<0.256	<0.269	0.277
					NA	NA	8.6
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	<0.262	<0.275	<0.275
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	<0.257	<0.270	<0.270
				Average %RPD	<0.260	<0.272	<0.272
					NA	NA	NA
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	<0.267	<0.280	0.281
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	<0.242	<0.254	<0.254
				Average %RPD	<0.255	<0.267	^(b) 0.281
					NA	NA	NA
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	<0.265	<0.278	<0.278
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2496	<0.251	<0.263	<0.263
				Average %RPD	<0.258	<0.270	<0.270
					NA	NA	NA
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	<0.239	<0.251	<0.251
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	<0.242	<0.254	<0.254
				Average %RPD	<0.241	<0.252	<0.252
					NA	NA	NA
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	<0.255	<0.267	<0.267
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	<0.248	0.291	<0.260
				Average %RPD	<0.251	^(b) 0.291	<0.263
					NA	NA	NA

Table 2 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA Conc (ng/g)	⁽¹⁾ PFOA Conc (ng/g)	⁽¹⁾ PFNA Conc (ng/g)
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	<0.258	<0.271	<0.271
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	<0.252	<0.264	<0.264
				Average	<0.255	<0.267	<0.267
				%RPD	NA	NA	NA
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	<0.246	<0.257	<0.257
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	<0.243	<0.254	<0.254
				Average	<0.244	<0.256	<0.256
				%RPD	NA	NA	NA

(1) Unless otherwise specified, the recovery of the 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±21% (PFHpA), 100±19% (PFOA), and 100±22% (PFNA). 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. PFHpA, PFOA, and PFNA results were generated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

(2) One sample result was above the limit of quantitation (LOQ) and the other replicate was below the LOQ. The average reflects only the value above the LOQ. A relative percent difference (%RPD) was not calculated.

(3) The %RPD exceeded 20%.

Table 3. Sample Results Summary: PFCA's (C10-C12).

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA Conc (ng/g)	⁽¹⁾ PFUnA Conc (ng/g)	⁽¹⁾ PFDoA Conc (ng/g)
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	1.58	0.654	0.361
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	1.70	0.659	0.388
			Average	%RPD	1.64	0.657	0.375
					7.0	0.72	7.3
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	1.55	0.889	0.438
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	1.52	0.957	0.487
			Average	%RPD	1.53	0.923	0.463
					1.6	7.3	11
E09-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	0.2468	0.681	0.321	0.497
E09-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			0.2328	0.728	0.329	0.548
			Average	%RPD	0.704	0.325	0.523
					6.7	2.3	9.6
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2364	2.46	1.24	0.555
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	2.46	1.09	0.588
			Average	%RPD	2.46	1.17	0.572
					0.24	12	5.8
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	0.267	<0.238	<0.239
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	0.299	<0.246	<0.247
			Average	%RPD	0.283	<0.242	<0.243
					11	NA	NA
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	1.23	1.16	0.631
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2482	1.58	1.07	0.728
			Average	%RPD	1.41	1.11	0.680
					25	8.0	14
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	2.07	1.62	0.971
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	2.00	1.48	1.03
			Average	%RPD	2.04	1.55	1.00
					3.5	8.6	6.2

Table 3. Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA Conc (ng/g)	⁽¹⁾ PFUnA Conc (ng/g)	⁽¹⁾ PFDoA Conc (ng/g)
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	1.12	0.988	1.05
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	1.19	0.973	1.18
				Average	1.15	0.981	1.11
				%RPD	5.6	1.5	12
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	0.811	0.813	1.21
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2699	1.02	0.725	1.09
				Average	0.915	0.769	1.15
				%RPD	23	11	10
E09-0372-010	Axys ID- L12932-4, Client ID- 090086	Smallmouth Bass	Section 2	0.2622	0.912	0.531	0.530
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090086 Dup			0.2561	0.747	0.572	0.554
				Average	0.829	0.552	0.542
				%RPD	20	7.4	4.5
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.2430	1.27	1.32	2.10
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	1.22	1.24	1.97
				Average	1.24	1.28	2.04
				%RPD	3.9	6.3	6.2
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	5.55	4.06	5.12
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	6.03	4.01	5.68
				Average	5.79	4.03	5.40
				%RPD	8.4	1.4	10
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	3.36	1.62	1.26
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2599	3.42	1.78	1.44
				Average	3.39	1.70	1.35
				%RPD	1.9	9.2	13
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	3.68	1.75	1.17
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	3.47	1.87	1.14
				Average	3.57	1.81	1.16
				%RPD	5.9	6.5	1.8

Table 3 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA Conc (ng/g)	⁽¹⁾ PFUnA Conc (ng/g)	⁽¹⁾ PFDoA Conc (ng/g)
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	3.87	1.94	1.26
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	3.11	1.89	1.44
				Average	3.49	1.91	1.35
				%RPD	^(a) 22	2.6	14
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.2570	2.81	1.54	1.03
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	2.83	1.68	0.995
				Average	2.82	1.61	1.01
				%RPD	0.75	9.1	3.5
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	3.36	2.01	1.01
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	3.15	1.80	0.919
				Average	3.26	1.90	0.962
				%RPD	6.3	11	8.9
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	3.19	1.41	0.716
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	2.55	1.29	0.660
				Average	2.87	1.35	0.688
				%RPD	^(a) 22	8.6	8.2
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	4.31	1.29	0.610
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	4.51	1.36	0.703
				Average	4.41	1.33	0.656
				%RPD	4.4	5.3	14
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	3.20	1.53	0.763
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	3.52	1.34	0.663
				Average	3.36	1.43	0.715
				%RPD	9.3	13	15
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	^(a) 3.38	3.29	2.56
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	^(a) 3.82	3.40	1.96
				Average	^(a) 3.60	3.35	2.26
				%RPD	12	3.1	^(a) 26

Table 3 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA Conc (ng/g)	⁽¹⁾ PFUnA Conc (ng/g)	⁽¹⁾ PFDoA Conc (ng/g)
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	1.65	0.812	0.369
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	1.45	0.698	0.445
			Average %RPD		1.55	0.755	0.407
					13	15	19
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	1.40	0.732	0.420
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	1.41	0.896	0.507
			Average %RPD		1.40	0.814	0.464
					0.90	20	19
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	1.65	0.411	<0.269
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	1.47	0.486	<0.264
			Average %RPD		1.56	0.448	<0.267
					11	17	NA
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	2.03	1.03	0.569
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	1.73	0.938	0.465
			Average %RPD		1.88	0.985	0.517
					16	9.6	20
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	3.29	1.80	0.727
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2495	2.76	1.88	1.09
			Average %RPD		3.02	1.84	0.910
					18	4.3	140
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	1.91	1.59	0.898
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	1.70	1.41	0.867
			Average %RPD		1.81	1.50	0.883
					11	12	3.6
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	2.28	1.54	⁽³⁾ 1.30
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	2.03	1.65	⁽³⁾ 0.990
			Average %RPD		2.16	1.60	⁽³⁾ 1.15
					12	7.3	⁽²⁾ 27

Table 3 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA Conc (ng/g)	⁽¹⁾ PFUnA Conc (ng/g)	⁽¹⁾ PFDoA Conc (ng/g)
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	⁽³⁾ 1.33	1.25	0.719
E09-0372-028 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	⁽³⁾ 1.05	0.874	0.764
				Average	⁽³⁾ 1.19	1.06	0.742
				%RPD	⁽³⁾ 24	⁽³⁾ 35	6.2
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	1.74	0.879	0.627
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	1.50	1.06	0.411
				Average	1.62	0.968	0.519
				%RPD	15	18	⁽³⁾ 42

(1) Unless otherwise specified, the recovery of the 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±17% (PFDA), 100±21% (PFUnA), and 100±13% (PFDoA). See Section 4.8 *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. PFDA, PFUnA, and PFDoA results were generated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

(2) The %RPD exceeded 20%.

(3) LMS recovery exceeded 100±30%, but was within 100±60%. The sample uncertainty has been adjusted to 100±60%.

Table 4. Sample Results Summary: PFBS, PFHS, and FOSA.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBS Conc (ng/g)	⁽¹⁾ PFHS Conc (ng/g)	⁽¹⁾ FOSA Conc (ng/g)
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	<0.262	0.479	0.538
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	<0.269	0.492	0.538
	Average			Average	<0.265	0.485	0.538
	%RPD			%RPD	NA	2.7	0.061
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	<0.247	<0.256	<0.245
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	<0.241	<0.251	<0.239
	Average			Average	<0.244	<0.254	<0.242
	%RPD			%RPD	NA	NA	NA
E09-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	0.2468	<0.255	<0.265	<0.253
E09-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			0.2328	<0.271	<0.281	<0.268
	Average			Average	<0.263	<0.273	<0.261
	%RPD			%RPD	NA	NA	NA
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2364	<0.266	0.948	0.403
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	<0.251	0.869	0.447
	Average			Average	<0.259	0.908	0.425
	%RPD			%RPD	NA	8.7	10
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	<0.234	0.290	0.605
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	<0.242	0.296	0.603
	Average			Average	<0.238	0.293	0.604
	%RPD			%RPD	NA	2.0	0.35
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	<0.239	<0.248	0.510
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2482	<0.254	<0.264	0.591
	Average			Average	<0.246	<0.256	0.551
	%RPD			%RPD	NA	NA	15
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	<0.261	<0.271	0.687
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	<0.246	<0.256	0.788
	Average			Average	<0.253	<0.263	0.737
	%RPD			%RPD	NA	NA	14

Table 4 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBS Conc (ng/g)	⁽¹⁾ PFHS Conc (ng/g)	⁽¹⁾ FOSA Conc (ng/g)
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	<0.242	<0.251	0.512
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	<0.251	<0.261	0.525
				Average	<0.246	<0.256	0.518
				%RPD	NA	NA	2.4
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	<0.265	<0.276	0.616
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2699	<0.233	<0.243	0.669
				Average	<0.249	<0.259	0.642
				%RPD	NA	NA	8.2
E09-0372-010	Axys ID- L12932-4, Client ID- 090098	Smallmouth Bass	Section 2	0.2622	<0.240	<0.250	0.344
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090098 Dup			0.2561	<0.246	<0.256	0.345
				Average	<0.243	<0.253	0.344
				%RPD	NA	NA	0.11
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.2430	<0.259	<0.270	0.774
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	<0.247	<0.257	0.790
				Average	<0.253	<0.263	0.782
				%RPD	NA	NA	2.1
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	<0.238	<0.248	35.5
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	<0.242	<0.252	35.2
				Average	<0.240	<0.250	35.3
				%RPD	NA	NA	1.0
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	<0.259	<0.269	1.30
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2599	<0.242	<0.252	1.24
				Average	<0.251	<0.261	1.27
				%RPD	NA	NA	5.0
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	<0.253	<0.263	1.41
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	<0.260	<0.270	1.39
				Average	<0.257	<0.267	1.40
				%RPD	NA	NA	1.0

Table 4 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBS Conc (ng/g)	⁽¹⁾ PFHS Conc (ng/g)	⁽¹⁾ FOSA Conc (ng/g)
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	<0.250	<0.260	1.56
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	<0.253	<0.263	1.81
			Average		<0.252	<0.261	1.68
			%RPD		NA	NA	15
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.2570	⁽²⁾ 1.45	<0.255	0.956
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	⁽²⁾ <0.259	<0.269	1.17
			Average		⁽³⁾ 1.45	<0.262	1.06
			%RPD		NA	NA	20
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	<0.258	<0.268	1.73
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	<0.248	<0.257	1.80
			Average		<0.253	<0.263	1.76
			%RPD		NA	NA	3.8
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	<0.239	<0.248	2.70
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	<0.259	<0.269	2.44
			Average		<0.249	<0.259	2.57
			%RPD		NA	NA	10
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	<0.245	<0.255	0.653
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	<0.255	<0.265	0.617
			Average		<0.250	<0.260	0.635
			%RPD		NA	NA	5.8
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	<0.266	<0.276	<0.263
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	<0.251	<0.261	0.297
			Average		<0.258	<0.269	⁽²⁾ 0.297
			%RPD		NA	NA	NA
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	<0.265	<0.276	1.29
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	<0.245	<0.254	1.29
			Average		<0.255	<0.265	1.29
			%RPD		NA	NA	0.23

Table 4 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBs Conc (ng/g)	⁽¹⁾ PFHS Conc (ng/g)	⁽¹⁾ FOSA Conc (ng/g)
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	<0.255	<0.265	0.440
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	<0.237	<0.247	0.382
				Average	<0.246	<0.256	0.411
				%RPD	NA	NA	14
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	<0.269	<0.279	<0.267
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	<0.248	<0.258	<0.246
				Average	<0.258	<0.269	<0.256
				%RPD	NA	NA	NA
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	<0.284	<0.275	0.289
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	<0.259	<0.270	<0.257
				Average	<0.262	<0.272	⁽³⁾ 0.289
				%RPD	NA	NA	NA
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	<0.269	<0.280	0.598
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	<0.244	<0.254	0.609
				Average	<0.257	<0.267	0.598
				%RPD	NA	NA	3.5
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	0.295	<0.278	1.00
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2495	0.260	<0.263	1.01
				Average	0.277	<0.270	1.01
				%RPD	13	NA	1.2
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	<0.241	<0.251	0.353
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	<0.244	<0.254	0.338
				Average	<0.243	<0.252	0.346
				%RPD	NA	NA	4.5
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	0.328	<0.267	2.18
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	0.336	<0.260	2.71
				Average	0.332	<0.263	2.45
				%RPD	2.4	NA	⁽⁴⁾ 22

Table 4 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBS Conc (ng/g)	⁽¹⁾ PFHS Conc (ng/g)	⁽¹⁾ FOSA Conc (ng/g)
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	<0.260	<0.271	0.685
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	<0.254	<0.264	0.544
				Average	<0.257	<0.267	0.614
				%RPD	NA	NA	⁽⁴⁾ 23
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	<0.248	<0.257	1.46
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	<0.244	<0.254	1.37
				Average	<0.246	<0.256	1.42
				%RPD	NA	NA	6.8

(1) Unless otherwise specified, the recovery of the 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±19% (PFBS), 100±27% (PFHS), and 100±20% (FOSA). 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. PFBS, PFHS, and FOSA results were generated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

(2) LMS recovery exceeded 100±30%, but was within 100±50%. The sample uncertainty has been adjusted to 100±50%.

(3) One sample result was above the limit of quantitation (LOQ) and the other replicate was below the LOQ. The average reflects only the value above the LOQ. A relative percent difference (%RPD) was not calculated.

(4) The %RPD exceeded 20%.

2 Study and Sample Information

2.1 Target Analytes.

Table 5 below provides pertinent information regarding the target analytes, internal standards, and surrogate recovery standards investigated in this study. Surrogate recovery standards were separate isotopically labeled compounds spiked into each sample aliquot prior to extraction. The recovery of the surrogate was used to evaluate the overall extraction process and any potential matrix effects.

Table 5. Target Analyte Summary.

Compound Name	Synonym or Acronym	Formula	Analytical Purpose	Reference Standard Source
Perfluorobutanoic acid	PFBA (C4 Acid)	C_4F_7COOH	Target	Aldrich
Perfluoropentanoic acid	PFPaA (C5 Acid)	C_5F_9COOH	Target	Alfa Aesar
Perfluorohexanoic acid	PFHxA (C6 Acid)	$C_6F_{11}COOH$	Target	Oakwood Products
Perfluoroheptanoic acid	PFHpA (C7 Acid)	$C_7F_{13}COOH$	Target	Aldrich
Perfluorooctanoic acid	PFOA (C8 Acid)	$C_8F_{17}COOH$	Target	L-PFOA (linear) Wellington Labs
		$C_7F_{15}COONH_4^+$		ECF PFOA (branched) 3M Production Lot 332
Perfluorononanoic acid	PFNA (C9 Acid)	$C_9F_{17}COOH$	Target	Oakwood Products
Perfluorodecanoic acid	PFDA (C10 Acid)	$C_{10}F_{19}COOH$	Target	Oakwood Products
Perfluoroundecanoic acid	PFUnA (C11 Acid)	$C_{11}F_{21}COOH$	Target	Oakwood Products
Perfluorododecanoic acid	PFDoA (C12 Acid)	$C_{12}F_{23}COOH$	Target	Oakwood Products
Perfluorobutane sulfonate	PFBS (C4 Sulfonate)	$C_4F_9SO_3K^+$	Target	3M Production Lot 2
Perfluorohexane sulfonate	PFHS (C6 Sulfonate)	$C_6F_{13}SO_3Na^+$	Target	L-PFHS (linear) Wellington Labs
Perfluorooctane sulfonate	PFOS (C8 Sulfonate)	$C_8F_{17}SO_3K^+$	Target	L-PFOS (linear) Wellington Labs
		$C_8F_{17}SO_3K^+$		ECF PFOS (branched) 3M Production Lot 217
Perfluorooctanesulfonamide	FOSA (C8 Sulfonamide)	$C_8F_{17}SO_2NH_2$	Target	3M (L-15709)

Table 5 Continued.

Compound Name	Synonym or Acronym	Formula	Analytical Purpose	Reference Standard Source
¹³ C ₄ -Perfluorobutanoic acid	[1,2,3,4- ¹³ C ₄]PFBA	¹³ CF ₃ (¹³ CF ₂) ₂ ¹³ COOH	IS	Wellington Labs
¹³ C ₂ -Perfluorohexanoic acid	[1,2- ¹³ C ₂]PFHxA	CF ₃ (CF ₂) ₃ (¹³ CF ₂) ¹³ COOH	IS	Wellington Labs
¹³ C ₄ -Perfluorooctanoic acid	[1,2,3,4- ¹³ C ₄]PFOA	CF ₃ (CF ₂) ₃ (¹³ CF ₂) ₃ ¹³ COOH	IS	Wellington Labs
¹³ C ₅ -Perfluorononanoic acid	[1,2,3,4,5- ¹³ C ₅]PFNA	CF ₃ (CF ₂) ₃ (¹³ CF ₂) ₄ ¹³ COOH	IS	Wellington Labs
¹³ C ₂ -Perfluorodecanoic acid	[1,2- ¹³ C ₂]PFDA	CF ₃ (CF ₂) ₇ (¹³ CF ₂) ¹³ COOH	IS	Wellington Labs
¹³ C ₂ -Perfluoroundecanoic acid	[1,2- ¹³ C ₂]PFUnA	CF ₃ (CF ₂) ₈ (¹³ CF ₂) ¹³ COOH	IS	Wellington Labs
¹³ C ₂ -Perfluorododecanoic acid	[1,2- ¹³ C ₂]PFDoA	CF ₃ (CF ₂) ₉ (¹³ CF ₂) ¹³ COOH	IS	Wellington Labs
¹⁸ O ₂ -Ammonium Perfluorobutane sulfonate	[¹⁸ O ₂]PFBS	[C ₄ F ₉ S ¹⁸ O ₂ O] ⁻ NH ₄ ⁺	IS	RTI International
¹⁸ O ₂ -Ammonium Perfluorohexane sulfonate	[¹⁸ O ₂]PFHS	[C ₆ F ₁₃ S ¹⁸ O ₂ O] ⁻ NH ₄ ⁺	IS	Wellington Labs
¹³ C ₄ -Sodium Perfluorooctane sulfonate	[1,2,3,4- ¹³ C ₄]PFOS	CF ₃ (CF ₂) ₃ (¹³ CF ₂) ₃ ¹³ CF ₂ SO ₃ ⁻ Na ⁺	IS	Wellington Labs
¹⁸ O ₂ -Perfluorooctanesulfonamide	[¹⁸ O ₂]FOSA	[C ₈ F ₁₇ S ¹⁸ O ₂ NH ₂]	IS	RTI International
¹³ C ₂ -Perfluorooctanoic acid	[1,2- ¹³ C ₂]PFOA	CF ₃ (CF ₂) ₅ (¹³ CF ₂) ¹³ COOH	Surrogate	Perkin Elmer
¹⁸ O ₂ -Ammonium Perfluorooctane sulfonate	[¹⁸ O ₂]PFOS	[C ₈ F ₁₇ S ¹⁸ O ₂ O] ⁻ NH ₄ ⁺	Surrogate	RTI International

2.2 Sample Collection and Receipt.

On August 6, 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 E09-0372 and placed in frozen storage. No information was provided regarding the sampling date.

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 and if the integrity of these samples has been comprised prior to receipt.

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 rehomogenized before extraction. See Section 3.1 for more information. 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 rehomogenized frozen using dry ice.

Frozen fish homogenate was added to a pre-chilled stainless steel bowl of a Robot Coupe RSI 2Y-1 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

A 0.25 g sub-sample of the fish tissue homogenate was accurately weighed in a 50 mL polyethylene centrifuge tube. Tissue aliquots were then spiked with internal standards (ISs) 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 (laboratory control spikes, LCSs) were spiked with surrogates at 1 ng/g and the other target analytes at approximately 0.5 ng/g, 1.5 ng/g, and 8 ng/g. After spiking, the tissue homogenates were allowed to sit for 30 minutes before 2.5 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.

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. Each extraction batch included the following quality control samples:

- a thirteen 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 of all target analytes (0.5 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
- triplicate lab control spikes of PFOS only at 100 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 6. Sample Preparation Summary.

<i>Batch Number</i>	<i>Preparation Dates</i>	<i>Samples</i>
Batch 1	8/25/2009 – 8/26/2009	E09-0372-001 to E09-0372-010
Batch 2	8/27/2009 – 8/28/2009	E09-0372-021 to E09-0372-030
Batch 3	8/31/2009 – 9/1/2009	E09-0372-011 to E09-0372-020

3.3 Analysis

Analysis for the PFCs listed in Table 5 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 7 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 7. 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>269 313>119	100
PFHpA (C7 Acid)	Target	363>319 363>169	50
PFOA (C8 Acid)	Target	413>369 413>219 413>169	50
PFNA (C9 Acid)	Target	463>419 463>219 463>169	50
PFDA (C10 Acid)	Target	513>469 513>269 513>219	50
PFUnA (C11 Acid)	Target	563>519 563>269 563>219	50
PFDaA (C12 Acid)	Target	613>569 613>319 613>169	50
PFBS (C4 Sulfonate)	Target	299>80 299>99	50
PFHS (C6 Sulfonate)	Target	399>80 399>99	50
PFOS (C8 Sulfonate)	Target	499>80 499>99 499>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]PFDaA	IS for PFDaA	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	503>80	50
[¹⁸ O ₂]FOSA	IS for FOSA	502>82	50
[1,2- ¹³ C ₂]PFOA	Surrogate (Acids)	415>370	50
[¹⁸ O ₂]PFOS	Surrogate(Sulfonates, FOSA)	503>84	50

Analysis of the short-chain 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 8. 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-chain 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 8. 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 (3.0 mm × 20 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 8. 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 11 minutes) collected the transitions for PFBS, PFHpA, PFOA, [1,2 - ¹³C₂]PFOA, PFHS, PFNA and their respective ISs. The second period (time=11 min to 21.0 minutes) collected the transitions for PFDA, PFUnA, PFOS, [¹⁸O₂]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 quantitate the results for that analyte.

*Exact location of the period break may vary as individual analytical columns may perform differently or if instrument tubing is changed.

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 0.25 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 thirteen spiked standards ranging from 0.25 ng/g to 300 ng/g (nominal) were prepared. Additionally, an unextracted acetonitrile solvent curve ranging from 0.0025 ng/mL to 50 ng/mL was also prepared which is equivalent to 0.025 ng/g to 500 ng/g tissue concentration.

All sample results reported here were quantitated against the extracted curve except for PFBA and PFPeA. For PFBA, the endogenous concentration present in the bluegill fillet control tissue (historical average = 4.44 ng/g $\pm$ 8.3% for N=6 measurements) was significantly higher than the PFBA levels observed in the study samples. Adjusting the concentration of the extracted calibration standards for the endogenous level would have produced limits of quantitation very close to the lab matrix spike level rendering the sample results generally unreportable. Therefore, the solvent curve was used for reporting the sample results. As for PFPeA, the solvent curve was used for quantitation because the response (raw area counts) of the [1,2,3,4-¹³C₄]PFBA surrogate internal standard in the study samples more closely approximated the surrogate internal standard response in the solvent curve than it did in the extracted calibration curve. The average [1,2,3,4-¹³C₄]PFBA area counts in the extracted bluegill control matrix was approximately 80% (Batch 1), 56% (Batch 2), and 77% (Batch 3) of the average area counts in the solvent curve. For the study samples, the average ([1,2,3,4-¹³C₄]PFBA area counts was 97% (Batch 1), 84% (Batch 2), and 91% (Batch 3) of the average area counts in the solvent curve.

A quadratic, 1/x weighted calibration curve was used to fit the data for each analyte. 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 back-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 $\pm$ 25% (100 $\pm$ 30% for the LOQ). 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. A separate set of LCSs using an electrochemical fluorination (ECF) source of PFOS and PFOA was also included.

*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). When unextracted solvent quantitation was used, the area counts of the extracted aqueous method blanks was used to assess the LOQ.

4.3 System Suitability

Three replicate injections of a 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. All analytes met method acceptance criteria for system suitability with the following exceptions:

- Extraction Batch 1: Area Count RSDs – PFNA (13%), PFDA (10%), PFUnA (13%), [1,2-¹³C₂]PFOA (8.1%), PFBS (9.1%), and PFHS (8.3%)
- Extraction Batch 2: Area Count RSDs – PFNA (7.5%), PFUnA (11%), PFBS (17%), and PFHS (12%). Additionally, no system suitability injections were analyzed for the short acids (PFBA, PFPeA, and PFHxA).
- Extraction Batch 3: Area Count RSDs – PFOA (10%)

A method deviation has been issued and is included in the data package. The overall impact to the sample results is considered minimal. An increase in variability in the area count precision is often observed with an increased number of MRM transitions collected within a given period of the data acquisition method. With a large number of MRMs, fewer scans across the chromatographic peak are collected and overall peak shape can vary more from injection to injection. However, the calibration curve, QCs, and samples all experience the same conditions. Any increased variability observed in the system suitability injections would also be observed in the LCS data which is used to determine overall method uncertainty. Thus, the variability is accounted for in the assigned method uncertainty reported with the sample results.

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.

- For PFCAs (C7-C12), PFBS, PFHS, FOSA, [1,2-¹³C₂]PFOA, and [¹⁸O₂]PFOS all CCV injections (extracted curve) produced recoveries within 100%±25%, which met method criteria.
- For PFBA, and PFPeA, no solvent curve CCVs were analyzed in the second batch (s090831a) as the solvent curve vials were inadvertently not placed on the autosampler tray until the end of the analysis. However, the extracted curve CCVs analyzed throughout the run were compliant except for one injection of PFPeA (74.1%). This indicated that the overall instrument response was stable throughout the run.
- In the third batch, one CCV injection did not meet method acceptance criteria for PFBA (139%) and PFHxA (198%). The samples prior to this injection were LCSs. These LCSs were included in the method uncertainty determination and any noncompliant recoveries essentially increased the overall uncertainty. The sample results after this injection were not reported for PFHxA due to non-compliant LMS recoveries.

A method deviation has been issued and is included in the data package.

4.5 Blanks

Five types of blanks were prepared and analyzed with the samples: matrix blanks of bluegill fillet 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 10.3

4.6 Lab Control Spikes (LCSs)

Triplicate lab control spikes of the purchased bluegill fillet control matrix at three different levels for all analytes 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. Additional triplicate PFOS LCSs at

100 ng/g were prepared as most of the study sample concentrations were anticipated to be around this concentration level. Separate technical grade (3M electrochemical fluorination) spikes of branched/linear isomer standards of 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 [1,2-¹³C₂]PFOA and [¹⁸O₂]PFOS surrogate were spiked at a consistent 1 ng/g (0.1 ng/mL) for all LCS samples. Lab control spike recoveries are provided in the Supplemental Information (Section 10.1). For ETS 8-45.0, method acceptance criteria for LCS recoveries is 100±30% with RSD <20%.

4.7 Sample Concentration Calculation

The extracted calibration curve or 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.

$$\text{Tissue Concentration } \left(\frac{\text{ng}}{\text{g}}\right) = \frac{\text{Extract Concentration } \left(\frac{\text{ng}}{\text{mL}}\right) * \text{Extraction Volume (mL)}}{\text{Extracted Mass (g)}}$$

Extraction Volume = 2.5 mL

Extracted Mass ~0.25 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 5 ng/g except PFOS, which was spiked at a concentration of approximately 110 ng/g. The recovery of the LMS was calculated using the following equation.

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

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 27.6 ng (~110 ng/g tissue concentration) except for sample E09-0372-012 where the LMS was 128 ng (~510 ng/g tissue concentration). The FOSA LMS for this sample was also increased to ~110 ng/g tissue concentration. The [1,2-¹³C₂]PFOA and [¹⁸O₂]PFOS surrogate was spiked at 0.261 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 recoveries 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 9 below provides the analytical uncertainty for the target analytes.

Table 9. Analytical Method Uncertainty

	⁽¹⁾ PFBA	⁽¹⁾ PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA
Average %Recovery	99.2	97.9	103	91.9	101	101	97.5
Standard Deviation	6.56	24.0	19.6	10.7	9.68	11.1	8.65
2*Standard Deviation	13.1	47.9	39.1	21.3	19.4	22.2	17.3
⁽²⁾ N	50	27	50	50	50	50	50
Analytical Uncertainty	±13%	±48%	±39%	±21%	±19	±22	±17
	PFUnA	PFDoA	PFBS	PFHS	FOSA	[1,2- ¹³ C ₂]PFOA	[¹⁸ O ₂]PFOS
Average %Recovery	101	96.6	96.9	99.1	101	105	106
Standard Deviation	10.5	6.34	9.55	13.3	10.2	9.63	8.73
2*Standard Deviation	21.1	12.7	19.1	26.6	20.5	19.3	17.5
⁽²⁾ N	50	50	50	50	50	50	50
Analytical Uncertainty	±21	±13	±19%	±27%	±20%	±19	±18%

- (1) All LCS recoveries for PFBA and PFPeA used to determine uncertainty were calculated using a solvent curve. The PFPeA LCSs from this study only were used for the analytical method uncertainty determination.
- (2) For N>27, some of the historical LCSs were prepared in walleye fillet.

5 Data Summary and Discussion

5.1 All Samples

The tables below provide the results for the sample, sample duplicate, and lab matrix spikes for the target analytes and the two surrogates. Except for PFBA and PFPeA, all sample results were reported from the extracted calibration curve prepared in bluegill fillet control matrix. In general, most samples produced lab matrix spike and surrogate spike recoveries within method acceptance criteria of 100±30% with RPDs less than 20% between the sample and sample duplicate. Exceptions have been flagged appropriately in each data table.

The recoveries of the 3M PFOA ECF LCSs prepared with each extraction batch (N=9 for the study) ranged from 82.3% to 113% with a calculated average of 98.0 (11% RSD). See Section 10.1. Similarly, the recoveries of the 3M PFOS ECF LCSs (E09-0372 Phase I Report) ranged from 98.9% to 114% with a calculated average of 107% (4.7% RSD). The results of the ECF QC spikes demonstrated that there is no discernible bias when PFOA and/or PFOS ECF isomers are quantitated against the predominantly linear reference standard. Therefore, the reported PFOA and PFOS values are considered accurate to within the stated method uncertainty regardless of the degree of branched isomers present in the sample extracts.

5.2 Sample E09-0372-012 (Client ID 090064, Smallmouth Bass River Section 4)

Sample E09-0372-012 exhibited the highest concentration of PFCs collectively: PFOS (570 ng/g – Phase I report), FOSA (35.3 ng/g), PFDA (5.79 ng/g), PFDoA (5.40 ng/g), PFUnA (4.03 ng/g), PFBA (0.748 ng/g), and PFPeA (0.298 ng/g). PFHxA was detected in this sample, but quantitative concentrations could not be reported due to QC failures. For this smallmouth bass sample only (River Section 4), branched isomers of PFUnA and PFDoA were present in the fillet extract. Isomers of PFDA were present, but at a significantly less amount than the PFUnA and PFDoA isomers.

Figure 1 (PFUnA) and Figure 2 (PFDaA) display the total ion chromatograms for both E09-0372-012 and a 15 ng/g extracted calibration standard prepared in the bluegill fillet control matrix using predominantly linear reference standards for comparison. Based on raw area counts, the PFUnA branched isomers in E09-0372-012 comprise approximately 15% of the total PFUnA signal and PFDaA branched isomers comprise approximately 29% of the total PFDaA signal. For comparison, Figure 3 displays the same chromatograms for PFDA. The PFDA isomer signature accounts for approximately 1% of the total PFDA signal.

Figure 4 shows the individual MRM transitions of PFUnA and PFDaA in E09-0372-012 confirming that smaller peaks preceding the linear isomer are structural equivalents of PFUnA and PFDaA and not unidentified co-extracted matrix components. Representative 3M PFOS production lots have trace levels of PFDA (0.0005% wt), PFUnA (0.0002% wt), and PFDaA (0.0004% wt) with significant degrees of branched isomers: PFDA (51%), PFUnA (46%), and PFDaA (33%).¹ Figure 5 displays a representative chromatogram of the PFDA, PFUnA, and PFDaA impurities present in a 10 µg/mL solvent standard of 3M ECF PFOA and PFOS. For E09-0372-012, the branching profile observed for PFUnA and PFDaA is consistent with the branching profile of PFCA impurities in 3M ECF production PFOS. The small presence of PFDA isomers in this sample is not consistent with 3M ECF production PFOS. In this study, no other fish tissue samples with measurable PFDA, PFUnA, or PFDaA showed the presence of branched isomers. All study samples had levels of PFDA, PFUnA, and PFDaA above the approximate LOQ of 0.25 ng/g with the following exceptions:

- E09-0372-005 (Carp Section 4): PFDA = 0.283 ng/g (near the LOQ), PFUnA <0.242 ng/g, and PFDaA <0.243 ng/g
- E09-0372-024 (Freshwater Drum Section 1): PFDA = 1.56 ng/g, PFUnA = 0.448 ng/g and PFDaA <0.267 ng/g.

Assuming that the branched isomer response factors are equivalent to that of the linear isomer, the estimated branched isomer LOQ as a percentage of the linear isomer detected is provided in Table 15 for each sample. The average branching LOQs were 17% (PFDA), 25% (PFUnA), and 34% (PFDaA).

Additionally, the PFUnA and PFDaA total ion chromatograms for sample E09-0372-021 (Fresh water drum, Section 4) are displayed in Figure 6. This sample represents the second highest detected concentrations of both PFUnA (3.35 ng/g) and PFDaA (2.26 ng/g) for this project; however, the branched isomer signature in E09-0372-021 is not detected at approximately 7.5% and 11% of the linear isomer for PFUnA and PFDaA, respectively. Therefore this sample is not consistent with the branching profile of PFCA impurities in 3M ECF production PFOS. The PFOS concentration of this sample was 23.6 ng/g (Phase I report). Additional samples from river sections 1, 2, and 3, with measurable levels of PFDA/PFUnA/PFDaA, could not be quantitatively assessed for isomer profiles due to limitations of branched isomer LOQs.

5.3 Specificity

The MRMs of 399>80 and 399>99 (PFHS) and 499>80 (PFOS) have been documented as potential matrix interferences in some biological tissues arising from the presence of 5-pregnan-3,20-diol-3-sulfate isomers and taurodeoxycholate isomers (bile salt), respectively.² To verify that the reported quantitation was free from significant unknown matrix interferences, the raw area count ratios of 399>80 to 399>99 and 499>80 to 499>99 were evaluated for each sample extract and compared to the average area count ratios observed in the solvent calibration standards and bluegill fillet control matrix extracted calibration curve and LCSs.

¹ William K. Reagen et al., "Characterization of 3M ECF PFCA Byproducts in PFOA and PFOS Production Lots", Milwaukee SETAC North America 28th Annual Meeting (2007).

² Benskin, J.P., Bataineh, M., Martin, J.W., *Anal. Chem* 2007, 79, 6455-6464.

Table 16 displays the area count ratio of the 499>80 to 499>99 MRM transitions for each sample replicate. The area count ratio for each sample replicate was within 25% of the corresponding instrument batch average for the solvent calibration curve. The average ratio varied between the two instruments used (API 4000 Q-trap and API 4000) in this study and is likely due to differences in tuning parameters. Only three samples produced PFHS concentrations greater than the stated LOQ: E09-0372-001, -004, and -005. The area count ratio of 399>80 to 399>99 (data not shown, but is provided in the data package) for each replicate of these three samples was also within 25% of the average ratio for the solvent calibration curve.

Table 10. Sample Results Summary with QC for PFCA: C4-C6.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	(11)PFBA Conc. (ng/g) %Rec	(11)PFPeA Conc. (ng/g) %Rec	(11)PFHxA Conc. (ng/g) %Rec
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	<0.275	NA	<0.265
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	<0.282	NA	<0.272
E09-0372-001 LMS	Axys ID- L12916-14, Client ID- 090191 LMS			0.2584	4.77	93.4	4.66
	Average			%RPD	<0.278	<0.106	<0.269
					NA	NA	NA
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	<0.258	NA	0.341
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	<0.253	NA	<0.244
E09-0372-002 LMS	Axys ID- L12916-24, Client ID- 090054 LMS			0.2475	4.98	93.4	5.34
	Average			%RPD	<0.255	0.158	0.341
					NA	143	NA
E09-0372-003	Axys ID- L12916-28, Client ID- 090056	Carp	Section 1	0.2468	<0.267	NA	0.312
E09-0372-003 Dup	Axys ID- L12916-28, Client ID- 090056 Dup			0.2328	<0.284	NA	<0.274
E09-0372-003 LMS	Axys ID- L12916-28, Client ID- 090056 LMS			0.2508	4.98	94.7	5.49
	Average			%RPD	<0.275	0.147	0.312
					NA	NA	NA
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2384	0.299	NA	<0.270
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	0.271	NA	<0.254
E09-0372-004 LMS	Axys ID- L12917-3, Client ID- 090275 LMS			0.2833	4.87	91.5	4.92
	Average			%RPD	0.285	0.183	<0.262
					10	176	NA
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	0.939	NA	1.21
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	0.979	NA	1.05
E09-0372-005 LMS	Axys ID- L12917-10, Client ID- 090282 LMS			0.2632	5.59	92.4	5.84
	Average			%RPD	0.959	<0.0954	1.13
					4.2	NA	14

Table 10 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ pFBA		⁽¹⁾ pPFA		⁽¹⁾ pFHxA	
					Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	<0.250	NA	<0.0957	NA	0.318	NA
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2482	<0.266	NA	<0.102	NA	0.293	NA
E09-0372-006 LMS	Axys ID- L12917-17, Client ID- 090123 LMS			0.2594	4.73	93.0	4.91	99.4	5.20	102
			Average		<0.258		<0.0987		0.306	
			%RPD		NA		NA		8.3	
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	<0.273	NA	<0.105	NA	<0.284	NA
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	<0.258	NA	<0.0985	NA	<0.249	NA
E09-0372-007 LMS	Axys ID- L12931-6, Client ID- 090173 LMS			0.2689	4.51	91.9	5.14	108	4.62	99.4
			Average		<0.265		<0.0988		<0.256	
			%RPD		NA		NA		NA	
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	<0.253	NA	<0.0969	NA	<0.245	NA
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	<0.263	NA	<0.101	NA	<0.254	NA
E09-0372-008 LMS	Axys ID- L12931-14, Client ID- 090032 LMS			0.2601	4.66	91.9	5.33	108	5.16	107
			Average		<0.258		<0.0999		<0.249	
			%RPD		NA		NA		NA	
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	<0.278	NA	<0.106	NA	<0.268	NA
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2699	<0.245	NA	<0.0936	NA	<0.236	NA
E09-0372-009 LMS	Axys ID- L12932-2, Client ID- 090094 LMS			0.2688	4.69	95.5	5.07	106	4.57	98.2
			Average		<0.261		<0.0999		<0.252	
			%RPD		NA		NA		NA	
E09-0372-010	Axys ID- L12932-4, Client ID- 090096	Smallmouth Bass	Section 2	0.2622	<0.252	NA	<0.0963	NA	<0.243	NA
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090096 Dup			0.2561	<0.258	NA	<0.0986	NA	<0.249	NA
E09-0372-010 LMS	Axys ID- L12932-4, Client ID- 090096 LMS			0.2434	5.24	96.6	5.69	108	4.99	97.2
			Average		<0.255		<0.0974		<0.246	
			%RPD		NA		NA		NA	

Table 10 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA		⁽¹⁾ PFPeA		⁽¹⁾ PFHxA	
					Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.243	0.350	NA	0.217	NA	0 ³ NR	NA
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	0.441	NA	0.270	NA	0 ³ NR	NA
E09-0372-011 LMS	Axys ID- L12932-9, Client ID- 090042 LMS			0.2483	4.68	80.6	4.67	85.9	0 ³ NR	NR
	Average			%RPD	0.395		0.243		0 ³ NR	
					4 ²³		4 ²²		NA	
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	0.891	NA	0.420	NA	0 ³ NR	NA
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	0.605	NA	0.175	NA	0 ³ NR	NA
E09-0372-012 LMS	Axys ID- L12932-26, Client ID- 090248 LMS			0.2667	4.89	83.7	4.33	84.0	0 ³ NR	NR
	Average			%RPD	0.748		0.298		0 ³ NR	
					4 ³⁸		4 ⁸³		NA	
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	0.538	NA	0.259	NA	0 ³ NR	NA
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2599	0.922	NA	0.509	NA	0 ³ NR	NA
E09-0372-013 LMS	Axys ID- L12933-18, Client ID- 090064 LMS			0.2547	4.96	81.5	4.66	85.1	0 ³ NR	NR
	Average			%RPD	0.730		0.384		0 ³ NR	
					4 ⁵³		4 ⁶⁵		NA	
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	<0.265	NA	<0.101	NA	<0.504	NA
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	<0.272	NA	<0.104	NA	<0.518	NA
E09-0372-014 LMS	Axys ID- L12933-20, Client ID- 090066 LMS			0.2666	4.88	98.5	4.44	92.4	4.40	93.8
	Average			%RPD	<0.269		<0.103		<0.511	
					NA		NA		NA	
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	<0.262	NA	<0.100	NA	<0.498	NA
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	<0.265	NA	<0.101	NA	<0.504	NA
E09-0372-015 LMS	Axys ID- L12933-25, Client ID- 090071 LMS			0.2461	5.18	96.2	4.65	89.5	4.87	95.8
	Average			%RPD	<0.263		<0.101		<0.501	
					NA		NA		NA	

Table 10 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA		⁽¹⁾ PFPeA		⁽¹⁾ PFHxA	
					Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.257	<0.257	NA	<0.0982	NA	<0.488	NA
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	<0.271	NA	<0.104	NA	<0.515	NA
E09-0372-016 LMS	Axys ID- L12934-10, Client ID- 090024 LMS			0.2633	4.70	93.8	4.28	88.1	4.45	93.8
			Average	%RPD	<0.264		<0.101		<0.502	
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	<0.270	NA	<0.103	NA	<0.514	NA
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	<0.259	NA	<0.0992	NA	<0.493	NA
E09-0372-017 LMS	Axys ID- L12934-12, Client ID- 090026 LMS			0.2528	4.93	94.5	4.98	98.4	4.70	95.0
			Average	%RPD	<0.265		<0.101		<0.504	
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	<0.250	NA	<0.0956	NA	<0.475	NA
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	<0.271	NA	<0.104	NA	<0.516	NA
E09-0372-018 LMS	Axys ID- L12934-18, Client ID- 090017 LMS			0.2549	5.08	98.1	4.81	95.7	4.59	93.6
			Average	%RPD	<0.261		<0.0997		<0.496	
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	<0.257	NA	<0.0983	NA	<0.489	NA
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	<0.267	NA	<0.102	NA	<0.507	NA
E09-0372-019 LMS	Axys ID- L12945-1, Client ID- 090153 LMS			0.2478	5.16	96.8	4.84	93.8	4.67	92.6
			Average	%RPD	<0.262		<0.100		<0.498	
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	<0.278	NA	<0.106	NA	<0.529	NA
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	<0.263	NA	<0.101	NA	<0.500	NA
E09-0372-020 LMS	Axys ID- L12945-4, Client ID- 090167 LMS			0.2618	4.76	94.3	4.31	88.1	4.47	93.6
			Average	%RPD	<0.271		<0.104		<0.515	
					NA		NA		NA	

Table 10 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA		⁽¹⁾ PFPeA		⁽¹⁾ PFHxA	
					Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	<0.278	NA	<0.106	NA	<0.268	NA
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	<0.256	NA	<0.0981	NA	<0.248	NA
E09-0372-021 LMS	Axys ID- L12945-14, Client ID- 090241 LMS			0.2341	5.39	95.6	5.71	104	5.16	96.6
			Average		<0.267		<0.102		<0.258	
			%RPD		NA		NA		NA	
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	<0.267	NA	<0.102	NA	<0.258	NA
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	<0.248	NA	<0.0950	NA	<0.240	NA
E09-0372-022 LMS	Axys ID- L12945-16, Client ID- 090078 LMS			0.2533	4.89	93.9	5.28	104	4.48	90.8
			Average		<0.258		<0.0987		<0.249	
			%RPD		NA		NA		NA	
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	<0.281	NA	<0.108	NA	<0.272	NA
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	<0.260	NA	0.132	NA	<0.251	NA
E09-0372-023 LMS	Axys ID- L12945-19, Client ID- 090081 LMS			0.2528	5.01	96.0	5.17	99.5	4.95	100
			Average		<0.271		⁽³⁾ 0.132		<0.262	
			%RPD		NA		NA		NA	
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	<0.277	NA	0.157	NA	<0.267	NA
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	<0.272	NA	<0.104	NA	<0.262	NA
E09-0372-024 LMS	Axys ID- L12945-30, Client ID- 090009 LMS			0.2470	5.04	94.3	5.37	101	4.57	90.4
			Average		<0.274		⁽³⁾ 0.157		<0.265	
			%RPD		NA		NA		NA	
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	<0.282	NA	<0.108	NA	<0.273	NA
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	<0.256	NA	<0.0978	NA	<0.247	NA
E09-0372-025 LMS	Axys ID- L12947-7, Client ID- 090266 LMS			0.2576	5.67	111	5.74	115	4.44	91.6
			Average		<0.269		<0.103		<0.260	
			%RPD		NA		NA		NA	

Table 10 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBA		⁽¹⁾ PFPeA		⁽¹⁾ PFHxA	
					Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec	Conc. (ng/g)	%Rec
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	<0.280	NA	0.124	NA	<0.271	NA
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2495	<0.265	NA	<0.101	NA	<0.256	NA
E09-0372-026 LMS	Axys ID- L12947-8, Client ID- 090267 LMS			0.2502	4.77	90.3	5.55	106	4.87	97.4
				Average	^(a) 0.272		^(a) 0.124		<0.263	
				%RPD	NA		NA		NA	
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	<0.253	NA	0.216	NA	<0.244	NA
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	<0.256	NA	0.261	NA	<0.247	NA
E09-0372-027 LMS	Axys ID- L12947-13, Client ID- 090111 LMS			0.2691	4.81	98.1	5.96	118	4.03	88.8
				Average	<0.254		0.238		<0.246	
				%RPD	NA		19		NA	
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	0.538	NA	<0.103	NA	<0.260	NA
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	0.420	NA	<0.100	NA	<0.253	NA
E09-0372-028 LMS	Axys ID- L12947-25, Client ID- 090205 LMS			0.2488	5.38	92.6	5.57	108	4.66	92.8
				Average	0.479		<0.101		<0.256	
				%RPD	^(a) 25		NA		NA	
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	0.738	NA	<0.104	NA	<0.263	NA
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	0.724	NA	<0.102	NA	0.340	NA
E09-0372-029 LMS	Axys ID- L12947-26, Client ID- 090206 LMS			0.2397	5.65	89.4	6.14	115	5.19	93.1
				Average	0.731		<0.103		0.340	
				%RPD	1.9		NA		NA	
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	<0.259	NA	0.124	NA	<0.251	NA
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	<0.256	NA	<0.0980	NA	<0.247	NA
E09-0372-030 LMS	Axys ID- L12948-17, Client ID- 090061 LMS			0.2350	5.70	102	5.90	106	4.94	92.8
				Average	<0.258		^(a) 0.124		<0.249	
				%RPD	NA		NA		NA	

(1) Unless otherwise specified, the recovery of the 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±13% (PFBA), 100±48% (PFPeA), and 100±39% (PFHxA). 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. PFBA and PFPeA

results were generated using a solvent (unextracted) curve for quantitation. PFHxA results were quantitated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

- (2) One sample result was above the limit of quantitation (LOQ) and the other replicate was below the LOQ. The average reflects only the value above the LOQ. A relative percent difference (%RPD) was not calculated.
- (3) NR = not reported. The percent recovery of the associated laboratory matrix spike did not meet method criteria of $100 \pm 30\%$. The sample results are not reported because the accuracy could not be assessed.
- (4) The %RPD exceeded 20%.

Table 11. Sample Results Summary with QC for PFCAs: C7-C9.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA		⁽¹⁾ PFOA		⁽¹⁾ PFNA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	<0.260	NA	<0.273	NA	0.351	NA
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	<0.267	NA	<0.279	NA	0.382	NA
E09-0372-001 LMS	Axys ID- L12916-14, Client ID- 090191 LMS			0.2584	4.39	92.3	5.07	102	5.17	96.2
	Average			%RPD	<0.263		<0.276		0.366	
					NA		NA		8.5	
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	<0.245	NA	<0.256	NA	<0.256	NA
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	<0.239	NA	<0.251	NA	<0.251	NA
E09-0372-002 LMS	Axys ID- L12916-24, Client ID- 090054 LMS			0.2475	4.61	92.7	5.59	107	4.75	91.1
	Average			%RPD	<0.242		<0.254		<0.254	
					NA		NA		NA	
E09-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	0.2468	<0.253	NA	<0.265	NA	<0.265	NA
E09-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			0.2328	<0.268	NA	<0.281	NA	<0.281	NA
E09-0372-003 LMS	Axys ID- L12916-26, Client ID- 090056 LMS			0.2508	4.50	91.7	5.57	108	5.38	105
	Average			%RPD	<0.261		<0.273		<0.273	
					NA		NA		NA	
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2364	<0.264	NA	<0.277	NA	0.435	NA
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	<0.249	NA	0.262	NA	0.459	NA
E09-0372-004 LMS	Axys ID- L12917-3, Client ID- 090275 LMS			0.2633	4.23	90.7	5.55	108	4.70	86.8
	Average			%RPD	<0.257		0.262		0.447	
					NA		NA		5.4	
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	<0.232	NA	1.14	NA	<0.243	NA
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	<0.240	NA	1.20	NA	<0.252	NA
E09-0372-005 LMS	Axys ID- L12917-10, Client ID- 090282 LMS			0.2632	4.26	91.1	6.06	99.7	4.99	102
	Average			%RPD	<0.236		1.17		<0.248	
					NA		4.9		NA	

Table 11 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA		⁽¹⁾ PFOA		⁽¹⁾ PFNA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	<0.237	NA	<0.248	NA	<0.248	NA
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2492	<0.252	NA	<0.264	NA	<0.264	NA
E09-0372-006 LMS	Axys ID- L12917-17, Client ID- 090123 LMS			0.2594	4.20	88.6	5.54	111	4.62	92.8
	Average %RPD				<0.244		<0.256		<0.256	
					NA		NA		NA	
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	<0.259	NA	<0.271	NA	<0.271	NA
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	<0.244	NA	<0.256	NA	<0.256	NA
E09-0372-007 LMS	Axys ID- L12931-6, Client ID- 090173 LMS			0.2689	3.82	83.5	5.16	108	4.87	102
	Average %RPD				<0.251		<0.263		<0.263	
					NA		NA		NA	
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	<0.240	NA	<0.251	NA	<0.251	NA
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	<0.249	NA	<0.261	NA	<0.261	NA
E09-0372-008 LMS	Axys ID- L12931-14, Client ID- 090032 LMS			0.2601	4.25	89.8	4.96	100	4.86	98.1
	Average %RPD				<0.245		<0.256		<0.256	
					NA		NA		NA	
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	<0.263	NA	<0.276	NA	<0.276	NA
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2699	<0.232	NA	<0.243	NA	<0.243	NA
E09-0372-009 LMS	Axys ID- L12932-2, Client ID- 090094 LMS			0.2688	4.01	87.6	5.71	119	4.40	91.7
	Average %RPD				<0.247		<0.259		<0.259	
					NA		NA		NA	
E09-0372-010	Axys ID- L12932-4, Client ID- 090096	Smallmouth Bass	Section 2	0.2622	<0.238	NA	<0.250	NA	<0.250	NA
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090096 Dup			0.2561	<0.244	NA	<0.256	NA	<0.256	NA
E09-0372-010 LMS	Axys ID- L12932-4, Client ID- 090096 LMS			0.2434	4.38	86.6	5.76	109	5.09	96.1
	Average %RPD				<0.241		<0.253		<0.253	
					NA		NA		NA	

Table 11 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA		⁽¹⁾ PFOA		⁽¹⁾ PFNA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.2430	<0.257	NA	<0.270	NA	<0.270	NA
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	<0.245	NA	<0.257	NA	<0.257	NA
E09-0372-011 LMS	Axys ID- L12932-9, Client ID- 090042 LMS			0.2483	3.89	78.5	4.95	95.3	5.03	96.9
	Average			%RPD	<0.251		<0.263		<0.263	
					NA		NA		NA	
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	<0.236	NA	<0.248	NA	<0.248	NA
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	<0.240	NA	<0.252	NA	<0.252	NA
E09-0372-012 LMS	Axys ID- L12932-26, Client ID- 090248 LMS			0.2667	3.92	85.0	4.68	96.7	5.14	106
	Average			%RPD	<0.238		<0.250		<0.250	
					NA		NA		NA	
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	<0.257	NA	<0.269	NA	0.282	NA
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2599	<0.240	NA	<0.252	NA	0.295	NA
E09-0372-013 LMS	Axys ID- L12933-18, Client ID- 090064 LMS			0.2547	4.37	90.4	4.80	94.8	5.17	96.4
	Average			%RPD	<0.249		<0.261		0.288	
					NA		NA		4.8	
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	<0.251	NA	<0.263	NA	<0.263	NA
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	<0.258	NA	<0.270	NA	<0.270	NA
E09-0372-014 LMS	Axys ID- L12933-20, Client ID- 090066 LMS			0.2666	4.41	95.5	4.65	96.1	4.89	101
	Average			%RPD	<0.255		<0.267		<0.267	
					NA		NA		NA	
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	<0.248	NA	<0.260	NA	0.387	NA
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	<0.251	NA	<0.263	NA	0.341	NA
E09-0372-015 LMS	Axys ID- L12933-25, Client ID- 090071 LMS			0.2461	4.98	99.6	5.74	109	5.16	91.5
	Average			%RPD	<0.250		<0.261		0.364	
					NA		NA		13	

Table 11 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA			⁽¹⁾ PFOA			⁽¹⁾ PFNA		
					Conc (ng/g)	LMS %Rec		Conc (ng/g)	LMS %Rec		Conc (ng/g)	LMS %Rec	
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.2570	<0.243	NA		<0.255	NA		<0.255	NA	
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	<0.257	NA		<0.269	NA		<0.269	NA	
E09-0372-016 LMS	Axys ID- L12934-10, Client ID- 090024 LMS			0.2633	4.33	92.7		4.68	95.5		4.72	96.3	
	Average %RPD				<0.250			<0.262			<0.262		
					NA			NA			NA		
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	<0.256	NA		<0.268	NA		0.275	NA	
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	<0.246	NA		<0.257	NA		0.298	NA	
E09-0372-017 LMS	Axys ID- L12934-12, Client ID- 090026 LMS			0.2528	4.80	98.6		5.27	103		5.30	98.3	
	Average %RPD				<0.251			<0.263			0.287		
					NA			NA			7.8		
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	<0.237	NA		<0.248	NA		0.504	NA	
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	<0.257	NA		<0.269	NA		0.542	NA	
E09-0372-018 LMS	Axys ID- L12934-18, Client ID- 090017 LMS			0.2549	4.67	96.7		5.02	99.2		5.38	96.1	
	Average %RPD				<0.247			<0.259			0.523		
					NA			NA			7.2		
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	<0.243	NA		<0.255	NA		0.526	NA	
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	<0.253	NA		<0.265	NA		0.516	NA	
E09-0372-019 LMS	Axys ID- L12945-1, Client ID- 090153 LMS			0.2478	5.06	102		4.95	95.2		5.73	100	
	Average %RPD				<0.248			<0.260			0.521		
					NA			NA			1.9		
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	<0.263	NA		<0.276	NA		0.364	NA	
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	<0.249	NA		<0.261	NA		0.337	NA	
E09-0372-020 LMS	Axys ID- L12945-4, Client ID- 090167 LMS			0.2618	4.15	88.4		4.69	95.2		5.00	94.4	
	Average %RPD				<0.256			<0.269			0.350		
					NA			NA			7.7		

Table 11 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA		⁽¹⁾ PFOA		⁽¹⁾ PFNA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	<0.263	NA	<0.276	NA	0.617	NA
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	<0.243	NA	<0.254	NA	0.616	NA
E09-0372-021 LMS	Axys ID- L12945-14, Client ID- 090241 LMS			0.2341	4.72	89.8	5.84	106	6.00	97.7
				Average %RPD	<0.253		<0.265		0.616	
					NA		NA		0.17	
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	<0.253	NA	<0.265	NA	0.400	NA
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	<0.235	NA	<0.247	NA	0.285	NA
E09-0372-022 LMS	Axys ID- L12945-16, Client ID- 090078 LMS			0.2533	4.35	89.6	5.27	103	5.49	101
				Average %RPD	<0.244		<0.256		0.343	
					NA		NA		0.34	
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	<0.267	NA	<0.279	NA	0.289	NA
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	<0.246	NA	<0.258	NA	0.265	NA
E09-0372-023 LMS	Axys ID- L12945-19, Client ID- 090081 LMS			0.2528	4.04	83.1	4.73	92.6	4.91	90.7
				Average %RPD	<0.256		<0.269		0.277	
					NA		NA		8.6	
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	<0.262	NA	<0.275	NA	<0.275	NA
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	<0.257	NA	<0.270	NA	<0.270	NA
E09-0372-024 LMS	Axys ID- L12945-30, Client ID- 090009 LMS			0.2470	4.87	97.8	5.83	112	4.90	93.8
				Average %RPD	<0.260		<0.272		<0.272	
					NA		NA		NA	
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	<0.267	NA	<0.280	NA	0.281	NA
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	<0.242	NA	<0.254	NA	<0.254	NA
E09-0372-025 LMS	Axys ID- L12947-7, Client ID- 090266 LMS			0.2576	4.17	87.4	4.54	90.7	5.64	107
				Average %RPD	<0.255		<0.267		0.281	
					NA		NA		NA	

Table 11 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFHpA			⁽¹⁾ PFOA			⁽¹⁾ PFNA		
					Conc (ng/g)	LMS %Rec		Conc (ng/g)	LMS %Rec		Conc (ng/g)	LMS %Rec	
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	<0.265	NA		<0.278	NA		<0.278	NA	
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2495	<0.251	NA		<0.263	NA		<0.263	NA	
E09-0372-026 LMS	Axys ID- L12947-8, Client ID- 090267 LMS			0.2502	3.83	77.8		4.39	85.1		5.20	101	
	Average			%RPD	<0.258			<0.270			<0.270		
					NA			NA			NA		
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	<0.239	NA		<0.251	NA		<0.251	NA	
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	<0.242	NA		<0.254	NA		<0.254	NA	
E09-0372-027 LMS	Axys ID- L12947-13, Client ID- 090111 LMS			0.2691	4.37	95.5		4.72	98.4		5.04	105	
	Average			%RPD	<0.241			<0.252			<0.252		
					NA			NA			NA		
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	<0.255	NA		<0.267	NA		<0.267	NA	
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	<0.248	NA		0.291	NA		<0.280	NA	
E09-0372-028 LMS	Axys ID- L12947-25, Client ID- 090205 LMS			0.2488	3.71	75.0		4.38	78.9		4.69	90.5	
	Average			%RPD	<0.251			0.291			<0.263		
					NA			NA			NA		
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	<0.258	NA		<0.271	NA		<0.271	NA	
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	<0.252	NA		<0.264	NA		<0.264	NA	
E09-0372-029 LMS	Axys ID- L12947-26, Client ID- 090206 LMS			0.2397	3.84	74.8		3.93	73.1		5.15	95.7	
	Average			%RPD	<0.255			<0.267			<0.267		
					NA			NA			NA		
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	<0.246	NA		<0.257	NA		<0.257	NA	
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	<0.243	NA		<0.254	NA		<0.254	NA	
E09-0372-030 LMS	Axys ID- L12948-17, Client ID- 090061 LMS			0.2350	4.74	90.7		5.37	97.9		5.89	107	
	Average			%RPD	<0.244			<0.256			<0.256		
					NA			NA			NA		

(1) Unless otherwise specified, the recovery of the 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±21% (PFHpA), 100±19% (PFOA), and 100±22% (PFNA). 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. PFHpA, PFOA, and PFNA results were generated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

- (2) The %RPD exceeded 20%.
- (3) One sample result was above the limit of quantitation (LOQ) and the other replicate was below the LOQ. The average reflects only the value above the LOQ. A relative percent difference (%RPD) was not calculated.

Table 12. Sample Results Summary with QC for PFCA: C10-C12.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	PFDA		PFUnA		PFDoA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	1.58	NA	0.654	NA	0.361	NA
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	1.70	NA	0.659	NA	0.388	NA
E09-0372-001 LMS	Axys ID- L12916-14, Client ID- 090191 LMS			0.2584	6.58	103	4.98	88.7	4.75	89.7
	Average			%RPD	1.64		0.657		0.375	
					7.0		0.72		7.3	
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	1.55	NA	0.889	NA	0.438	NA
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	1.52	NA	0.957	NA	0.487	NA
E09-0372-002 LMS	Axys ID- L12916-24, Client ID- 090054 LMS			0.2475	6.72	103	5.73	94.4	5.28	94.7
	Average			%RPD	1.53		0.923		0.463	
					1.6		7.3		11	
E09-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	0.2468	0.681	NA	0.321	NA	0.497	NA
E09-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			0.2328	0.728	NA	0.328	NA	0.548	NA
E09-0372-003 LMS	Axys ID- L12916-26, Client ID- 090056 LMS			0.2508	6.21	111	4.99	92.9	5.25	94.2
	Average			%RPD	0.704		0.325		0.523	
					6.7		2.3		9.6	
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2364	2.46	NA	1.24	NA	0.555	NA
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	2.46	NA	1.09	NA	0.588	NA
E09-0372-004 LMS	Axys ID- L12917-3, Client ID- 090275 LMS			0.2633	7.16	99.8	5.84	97.7	5.28	98.4
	Average			%RPD	2.46		1.17		0.572	
					0.24		12		5.8	
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	0.267	NA	<0.238	NA	<0.239	NA
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	0.299	NA	<0.246	NA	<0.247	NA
E09-0372-005 LMS	Axys ID- L12917-10, Client ID- 090282 LMS			0.2632	5.15	103	4.71	98.4	4.61	96.2
	Average			%RPD	0.283		<0.242		<0.243	
					11		NA		NA	

Table 12 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA		⁽¹⁾ PFUnA		⁽¹⁾ PFDoA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	1.23	NA	1.16	NA	0.631	NA
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2482	1.58	NA	1.07	NA	0.728	NA
E09-0372-006 LMS	Axys ID- L12917-17, Client ID- 090123 LMS			0.2594	6.02	96.6	5.21	84.4	5.44	97.9
	Average			%RPD	1.41		1.11		0.680	
					25		8.0		14	
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	2.07	NA	1.62	NA	0.971	NA
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	2.00	NA	1.48	NA	1.03	NA
E09-0372-007 LMS	Axys ID- L12931-6, Client ID- 090173 LMS			0.2689	5.43	73.6	5.65	87.6	5.07	86.7
	Average			%RPD	2.04		1.55		1.00	
					3.5		8.6		6.2	
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	1.12	NA	0.988	NA	1.05	NA
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	1.19	NA	0.973	NA	1.18	NA
E09-0372-008 LMS	Axys ID- L12931-14, Client ID- 090032 LMS			0.2601	5.62	93.7	5.58	95.0	5.79	96.5
	Average			%RPD	1.15		0.981		1.11	
					5.6		1.5		12	
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	0.811	NA	0.813	NA	1.21	NA
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2699	1.02	NA	0.725	NA	1.09	NA
E09-0372-009 LMS	Axys ID- L12932-2, Client ID- 090094 LMS			0.2688	4.58	79.4	5.35	97.7	5.39	90.3
	Average			%RPD	0.915		0.769		1.15	
					23		11		10	
E09-0372-010	Axys ID- L12932-4, Client ID- 090096	Smallmouth Bass	Section 2	0.2622	0.912	NA	0.531	NA	0.530	NA
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090096 Dup			0.2561	0.747	NA	0.572	NA	0.554	NA
E09-0372-010 LMS	Axys ID- L12932-4, Client ID- 090096 LMS			0.2434	4.67	75.5	5.13	88.4	6.30	111
	Average			%RPD	0.829		0.552		0.542	
					20		7.4		4.5	

Table 12 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA		⁽¹⁾ PFUnA		⁽¹⁾ PFDoA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.2430	1.27	NA	1.32	NA	2.10	NA
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	1.22	NA	1.24	NA	1.97	NA
E09-0372-011 LMS	Axys ID- L12932-9, Client ID- 090042 LMS			0.2483	5.69	89.1	6.40	101	6.41	86.3
	Average				1.24		1.28		2.04	
	%RPD				3.9		6.3		6.2	
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	5.55	NA	4.06	NA	5.12	NA
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	6.03	NA	4.01	NA	5.68	NA
E09-0372-012 LMS	Axys ID- L12932-26, Client ID- 090248 LMS			0.2667	9.84	87.2	8.49	94.4	9.47	86.1
	Average				5.79		4.03		5.40	
	%RPD				8.4		1.4		10	
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	3.36	NA	1.62	NA	1.26	NA
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2598	3.42	NA	1.78	NA	1.44	NA
E09-0372-013 LMS	Axys ID- L12933-18, Client ID- 090064 LMS			0.2547	8.19	98.5	6.20	91.0	5.68	86.9
	Average				3.39		1.70		1.35	
	%RPD				1.9		9.2		13	
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	3.68	NA	1.75	NA	1.17	NA
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	3.47	NA	1.87	NA	1.14	NA
E09-0372-014 LMS	Axys ID- L12933-20, Client ID- 090066 LMS			0.2666	8.92	115	7.18	114	5.68	95.8
	Average				3.69		1.81		1.16	
	%RPD				5.9		6.5		1.8	
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	3.87	NA	1.94	NA	1.26	NA
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	3.11	NA	1.89	NA	1.44	NA
E09-0372-015 LMS	Axys ID- L12933-25, Client ID- 090071 LMS			0.2461	8.10	91.4	7.68	113	5.91	89.1
	Average				3.49		1.91		1.35	
	%RPD				21.22		2.6		14	

Table 12 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA		⁽¹⁾ PFUnA		⁽¹⁾ PFDoA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.2570	2.81	NA	1.54	NA	1.03	NA
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	2.83	NA	1.68	NA	0.995	NA
E09-0372-016 LMS	Axys ID- L12934-10, Client ID- 090024 LMS			0.2633	7.07	90.3	5.37	78.7	5.10	85.4
	Average			%RPD	2.82		1.61		1.01	
					0.75		9.1		3.5	
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	3.36	NA	2.01	NA	1.01	NA
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	3.15	NA	1.80	NA	0.919	NA
E09-0372-017 LMS	Axys ID- L12934-12, Client ID- 090026 LMS			0.2528	8.50	107	6.87	99.7	5.47	90.4
	Average			%RPD	3.26		1.90		0.962	
					6.3		11		8.9	
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	3.19	NA	1.41	NA	0.716	NA
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	2.55	NA	1.29	NA	0.660	NA
E09-0372-018 LMS	Axys ID- L12934-18, Client ID- 090017 LMS			0.2549	6.88	82.3	6.74	109	6.11	110
	Average			%RPD	2.87		1.35		0.688	
					2.22		8.6		8.2	
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	4.31	NA	1.29	NA	0.610	NA
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	4.51	NA	1.36	NA	0.703	NA
E09-0372-019 LMS	Axys ID- L12945-1, Client ID- 090153 LMS			0.2478	9.19	95.5	6.27	97.1	5.31	91.5
	Average			%RPD	4.41		1.33		0.656	
					4.4		5.3		14	
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	3.20	NA	1.53	NA	0.768	NA
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	3.52	NA	1.34	NA	0.663	NA
E09-0372-020 LMS	Axys ID- L12945-4, Client ID- 090167 LMS			0.2618	8.07	99.4	6.65	108	5.06	90.3
	Average			%RPD	3.36		1.43		0.715	
					9.3		13		15	

Table 12 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA		⁽¹⁾ PFUnA		⁽¹⁾ PFDoA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	⁽³⁾ 3.38	NA	3.29	NA	2.56	NA
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	⁽³⁾ 3.82	NA	3.40	NA	1.96	NA
E09-0372-021 LMS	Axys ID- L12945-14, Client ID- 090241 LMS			0.2341	11.4	148	7.90	84.6	7.36	94.7
	Average			%RPD	⁽³⁾ 3.60		3.35		2.26	
					12		3.1		26	
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	1.65	NA	0.812	NA	0.369	NA
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	1.45	NA	0.698	NA	0.445	NA
E09-0372-022 LMS	Axys ID- L12945-16, Client ID- 090078 LMS			0.2533	6.60	103	5.59	97.1	5.14	95.2
	Average			%RPD	1.55		0.755		0.407	
					13		15		19	
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	1.40	NA	0.732	NA	0.420	NA
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	1.41	NA	0.896	NA	0.507	NA
E09-0372-023 LMS	Axys ID- L12945-19, Client ID- 090081 LMS			0.2528	5.86	91.0	5.07	85.5	5.02	91.5
	Average			%RPD	1.40		0.814		0.464	
					0.90		20		19	
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	1.65	NA	0.411	NA	<0.269	NA
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	1.47	NA	0.486	NA	<0.264	NA
E09-0372-024 LMS	Axys ID- L12945-30, Client ID- 090009 LMS			0.2470	7.35	115	6.07	110	4.62	90.5
	Average			%RPD	1.56		0.448		<0.267	
					11		17		NA	
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	2.03	NA	1.03	NA	0.569	NA
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	1.73	NA	0.938	NA	0.465	NA
E09-0372-025 LMS	Axys ID- L12947-7, Client ID- 090266 LMS			0.2576	7.40	115	5.38	89.8	4.81	87.8
	Average			%RPD	1.88		0.985		0.517	
					16		9.6		20	

Table 12 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFDA		⁽¹⁾ PFUnA		⁽¹⁾ PFDoA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	3.29	NA	1.80	NA	0.727	NA
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2495	2.76	NA	1.88	NA	1.09	NA
E09-0372-026 LMS	Axys ID- L12947-8, Client ID- 090267 LMS			0.2502	7.94	99.3	7.88	120	5.17	84.5
	Average				3.02		1.84		0.910	
	%RPD				18		4.3		⁽²⁾ 40	
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	1.91	NA	1.59	NA	0.898	NA
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	1.70	NA	1.41	NA	0.867	NA
E09-0372-027 LMS	Axys ID- L12947-13, Client ID- 090111 LMS			0.2691	6.77	108	5.62	88.0	5.33	95.0
	Average				1.81		1.50		0.883	
	%RPD				11		12		3.6	
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	2.28	NA	1.54	NA	⁽³⁾ 1.30	NA
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	2.03	NA	1.65	NA	⁽³⁾ 0.990	NA
E09-0372-028 LMS	Axys ID- L12947-25, Client ID- 090205 LMS			0.2488	6.48	86.8	8.12	129	4.64	69.0
	Average				2.16		1.60		⁽³⁾ 1.15	
	%RPD				12		7.3		⁽³⁾ 27	
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	⁽³⁾ 1.33	NA	1.25	NA	0.719	NA
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	⁽³⁾ 1.05	NA	0.874	NA	0.764	NA
E09-0372-029 LMS	Axys ID- L12947-26, Client ID- 090206 LMS			0.2397	9.11	153	5.16	78.0	6.24	105
	Average				⁽³⁾ 1.19		1.06		0.742	
	%RPD				⁽³⁾ 24		⁽³⁾ 35		6.2	
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	1.74	NA	0.879	NA	0.627	NA
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	1.50	NA	1.06	NA	0.411	NA
E09-0372-030 LMS	Axys ID- L12948-17, Client ID- 090061 LMS			0.2350	7.30	108	5.86	91.3	4.44	73.1
	Average				1.62		0.968		0.519	
	%RPD				15		18		⁽³⁾ 42	

(1) Unless otherwise specified, the recovery of the 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±17% (PFDA), 100±21% (PFUnA), and 100±13% (PFDoA). 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. PFDA, PFUnA, and PFDoA results were generated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

- (2) The %RPD exceeded 20%.
- (3) LMS recovery exceeded $100 \pm 30\%$, but was within $100 \pm 60\%$. The sample uncertainty has been adjusted to $100 \pm 60\%$.

Table 13. Sample Results Summary with QC for PFBS, PFHS, and FOSA.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBS		⁽¹⁾ PFHS		⁽¹⁾ FOSA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	<0.262	NA	0.479	NA	0.538	NA
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	<0.269	NA	0.492	NA	0.538	NA
E09-0372-001 LMS	Axys ID- L12916-14, Client ID- 090191 LMS			0.2584	4.39	91.5	5.64	103	5.46	103
	Average			%RPD	<0.265		0.485		0.538	
					NA		2.7		0.061	
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	<0.247	NA	<0.256	NA	<0.245	NA
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	<0.241	NA	<0.251	NA	<0.239	NA
E09-0372-002 LMS	Axys ID- L12916-24, Client ID- 090054 LMS			0.2475	4.46	89.1	5.43	104	5.24	105
	Average			%RPD	<0.244		<0.254		<0.242	
					NA		NA		NA	
E09-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	0.2468	<0.255	NA	<0.265	NA	<0.253	NA
E09-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			0.2328	<0.271	NA	<0.281	NA	<0.268	NA
E09-0372-003 LMS	Axys ID- L12916-26, Client ID- 090056 LMS			0.2508	4.40	88.9	4.85	94.4	5.06	103
	Average			%RPD	<0.263		<0.273		<0.261	
					NA		NA		NA	
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2364	<0.266	NA	0.948	NA	0.403	NA
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	<0.251	NA	0.869	NA	0.447	NA
E09-0372-004 LMS	Axys ID- L12917-3, Client ID- 090275 LMS			0.2633	4.39	93.1	5.47	93.1	5.37	106
	Average			%RPD	<0.259		0.908		0.425	
					NA		8.7		10	
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	<0.234	NA	0.290	NA	0.605	NA
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	<0.242	NA	0.296	NA	0.603	NA
E09-0372-005 LMS	Axys ID- L12917-10, Client ID- 090282 LMS			0.2632	3.79	80.4	4.36	83.0	5.32	101
	Average			%RPD	<0.238		0.293		0.604	
					NA		2.0		0.35	

Table 13 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	(1) PFBS		(1) PFBS		(1) FOSA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	<0.239	NA	<0.248	NA	0.510	NA
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2482	<0.254	NA	<0.264	NA	0.591	NA
E09-0372-006 LMS	Axys ID- L12917-17, Client ID- 090123 LMS			0.2594	4.73	99.0	4.92	99.0	4.77	89.0
	Average			%RPD	<0.246		<0.256		0.551	
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	<0.261	NA	<0.271	NA	0.687	NA
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	<0.246	NA	<0.256	NA	0.788	NA
E09-0372-007 LMS	Axys ID- L12931-6, Client ID- 090173 LMS			0.2689	4.39	95.2	4.82	100	4.91	91.2
	Average			%RPD	<0.253		<0.263		0.737	
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	<0.242	NA	<0.251	NA	0.512	NA
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	<0.251	NA	<0.261	NA	0.525	NA
E09-0372-008 LMS	Axys ID- L12931-14, Client ID- 090032 LMS			0.2601	4.68	98.2	4.81	96.9	4.92	93.1
	Average			%RPD	<0.246		<0.256		0.518	
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	<0.265	NA	<0.276	NA	0.616	NA
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2689	<0.233	NA	<0.243	NA	0.689	NA
E09-0372-009 LMS	Axys ID- L12932-2, Client ID- 090094 LMS			0.2688	3.96	85.9	4.59	95.7	5.20	98.6
	Average			%RPD	<0.249		<0.259		0.642	
E09-0372-010	Axys ID- L12932-4, Client ID- 090096	Smallmouth Bass	Section 2	0.2622	<0.240	NA	<0.250	NA	0.344	NA
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090096 Dup			0.2561	<0.246	NA	<0.256	NA	0.345	NA
E09-0372-010 LMS	Axys ID- L12932-4, Client ID- 090096 LMS			0.2434	4.37	85.7	6.12	116	4.88	89.7
	Average			%RPD	<0.243		<0.253		0.344	
					NA		NA		0.11	

Table 13 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBS		⁽¹⁾ PFHS		⁽¹⁾ FOSA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.2430	<0.259	NA	<0.270	NA	0.774	NA
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	<0.247	NA	<0.257	NA	0.790	NA
E09-0372-011 LMS	Axys ID- L12932-9, Client ID- 090042 LMS			0.2483	4.38	87.7	4.90	94.4	5.39	93.0
	Average			%RPD	<0.253		<0.263		0.782	
					NA		NA		2.1	
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	<0.238	NA	<0.248	NA	35.5	NA
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	<0.242	NA	<0.252	NA	35.2	NA
E09-0372-012 LMS	Axys ID- L12932-26, Client ID- 090248 LMS			0.2667	4.59	98.8	4.65	98.1	142	101
	Average			%RPD	<0.240		<0.250		35.3	
					NA		NA		1.0	
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	<0.259	NA	<0.269	NA	1.30	NA
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2569	<0.242	NA	<0.252	NA	1.24	NA
E09-0372-013 LMS	Axys ID- L12933-18, Client ID- 090064 LMS			0.2547	4.81	98.8	4.78	94.4	5.73	92.3
	Average			%RPD	<0.251		<0.261		1.27	
					NA		NA		5.0	
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	<0.253	NA	<0.263	NA	1.41	NA
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	<0.260	NA	<0.270	NA	1.39	NA
E09-0372-014 LMS	Axys ID- L12933-20, Client ID- 090066 LMS			0.2666	4.59	98.8	4.50	93.0	5.96	98.9
	Average			%RPD	<0.257		<0.267		1.40	
					NA		NA		1.0	
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	<0.250	NA	<0.260	NA	1.56	NA
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	<0.253	NA	<0.263	NA	1.81	NA
E09-0372-015 LMS	Axys ID- L12933-25, Client ID- 090071 LMS			0.2461	5.31	105	5.87	112	6.03	87.1
	Average			%RPD	<0.252		<0.261		1.68	
					NA		NA		15	

Table 13 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	(1) PFBS		(1) PFHS		(1) FOSA	
					Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec	Conc (ng/g)	LMS %Rec
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.2570	(2) 1.45	NA	<0.255	NA	0.956	NA
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	(2) <0.259	NA	<0.269	NA	1.17	NA
E09-0372-016 LMS	Axys ID- L12934-10, Client ID- 090024 LMS			0.2633	4.68	68.6	4.52	92.2	5.35	91.7
				Average	(2,3) 1.45		<0.262		1.06	
				%RPD	NA		NA		20	
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	<0.258	NA	<0.268	NA	1.73	NA
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	<0.248	NA	<0.257	NA	1.80	NA
E09-0372-017 LMS	Axys ID- L12934-12, Client ID- 090026 LMS			0.2528	5.12	104	4.78	93.6	6.30	93.2
				Average	<0.253		<0.263		1.76	
				%RPD	NA		NA		3.8	
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	<0.239	NA	<0.248	NA	2.70	NA
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	<0.259	NA	<0.269	NA	2.44	NA
E09-0372-018 LMS	Axys ID- L12934-18, Client ID- 090017 LMS			0.2549	4.90	101	4.98	98.4	6.99	91.7
				Average	<0.249		<0.259		2.57	
				%RPD	NA		NA		10	
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	<0.245	NA	<0.255	NA	0.653	NA
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	<0.255	NA	<0.265	NA	0.617	NA
E09-0372-019 LMS	Axys ID- L12945-1, Client ID- 090153 LMS			0.2478	5.13	102	5.03	96.7	5.22	92.3
				Average	<0.250		<0.260		0.635	
				%RPD	NA		NA		5.8	
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	<0.266	NA	<0.276	NA	<0.263	NA
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	<0.251	NA	<0.261	NA	0.297	NA
E09-0372-020 LMS	Axys ID- L12945-4, Client ID- 090167 LMS			0.2618	4.59	97.0	4.70	95.3	4.86	97.1
				Average	<0.258		<0.269		(2) 0.297	
				%RPD	NA		NA		NA	

Table 13 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBS			⁽¹⁾ PFHS			⁽¹⁾ FOSA		
					Conc (ng/g)	LMS %Rec		Conc (ng/g)	LMS %Rec		Conc (ng/g)	LMS %Rec	
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	<0.265	NA		<0.276	NA		1.29	NA	
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	<0.245	NA		<0.254	NA		1.29	NA	
E09-0372-021 LMS	Axys ID- L12945-14, Client ID- 090241 LMS			0.2341	5.43	102		6.61	120		6.44	97.9	
				Average %RPD	<0.255			<0.265			1.29		
					NA			NA			0.23		
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	<0.255	NA		<0.265	NA		0.440	NA	
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	<0.237	NA		<0.247	NA		0.382	NA	
E09-0372-022 LMS	Axys ID- L12945-16, Client ID- 090078 LMS			0.2533	6.16	126		4.55	88.3		4.00	73.9	
				Average %RPD	<0.246			<0.256			0.411		
					NA			NA			14		
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	<0.269	NA		<0.279	NA		<0.267	NA	
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	<0.248	NA		<0.258	NA		<0.246	NA	
E09-0372-023 LMS	Axys ID- L12945-19, Client ID- 090081 LMS			0.2528	6.05	123		5.19	102		4.04	83.1	
				Average %RPD	<0.258			<0.269			<0.256		
					NA			NA			NA		
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	<0.264	NA		<0.275	NA		0.289	NA	
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	<0.259	NA		<0.270	NA		<0.257	NA	
E09-0372-024 LMS	Axys ID- L12945-30, Client ID- 090009 LMS			0.2470	5.57	111		5.58	107		4.77	89.9	
				Average %RPD	<0.262			<0.272			0.289		
					NA			NA			NA		
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	<0.269	NA		<0.280	NA		0.588	NA	
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	<0.244	NA		<0.254	NA		0.609	NA	
E09-0372-025 LMS	Axys ID- L12947-7, Client ID- 090266 LMS			0.2576	4.92	102		6.05	121		5.21	96.6	
				Average %RPD	<0.257			<0.267			0.598		
					NA			NA			3.5		

Table 13 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	⁽¹⁾ PFBS			⁽¹⁾ PFHS			⁽¹⁾ FOSA	
					Conc (ng/g)	LMS %Rec		Conc (ng/g)	LMS %Rec		Conc (ng/g)	LMS %Rec
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	0.295	NA		<0.278	NA		1.00	NA
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2495	0.260	NA		<0.263	NA		1.01	NA
E09-0372-026 LMS	Axys ID- L12947-8, Client ID- 090267 LMS			0.2502	4.86	92.4		5.36	104		5.80	97.4
	Average			%RPD	0.277			<0.270			1.01	
					13			NA			1.2	
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	<0.241	NA		<0.251	NA		0.353	NA
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	<0.244	NA		<0.254	NA		0.338	NA
E09-0372-027 LMS	Axys ID- L12947-13, Client ID- 090111 LMS			0.2691	4.03	87.5		4.56	95.2		5.01	102
	Average			%RPD	<0.243			<0.252			0.346	
					NA			NA			4.5	
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	0.328	NA		<0.267	NA		2.18	NA
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	0.336	NA		<0.260	NA		2.71	NA
E09-0372-028 LMS	Axys ID- L12947-25, Client ID- 090205 LMS			0.2488	5.28	98.2		4.58	88.4		6.70	86.1
	Average			%RPD	0.332			<0.263			2.45	
					2.4			NA			4.22	
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	<0.260	NA		<0.271	NA		0.685	NA
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	<0.254	NA		<0.264	NA		0.544	NA
E09-0372-029 LMS	Axys ID- L12947-26, Client ID- 090206 LMS			0.2387	5.68	110		5.98	111		5.47	94.5
	Average			%RPD	<0.257			<0.267			0.614	
					NA			NA			4.23	
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	<0.248	NA		<0.257	NA		1.46	NA
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	<0.244	NA		<0.254	NA		1.37	NA
E09-0372-030 LMS	Axys ID- L12948-17, Client ID- 090061 LMS			0.2350	4.74	89.9		6.68	122		6.32	93.7
	Average			%RPD	<0.246			<0.256			1.42	
					NA			NA			6.8	

(1) Unless otherwise specified, the recovery of the 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±19% (PFBS), 100±27% (PFHS), and 100±20% (FOSA). 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. PFBS, PFHS, and FOSA results were generated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

- (2) LMS recovery exceeded $100 \pm 30\%$, but was within $100 \pm 50\%$. The sample uncertainty has been adjusted to $100 \pm 50\%$.
- (3) One sample result was above the limit of quantitation (LOQ) and the other replicate was below the LOQ. The average reflects only the value above the LOQ. A relative percent difference (%RPD) was not calculated.
- (4) The %RPD exceeded 20%.

Table 14. Summary Results for Surrogate Recovery Standards.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	Surrogate %Recovery	
					⁽¹⁾ [1,2- ¹³ C ₃]PFOA	⁽¹⁾ [¹⁸ O ₂]PFOS
E08-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	0.2403	105	96.2
E08-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			0.2344	105	90.1
E08-0372-001 LMS	Axys ID- L12916-14, Client ID- 090191 LMS			0.2584	100	99.0
				Average	103	95.1
				%RSD	2.7	4.8
E08-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	0.2555	111	105
E08-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			0.2613	105	87.1
E08-0372-002 LMS	Axys ID- L12916-24, Client ID- 090054 LMS			0.2475	111	99.0
				Average	109	97.0
				%RSD	3.1	9.3
E08-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	0.2466	112	96.0
E08-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			0.2328	111	107
E08-0372-003 LMS	Axys ID- L12916-26, Client ID- 090056 LMS			0.2508	106	103
				Average	109	102
				%RSD	2.8	5.4
E08-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	0.2384	104	97.1
E08-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			0.2512	99.0	96.2
E08-0372-004 LMS	Axys ID- L12917-3, Client ID- 090275 LMS			0.2633	112	103
				Average	105	98.7
				%RSD	6.0	3.7
E08-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	0.2690	113	95.8
E08-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			0.2604	110	94.5
E08-0372-005 LMS	Axys ID- L12917-10, Client ID- 090282 LMS			0.2632	108	96.2
				Average	110	95.5
				%RSD	2.7	0.90

Table 14 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	Surrogate %Recovery	
					⁽¹⁾ [1,2- ¹³ C ₂]PFOA	⁽¹⁾ [¹⁸ O ₂]PFOS
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	0.2638	112	96.2
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			0.2482	114	83.2
E09-0372-006 LMS	Axys ID- L12917-17, Client ID- 090123 LMS			0.2594	101	90.2
				Average	109	89.8
				%RSD	6.5	7.2
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	0.2414	108	88.9
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			0.2563	108	88.5
E09-0372-007 LMS	Axys ID- L12931-6, Client ID- 090173 LMS			0.2689	113	91.7
				Average	109	89.7
				%RSD	2.5	2.0
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	0.2607	119	98.1
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			0.2507	121	118
E09-0372-008 LMS	Axys ID- L12931-14, Client ID- 090032 LMS			0.2601	102	101
				Average	114	106
				%RSD	9.3	10
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	0.2375	127	90.9
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			0.2699	114	101
E09-0372-009 LMS	Axys ID- L12932-2, Client ID- 090094 LMS			0.2688	123	99.0
				Average	121	97.0
				%RSD	5.3	5.5
E09-0372-010	Axys ID- L12932-4, Client ID- 090096	Smallmouth Bass	Section 2	0.2622	109	104
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090096 Dup			0.2561	1135	106
E09-0372-010 LMS	Axys ID- L12932-4, Client ID- 090096 LMS			0.2434	121	107
				Average	121	105
				%RSD	11	1.4

Table 14 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	Surrogate %Recovery	
					¹¹ [1,2- ¹³ C ₂]PFOA	¹¹ [¹⁸ O ₂]PFOS
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	0.2430	104	101
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			0.2548	105	116
E09-0372-011 LMS	Axys ID- L12932-9, Client ID- 090042 LMS			0.2483	95.3	112
				Average	101	110
				%RSD	5.2	7.2
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	0.2646	94.9	¹¹ 114
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			0.2602	99.0	¹¹ 106
E09-0372-012 LMS	Axys ID- L12932-26, Client ID- 090248 LMS			0.2667	97.1	¹¹ 107
				Average	97.0	¹¹ 109
				%RSD	2.1	4.4
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	0.2433	101	117
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			0.2599	102	118
E09-0372-013 LMS	Axys ID- L12933-18, Client ID- 090064 LMS			0.2547	110	111
				Average	104	115
				%RSD	4.6	3.6
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	0.2488	96.2	103
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			0.2424	125	107
E09-0372-014 LMS	Axys ID- L12933-20, Client ID- 090066 LMS			0.2666	122	113
				Average	114	108
				%RSD	14	5.0
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	0.2518	108	112
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			0.2492	98.1	109
E09-0372-015 LMS	Axys ID- L12933-25, Client ID- 090071 LMS			0.2461	107	107
				Average	104	109
				%RSD	5.1	2.2

Table 14 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	Surrogate %Recovery	
					⁽¹⁾ [1,2- ¹³ C ₂]PFOA	⁽¹⁾ [¹⁸ O ₂]PFOS
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	0.2570	108	97.1
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			0.2436	98.1	117
E09-0372-016 LMS	Axys ID- L12934-10, Client ID- 090024 LMS			0.2633	107	113
				Average %RSD	104	109
					5.1	9.8
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	0.2442	104	114
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			0.2545	113	112
E09-0372-017 LMS	Axys ID- L12934-12, Client ID- 090026 LMS			0.2528	110	124
				Average %RSD	109	117
					4.1	5.6
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	0.2640	104	120
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			0.2433	100	102
E09-0372-018 LMS	Axys ID- L12934-18, Client ID- 090017 LMS			0.2549	103	124
				Average %RSD	102	115
					2.0	10
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	0.2568	98.1	121
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			0.2473	103	110
E09-0372-019 LMS	Axys ID- L12945-1, Client ID- 090153 LMS			0.2478	105	126
				Average %RSD	102	119
					3.4	7.1
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	0.2372	99.0	115
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			0.2509	104	112
E09-0372-020 LMS	Axys ID- L12945-4, Client ID- 090167 LMS			0.2618	109	113
				Average %RSD	104	113
					4.6	1.8

Table 14 Continued.

3M LMS ID	Sample Description	Species	River Location	Extracted Mass (g)	Surrogate %Recovery	
					⁽¹⁾ [1,2- ¹³ C ₃]PFOA	⁽¹⁾ [¹⁸ O ₂]PFOS
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	0.2375	122	98.1
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			0.2574	118	104
E09-0372-021 LMS	Axys ID- L12945-14, Client ID- 090241 LMS			0.2341	109	127
				Average	116	110
				%RPD	6.0	14
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	0.2468	125	92.8
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			0.2657	115	120
E09-0372-022 LMS	Axys ID- L12945-16, Client ID- 090078 LMS			0.2533	111	108
				Average	117	107
				%RPD	6.3	13
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	0.2345	100	105
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			0.2537	115	104
E09-0372-023 LMS	Axys ID- L12945-19, Client ID- 090081 LMS			0.2528	130	98.1
				Average	115	102
				%RPD	13	3.6
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	0.2385	89.5	102
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			0.2430	116	108
E09-0372-024 LMS	Axys ID- L12945-30, Client ID- 090009 LMS			0.2470	111	110
				Average	105	106
				%RPD	13	3.8
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	0.2338	107	100
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			0.2583	112	113
E09-0372-025 LMS	Axys ID- L12947-7, Client ID- 090266 LMS			0.2576	102	119
				Average	107	111
				%RPD	4.5	8.8

Table 14 Continued.

3M LIMS ID	Sample Description	Species	River Location	Extracted Mass (g)	Surrogate %Recovery	
					⁽¹⁾ [1,2- ¹³ C] ₂ PFOA	⁽¹⁾ [¹⁸ O] ₂ PFOS
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	0.2355	120	107
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			0.2495	126	112
E09-0372-026 LMS	Axys ID- L12947-8, Client ID- 090267 LMS			0.2502	92.7	105
	Average			%RPD	113	108
					16	3.2
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	0.2610	⁽²⁾ 136	111
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			0.2581	87.8	96.1
E09-0372-027 LMS	Axys ID- L12947-13, Client ID- 090111 LMS			0.2691	101	114
	Average			%RPD	108	107
					⁽⁴⁾ 23	9.1
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	0.2455	103	99.0
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			0.2524	⁽²⁾ 145	103
E09-0372-028 LMS	Axys ID- L12947-25, Client ID- 090205 LMS			0.2488	106	107
	Average			%RPD	118	103
					20	3.7
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	0.2421	90.7	⁽²⁾ 136
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			0.2479	115	⁽²⁾ 138
E09-0372-029 LMS	Axys ID- L12947-26, Client ID- 090206 LMS			0.2397	108	⁽²⁾ 130
	Average			%RPD	105	⁽²⁾ 134
					12	3.0
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	0.2544	⁽²⁾ 138	116
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			0.2577	122	⁽²⁾ 133
E09-0372-030 LMS	Axys ID- L12948-17, Client ID- 090061 LMS			0.2350	98.1	101
	Average			%RSD	120	117
					17	14

(1) Unless otherwise specified, the recovery of the 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±19% ([1,2-¹³C]₂PFOA) and 100±18% ([¹⁸O]₂PFOS). 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. [1,2-¹³C]₂PFOA and [¹⁸O]₂PFOS results were generated using an extracted calibration curve prepared in a control matrix of bluegill fillet.

- (2) Recovery exceeded method criteria of 100±30%.
- (3) Results reported from a 1:100 dilution of the sample extract that was respiked with surrogates post dilution. PFOS endogenous levels greater than 500 ng/g biased the [¹⁸O₂]PFOS signal in the undiluted extract.
- (4) %RSD>20%.

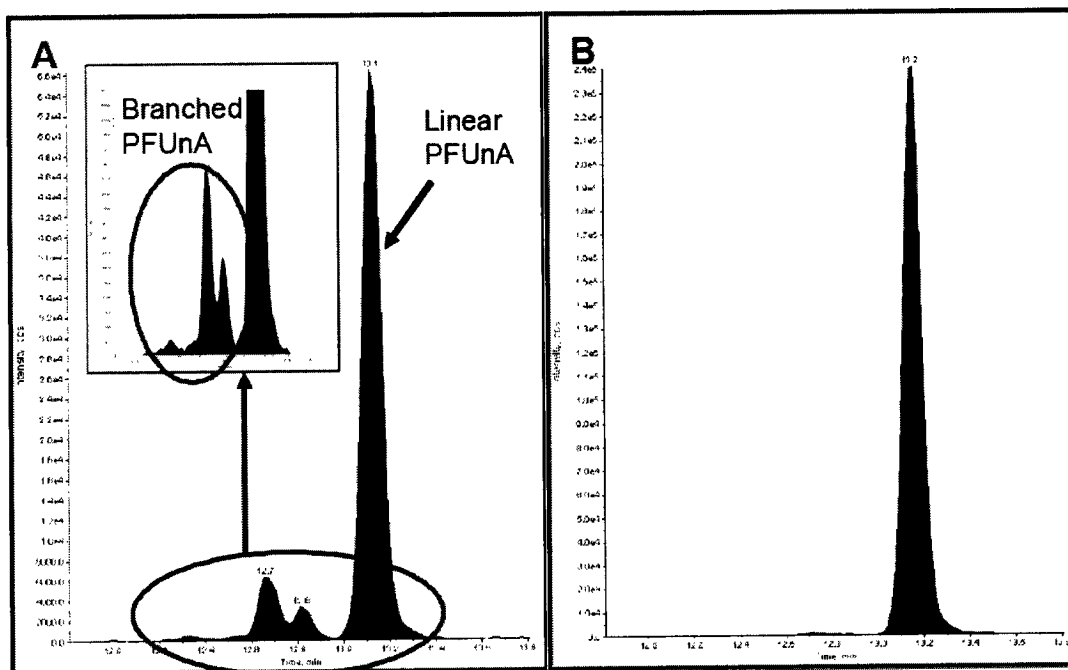


Figure 1. (A) E09-0372-012 PFUnA Total Ion Chromatogram (sum of 563>519, 563>269, and 563>219). Average PFUnA calculated sample concentration = 4.03 ng/g. Inset displays a baseline zoom of the branched isomer region. (B) PFUnA Total Ion Chromatogram of an extracted bluegill fillet calibration standard spiked at 15 ng/g with a predominantly-linear reference standard.

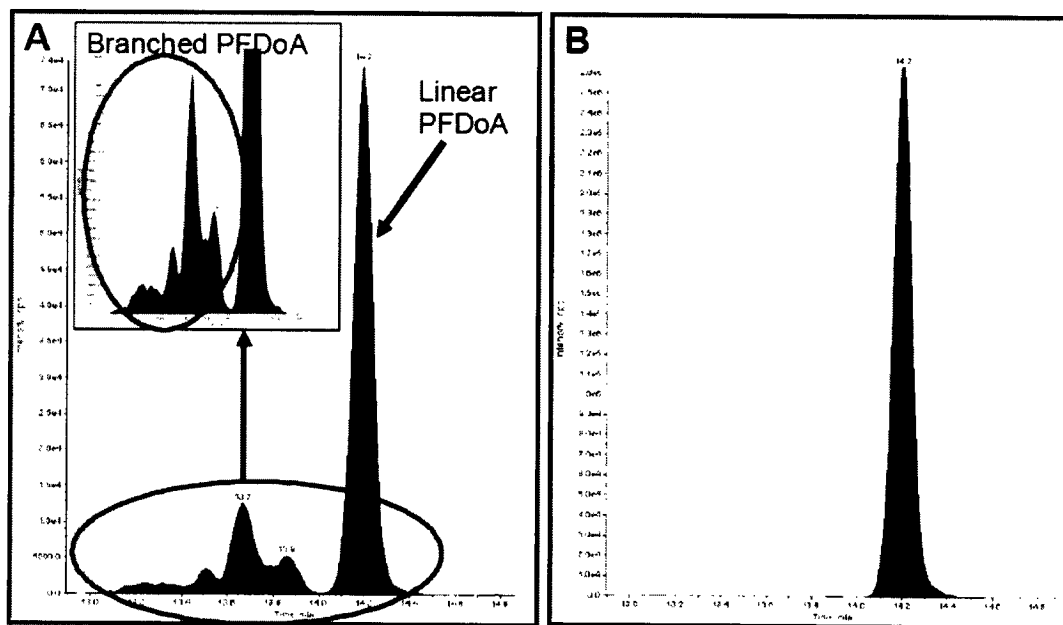


Figure 2. (A) E09-0372-012 PFDoA Total Ion Chromatogram (sum of 613>569, 613>169, and 613>319). Average PFDoA calculated sample concentration = 5.40 ng/g. Inset displays a baseline zoom of the branched isomer region. (B) PFDoA Total Ion Chromatogram of an extracted bluegill fillet calibration standard spiked at 15 ng/g with a predominantly-linear reference standard.

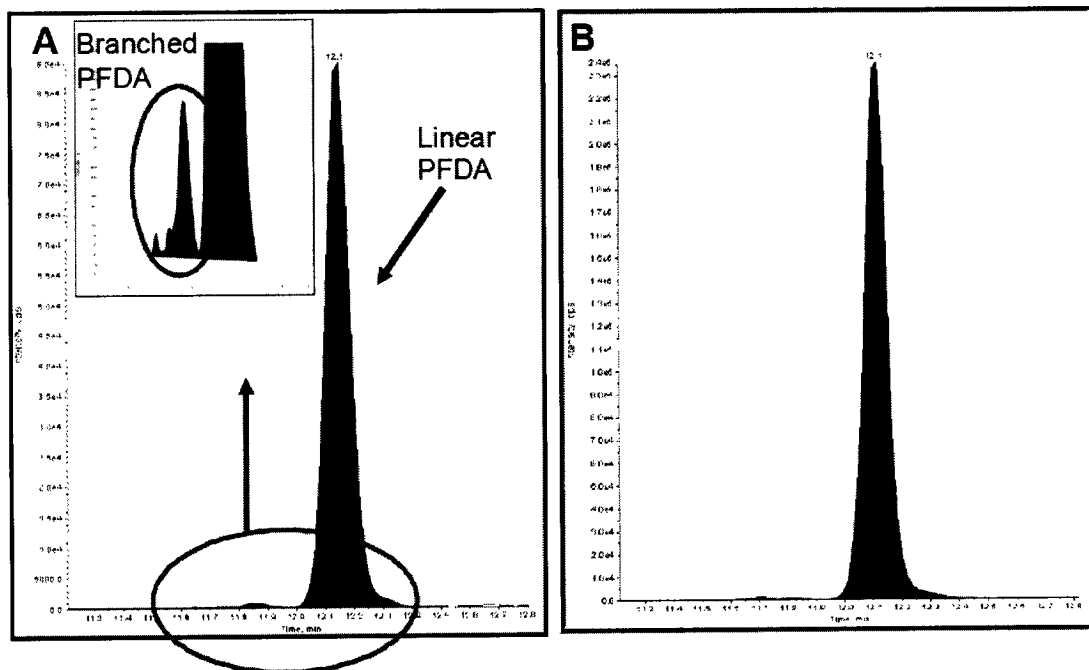


Figure 3. (A) E09-0372-012 PFDA Total Ion Chromatogram (sum of 513>469, 513>269, and 513>269). Average PFDA calculated sample concentration = 5.79 ng/g. Inset displays a baseline zoom of the branched isomer region. (B) PFDA Total Ion Chromatogram of an extracted bluegill fillet calibration standard spiked at 15 ng/g with a predominantly-linear reference standard.

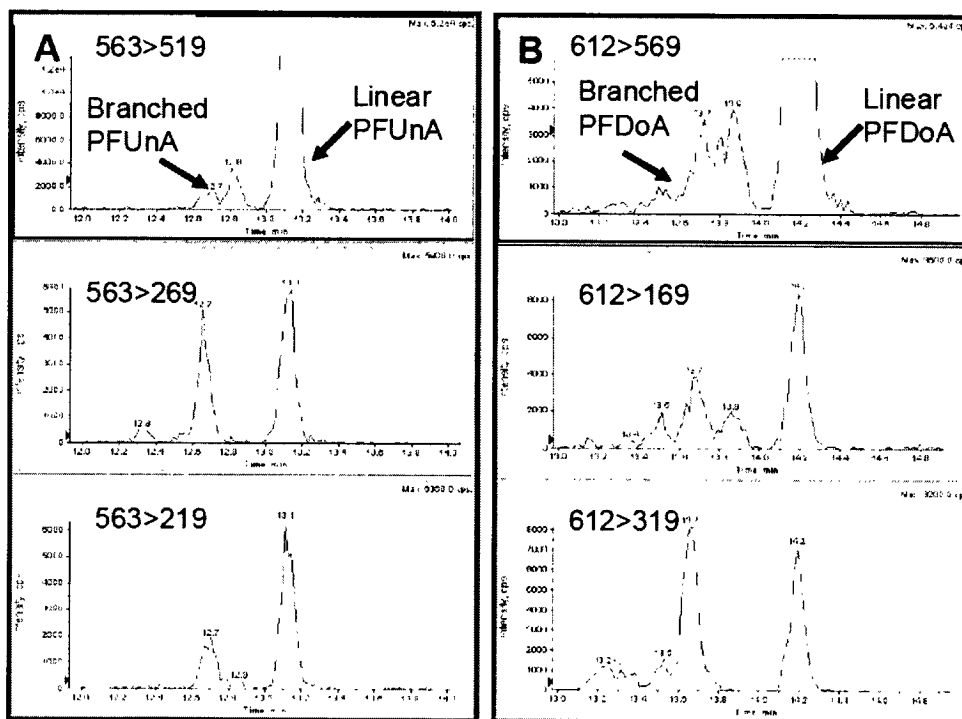


Figure 4. Individual MRM transitions of E09-0372-012. (A) PFUnA. (B). PFDoA.

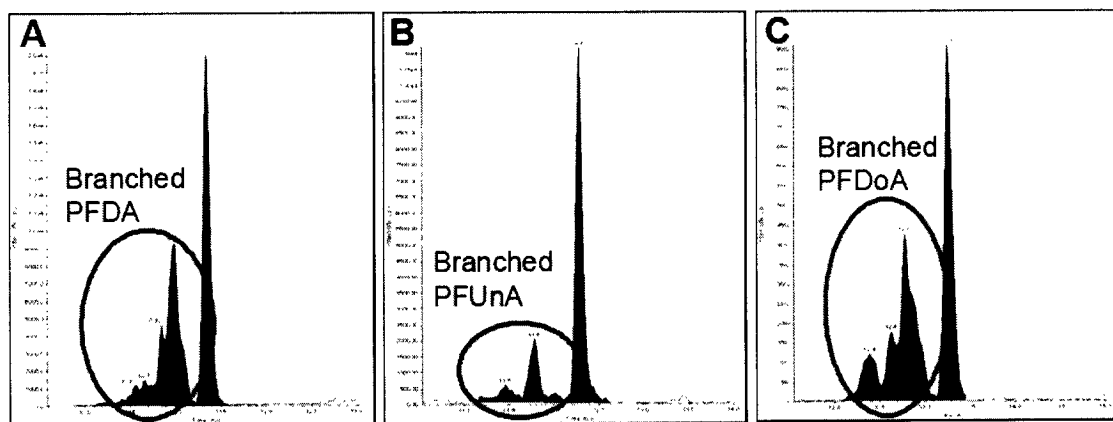


Figure 5. Representative Total Ion Chromatograms of (A) PFDA, (B) PFUnA, and (C) PFDoA Impurities in a 10 µg/mL solvent standard of technical grade (3M ECF) PFOA and PFOS.

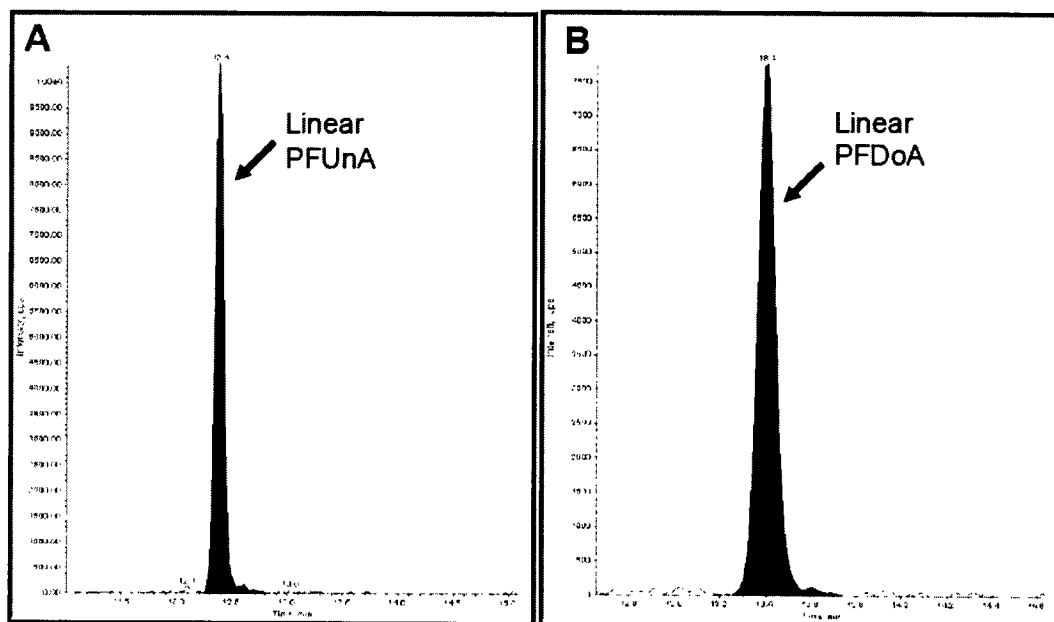


Figure 6. Total Ion Chromatograms of E09-0372-021. (A) PFUnA – average calculated sample concentration 3.35 ng/g. (B) PFDoA – average calculated sample concentration 2.26 ng/g.

Table 15. Branched Isomer Summary for PFDA, PFUnA, and PFDoA.

3M LIMS ID	Species	River Location	⁽¹⁾ Estimated Branching (as % of Linear Detected)		
			PFDA	PFUnA	PFDoA
E09-0372-001	Carp	Section 3	<15	<38	<67
E09-0372-002	Carp	Section 1	<16	<27	<54
E09-0372-003	Carp	Section 1	<36	<77	<48
E09-0372-004	Carp	Section 4	<10	<21	<44
E09-0372-005	Carp	Section 4	<88	NA	NA
E09-0372-006	Carp	Section 2	<18	<23	<37
E09-0372-007	Smallmouth Bass	Section 3	<12	<16	<25
E09-0372-008	Smallmouth Bass	Section 1	<22	<25	<23
E09-0372-009	Smallmouth Bass	Section 2	<27	<33	<22
E09-0372-010	Smallmouth Bass	Section 2	<30	<45	<46
E09-0372-011	Smallmouth Bass	Section 1	<20	<20	<12
E09-0372-012	Smallmouth Bass	Section 4	⁽¹⁾ 1% of total	⁽²⁾ 15% of total	⁽²⁾ 29% of total
E09-0372-013	White Bass	Section 2	<7.4	<15	<19
E09-0372-014	White Bass	Section 2	<7.0	<14	<22
E09-0372-015	White Bass	Section 2	<7.2	<13	<19
E09-0372-016	White Bass	Section 1	<8.9	<16	<25
E09-0372-017	White Bass	Section 1	<7.7	<13	<26
E09-0372-018	White Bass	Section 1	<8.7	<19	<36
E09-0372-019	Fresh Water Drum	Section 3	<5.7	<19	<38
E09-0372-020	Fresh Water Drum	Section 3	<7.4	<17	<35
E09-0372-021	Fresh Water Drum	Section 4	<6.9	<7.5	<11
E09-0372-022	Fresh Water Drum	Section 2	<16	<33	<61
E09-0372-023	Fresh Water Drum	Section 2	<18	<31	<54
E09-0372-024	Fresh Water Drum	Section 1	<16	<56	NA
E09-0372-025	Bluegill Sunfish	Section 3	<13	<25	<48
E09-0372-026	Bluegill Sunfish	Section 3	<8.3	<14	<27
E09-0372-027	Bluegill Sunfish	Section 2	<14	<17	<28
E09-0372-028	Bluegill Sunfish	Section 4	<12	<16	<22
E09-0372-029	Bluegill Sunfish	Section 4	<21	<24	<34
E09-0372-030	Bluegill Sunfish	Section 1	<15	<26	<48

(1) Branching LOQs estimated using the approximate linear LOQ for PFDA, PFUnA, and PFDoA (0.25 ng/g).

(2) Branched isomers detected in this sample. Reported value represents the percentage of the total PFUnA and PFDoA quantitated.

3M ENVIRONMENTAL LABORATORY
REPORT NO. E09-0372

Table 16. PFOS sample specificity: Comparison of Area Count Ratio of 499>80 to 499>99.

Instrument Batch		Average Area Count Ratio		%RSD
o090826a (API 4000 Q-trap)		499>80:499>99		
Solvent Standards (0.025 ng/mL - 50 ng/mL)		3.13		6.5
Bluegill Fillet Control Tissue (Extracted Curve and LCSs)		3.32		8.8
Sample ID	Sample Description	Species	Location	499>80:499>99 Area Count Ratio
E09-0372-001	Axys ID- L12916-14, Client ID- 090191	Carp	Section 3	3.36
E09-0372-001 Dup	Axys ID- L12916-14, Client ID- 090191 Dup			3.46
E09-0372-002	Axys ID- L12916-24, Client ID- 090054	Carp	Section 1	3.43
E09-0372-002 Dup	Axys ID- L12916-24, Client ID- 090054 Dup			3.45
E09-0372-003	Axys ID- L12916-26, Client ID- 090056	Carp	Section 1	3.58
E09-0372-003 Dup	Axys ID- L12916-26, Client ID- 090056 Dup			3.20
E09-0372-004	Axys ID- L12917-3, Client ID- 090275	Carp	Section 4	3.08
E09-0372-004 Dup	Axys ID- L12917-3, Client ID- 090275 Dup			3.37
E09-0372-005	Axys ID- L12917-10, Client ID- 090282	Carp	Section 4	3.31
E09-0372-005 Dup	Axys ID- L12917-10, Client ID- 090282 Dup			3.50
E09-0372-006	Axys ID- L12917-17, Client ID- 090123	Carp	Section 2	3.37
E09-0372-006 Dup	Axys ID- L12917-17, Client ID- 090123 Dup			3.48
E09-0372-007	Axys ID- L12931-6, Client ID- 090173	Smallmouth Bass	Section 3	3.42
E09-0372-007 Dup	Axys ID- L12931-6, Client ID- 090173 Dup			3.23
E09-0372-008	Axys ID- L12931-14, Client ID- 090032	Smallmouth Bass	Section 1	2.92
E09-0372-008 Dup	Axys ID- L12931-14, Client ID- 090032 Dup			3.18
E09-0372-009	Axys ID- L12932-2, Client ID- 090094	Smallmouth Bass	Section 2	3.08
E09-0372-009 Dup	Axys ID- L12932-2, Client ID- 090094 Dup			3.10
E09-0372-010	Axys ID- L12932-4, Client ID- 090096	Smallmouth Bass	Section 2	3.22
E09-0372-010 Dup	Axys ID- L12932-4, Client ID- 090096 Dup			2.93

3M ENVIRONMENTAL LABORATORY
REPORT NO. E09-0372

Table 16 Continued.

Instrument Batch		Average Area Count Ratio		%RSD
s090909a (API 4000)		499>80:499>99		
Solvent Stds (0.025 ng/mL - 50 ng/mL)		1.48		3.7
Bluegill Fillet Control Tissue (Extracted Curve and LCSs)		1.48		8.9
				499>80:499>99
				9
Sample ID	Sample Description	Species	Location	Area Count Ratio
E09-0372-011	Axys ID- L12932-9, Client ID- 090042	Smallmouth Bass	Section 1	1.44
E09-0372-011 Dup	Axys ID- L12932-9, Client ID- 090042 Dup			1.49
E09-0372-012	Axys ID- L12932-26, Client ID- 090248	Smallmouth Bass	Section 4	1.50
E09-0372-012 Dup	Axys ID- L12932-26, Client ID- 090248 Dup			1.47
E09-0372-013	Axys ID- L12933-18, Client ID- 090064	White Bass	Section 2	1.46
E09-0372-013 Dup	Axys ID- L12933-18, Client ID- 090064 Dup			1.44
E09-0372-014	Axys ID- L12933-20, Client ID- 090066	White Bass	Section 2	1.50
E09-0372-014 Dup	Axys ID- L12933-20, Client ID- 090066 Dup			1.54
E09-0372-015	Axys ID- L12933-25, Client ID- 090071	White Bass	Section 2	1.51
E09-0372-015 Dup	Axys ID- L12933-25, Client ID- 090071 Dup			1.44
E09-0372-016	Axys ID- L12934-10, Client ID- 090024	White Bass	Section 1	1.49
E09-0372-016 Dup	Axys ID- L12934-10, Client ID- 090024 Dup			1.50
E09-0372-017	Axys ID- L12934-12, Client ID- 090026	White Bass	Section 1	1.51
E09-0372-017 Dup	Axys ID- L12934-12, Client ID- 090026 Dup			1.43
E09-0372-018	Axys ID- L12934-18, Client ID- 090017	White Bass	Section 1	1.49
E09-0372-018 Dup	Axys ID- L12934-18, Client ID- 090017 Dup			1.45
E09-0372-019	Axys ID- L12945-1, Client ID- 090153	Fresh Water Drum	Section 3	1.44
E09-0372-019 Dup	Axys ID- L12945-1, Client ID- 090153 Dup			1.45
E09-0372-020	Axys ID- L12945-4, Client ID- 090167	Fresh Water Drum	Section 3	1.46
E09-0372-020 Dup	Axys ID- L12945-4, Client ID- 090167 Dup			1.43

3M ENVIRONMENTAL LABORATORY
REPORT NO. E09-0372

Table 16 continued.

Instrument Batch		Average Area Count Ratio		%RSD
o090828a (API 4000 Q-trap)		499>80:499>99		
Solvent Stds (0.025 ng/mL - 50 ng/mL)		3.19		8.0
Bluegill Fillet Control Tissue (Extracted Curve and LCSs)		2.97		11
				499>80:499>99 Area Count Ratio
Sample ID	Sample Description	Species	Location	
E09-0372-021	Axys ID- L12945-14, Client ID- 090241	Fresh Water Drum	Section 4	2.95
E09-0372-021 Dup	Axys ID- L12945-14, Client ID- 090241 Dup			2.83
E09-0372-022	Axys ID- L12945-16, Client ID- 090078	Fresh Water Drum	Section 2	3.19
E09-0372-022 Dup	Axys ID- L12945-16, Client ID- 090078 Dup			3.36
E09-0372-023	Axys ID- L12945-19, Client ID- 090081	Fresh Water Drum	Section 2	3.01
E09-0372-023 Dup	Axys ID- L12945-19, Client ID- 090081 Dup			2.64
E09-0372-024	Axys ID- L12945-30, Client ID- 090009	Fresh Water Drum	Section 1	3.22
E09-0372-024 Dup	Axys ID- L12945-30, Client ID- 090009 Dup			2.82
E09-0372-025	Axys ID- L12947-7, Client ID- 090266	Bluegill Sunfish	Section 3	2.97
E09-0372-025 Dup	Axys ID- L12947-7, Client ID- 090266 Dup			3.07
E09-0372-026	Axys ID- L12947-8, Client ID- 090267	Bluegill Sunfish	Section 3	3.02
E09-0372-026 Dup	Axys ID- L12947-8, Client ID- 090267 Dup			3.12
E09-0372-027	Axys ID- L12947-13, Client ID- 090111	Bluegill Sunfish	Section 2	2.87
E09-0372-027 Dup	Axys ID- L12947-13, Client ID- 090111 Dup			3.11
E09-0372-028	Axys ID- L12947-25, Client ID- 090205	Bluegill Sunfish	Section 4	3.16
E09-0372-028 Dup	Axys ID- L12947-25, Client ID- 090205 Dup			2.99
E09-0372-029	Axys ID- L12947-26, Client ID- 090206	Bluegill Sunfish	Section 4	2.93
E09-0372-029 Dup	Axys ID- L12947-26, Client ID- 090206 Dup			2.98
E09-0372-030	Axys ID- L12948-17, Client ID- 090061	Bluegill Sunfish	Section 1	3.03
E09-0372-030 Dup	Axys ID- L12948-17, Client ID- 090061 Dup			3.12

6 Conclusions

Sample results presented in Tables 1-4 and Tables 10 -14 are considered accurate within the stated overall method uncertainty for each analyte. The accuracy (% recovery) and precision (%RSD) of 3M ECF (branched) PFOA and PFOS laboratory control spikes quantitated against the predominantly linear reference standard demonstrated that the isomers produce no discernable quantitative bias. Area count ratios for 399>80:399>99 and 499>80:499>99 for each sample extract were within 25% of the average area count ratio observed for the solvent calibration curve analyzed within the given instrument batch thus verifying that the reported quantitation was free from significant matrix interferences.

7 Data/Sample Retention

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

8 Analytical Personnel

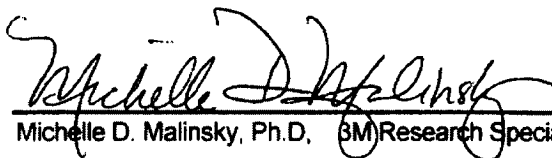
8.1 3M Environmental Laboratory

Michelle D. Malinsky, Ph.D Research Specialist

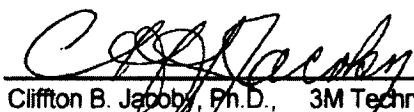
8.2 Pace Lab Ops

Jonathan Steege

3M ENVIRONMENTAL LABORATORY
REPORT NO. E09-0372**9 Signatures**

 12/16/2009
Michelle D. Malinsky, Ph.D., 3M Research Specialist, Principal Analytical Investigator Date

 16 Dec 2009
William K. Reagen, Ph.D., Environmental Laboratory Management Date

 16 Dec 2009
Clifton B. Jacoby, Ph.D., 3M Technical Reviewer Date

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

 12/15/09
QAI Representative Date

10 Supplemental Information

10.1 Lab Control Spike Recoveries by Extracted Matrix-Matched Calibration with IS Quantitation.

Table 17. LCS Recoveries for Extraction Batch 1 Using Extracted Matrix-Matched Calibration: (PFBA, PFPeA, PFHxA).

Sample ID	PFBA (extracted curve quant)			PFPeA (extracted curve quant)			PFHxA (extracted curve quant)		
	⁽¹⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Std. Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Std. Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.504	0.520	103	0.0528	0.0594	113	0.0502	0.0431	85.9
090826 BGF LCS Low 2	0.493	0.514	104	0.0528	0.0617	117	0.0502	0.0425	84.6
090826 BGF LCS Low 3	0.493	0.513	104	0.0528	0.0606	115	0.0502	0.0460	91.6
090826 BGF LCS Mid 1	0.613	0.609	99.4	0.159	0.172	108	0.151	0.156	103
090826 BGF LCS Mid 2	0.587	0.606	103	0.159	0.180	113	0.151	0.161	106
090826 BGF LCS Mid 3	0.593	0.577	97.2	0.159	0.174	110	0.151	0.145	96.1
090826 BGF LCS High 1	1.29	1.35	105	0.838	0.969	116	0.797	0.870	109
090826 BGF LCS High 2	1.28	1.28	100	0.838	0.892	106	0.797	0.762	95.7
090826 BGF LCS High 3	1.29	1.34	104	0.838	0.975	116	0.797	0.862	108
Average			102			113			97.8
%RSD			2.6			3.4			9.5

(1) Spike level of PFBA corrected for endogenous concentration of the bluegill fillet control matrix as determined by method of standard addition (PFBA Conc = 4.25 ng/g).

Table 18. LCS Recoveries for Extraction Batch 2 Using Extracted Matrix-Matched Calibration: PFBA, PFPeA, and PFHxA.

Sample ID	PFBA (extracted curve quant)			PFPeA (extracted curve quant)			PFHxA (extracted curve quant)		
	⁽¹⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Std. Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Std. Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.580	0.530	94.6	0.0510	0.0481	94.3	0.0502	0.0476	94.8
090828 BGF LCS Low 2	0.581	0.562	96.8	0.0510	0.0429	84.2	0.0502	0.0393	78.4
090828 BGF LCS Low 3	0.540	0.499	92.3	0.0510	0.0440	86.2	0.0502	0.0506	101
090828 BGF LCS Mid 1	0.681	0.632	92.9	0.154	0.137	88.8	0.151	0.142	93.8
090828 BGF LCS Mid 2	0.672	0.771	115	0.154	0.276	179	0.151	0.151	100
090828 BGF LCS Mid 3	0.686	0.674	98.2	0.154	0.141	91.3	0.151	0.138	91.1
090828 BGF LCS High 1	1.34	1.36	101	0.810	0.759	93.7	0.797	0.813	102
090828 BGF LCS High 2	1.36	1.41	103	0.810	0.805	99.3	0.797	0.814	102
090828 BGF LCS High 3	1.35	1.24	91.8	0.810	0.703	86.8	0.797	0.745	93.5
Average			98.5			100			95.2
%RSD			7.4			30			7.9

(1) Spike level of PFBA corrected for endogenous concentration of the bluegill fillet control matrix as determined by method of standard addition (PFBA Conc = 5.00 ng/g).

(2) LCS recovery exceeded 100±30%.

(3) RSD exceeds method acceptance of <±20% for all LCSs considered collectively.

Table 19. LCS Recoveries for Extraction Batch 3 Using Extracted Matrix-Matched Calibration: PFBA, PFPeA, and PFHxA.

Sample ID	PFBA (extracted quant)			PFPeA (extracted curve quant)			PFHxA (extracted curve quant)		
	⁽¹⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Std. Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Std. Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.433	0.408	94.3	0.0671	0.0585	87.2	0.0502	0.0579	115
090901 BGF LCS Low 2	0.459	0.461	101	0.0682	0.0593	86.9	0.0502	0.0878	175
090901 BGF LCS Low 3	0.467	0.428	91.7	0.0686	0.0559	81.5	0.0502	0.0722	144
090901 BGF LCS Mid 1	0.573	0.604	105	0.171	0.154	89.9	0.151	0.179	118
090901 BGF LCS Mid 2	0.551	0.548	100	0.170	0.161	94.5	0.151	0.221	146
090901 BGF LCS Mid 3	0.562	0.572	102	0.171	0.164	95.7	0.151	0.220	146
090901 BGF LCS High 1	1.26	1.25	100	0.826	0.754	91.2	0.797	0.819	103
090901 BGF LCS High 2	1.25	1.29	103	0.826	0.806	97.6	0.797	0.875	110
090901 BGF LCS High 3	1.24	1.16	93.4	0.825	0.730	88.5	0.797	0.851	107
Average			98.9			90.3			129
%RSD			4.8			5.6			19

(1) Spike level of PFBA corrected for endogenous concentration of the bluegill fillet control matrix as determined by method of standard addition (PFBA Conc = 3.92 ng/g).

(2) LCS recovery exceeded 100±30%.

Table 20. LCS Recoveries for Extraction Batch 1 Using Extracted Matrix-Matched Calibration: PFCA's (C7-C12).

Sample ID	PFHpA			PFOA			PFNA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.0492	0.0509	104	0.0516	0.0460	89.2	0.0515	0.0486	94.4
090826 BGF LCS Low 2	0.0492	0.0546	111	0.0516	0.0548	106	0.0515	0.0472	91.7
090826 BGF LCS Low 3	0.0492	0.0492	100	0.0516	0.0535	104	0.0515	0.0489	91.1
090826 BGF LCS Mid 1	0.148	0.144	97.4	0.156	0.162	104	0.155	0.147	94.9
090826 BGF LCS Mid 2	0.148	0.166	112	0.156	0.180	115	0.155	0.162	104
090826 BGF LCS Mid 3	0.148	0.145	98.2	0.156	0.159	102	0.155	0.149	96.1
090826 BGF LCS High 1	0.781	0.781	100	0.819	0.880	107	0.818	0.880	108
090826 BGF LCS High 2	0.781	0.720	92.1	0.819	0.787	96.0	0.818	0.794	97.1
090826 BGF LCS High 3	0.781	0.705	90.3	0.819	0.763	93.1	0.818	0.792	96.8
090826 BGF ECF LCS 1				0.520	0.558	107			
090826 BGF ECF LCS 2				0.520	0.570	110			
090826 BGF ECF LCS 3				0.520	0.587	113			
Average			101			102			97.1
%RSD			7.4			7.7			5.7
Sample ID	PFDA			PFUnA			PFDoA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.0497	0.0455	91.5	0.0503	0.0476	94.7	0.0506	0.0496	98.1
090826 BGF LCS Low 2	0.0497	0.0471	94.8	0.0503	0.0472	93.9	0.0506	0.0449	88.8
090826 BGF LCS Low 3	0.0497	0.0452	91.0	0.0503	0.0509	101	0.0506	0.0494	97.7
090826 BGF LCS Mid 1	0.150	0.151	101	0.152	0.146	95.9	0.152	0.157	103
090826 BGF LCS Mid 2	0.150	0.159	106	0.152	0.139	91.3	0.152	0.162	107
090826 BGF LCS Mid 3	0.150	0.168	112	0.152	0.142	93.4	0.152	0.150	98.5
090826 BGF LCS High 1	0.789	0.871	110	0.799	0.819	102	0.802	0.780	97.2
090826 BGF LCS High 2	0.789	0.773	98.0	0.799	0.693	86.7	0.802	0.775	96.6
090826 BGF LCS High 3	0.789	0.856	108	0.799	0.779	97.5	0.802	0.785	97.8
Average			101			95.2			98.3
%RSD			7.9			5.0			5.0

Table 21. LCS Recoveries for Extraction Batch 2 Using Extracted Matrix-Matched Calibration: PFCAs (C7-C12).

Sample ID	PFHpA			PFOA			PFNA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.0492	0.0457	92.9	0.0516	0.0644	125	0.0515	0.0475	92.1
090828 BGF LCS Low 2	0.0492	0.0314	⁽¹⁾ 63.8	0.0516	0.0502	97.3	0.0515	0.0478	92.9
090828 BGF LCS Low 3	0.0492	0.0349	71.0	0.0516	0.0457	88.5	0.0515	0.0565	110
090828 BGF LCS Mid 1	0.148	0.124	83.7	0.156	0.136	87.2	0.155	0.150	97.0
090828 BGF LCS Mid 2	0.148	0.156	105	0.156	0.183	117	0.155	0.162	104
090828 BGF LCS Mid 3	0.148	0.115	78.0	0.156	0.153	98.2	0.155	0.160	103
090828 BGF LCS High 1	0.781	0.807	103	0.819	1.07	⁽¹⁾ 131	0.818	0.822	101
090828 BGF LCS High 2	0.781	0.820	105	0.819	0.925	113	0.818	0.769	94.1
090828 BGF LCS High 3	0.781	0.824	79.9	0.819	0.813	99.2	0.818	0.780	95.4
090828 BGF ECF LCS 1				0.520	0.460	88.4			
090828 BGF ECF LCS 2				0.520	0.428	82.3			
090828 BGF ECF LCS 3				0.520	0.550	106			
Average			86.9			106			98.8
%RSD			18			15			6.1
Sample ID	PFDA			PFUnA			PFDoA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.0497	0.0428	86.1	0.0503	0.0568	113	0.0506	0.0511	101
090828 BGF LCS Low 2	0.0497	0.0513	103	0.0503	0.0690	⁽¹⁾ 137	0.0506	0.0463	91.6
090828 BGF LCS Low 3	0.0497	0.0407	81.8	0.0503	0.0676	⁽¹⁾ 134	0.0506	0.0484	95.6
090828 BGF LCS Mid 1	0.150	0.154	103	0.152	0.125	82.0	0.152	0.149	97.8
090828 BGF LCS Mid 2	0.150	0.166	111	0.152	0.161	106	0.152	0.130	85.8
090828 BGF LCS Mid 3	0.150	0.155	103	0.152	0.146	96.2	0.152	0.159	105
090828 BGF LCS High 1	0.789	0.963	122	0.799	0.770	96.4	0.802	0.826	103
090828 BGF LCS High 2	0.789	0.834	106	0.799	0.913	114	0.802	0.760	94.7
090828 BGF LCS High 3	0.789	0.796	101	0.799	0.719	90.0	0.802	0.742	92.5
Average			102			108			96.3
%RSD			12			18			6.3

(1) LCS recovery exceeded 100±30%.

Table 22. LCS Recoveries for Extraction Batch 3 Using Extracted Matrix-Matched Calibration: PFCAs (C7-C12).

Sample ID	PFHpA			PFOA			PFNA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.0492	0.0439	89.2	0.0516	0.0490	95.0	0.0515	0.0520	101
090901 BGF LCS Low 2	0.0492	0.0341	(1)89.4	0.0516	0.0482	93.4	0.0515	0.0474	92.0
090901 BGF LCS Low 3	0.0492	0.0396	80.4	0.0516	0.0561	109	0.0515	0.0523	102
090901 BGF LCS Mid 1	0.148	0.117	79.1	0.156	0.143	91.9	0.155	0.146	94.5
090901 BGF LCS Mid 2	0.148	0.128	86.7	0.156	0.153	97.9	0.155	0.170	110
090901 BGF LCS Mid 3	0.148	0.116	78.3	0.156	0.150	96.0	0.155	0.158	102
090901 BGF LCS High 1	0.781	0.595	76.2	0.819	0.827	101	0.818	0.890	109
090901 BGF LCS High 2	0.781	0.693	88.7	0.819	0.934	114	0.818	0.906	111
090901 BGF LCS High 3	0.781	0.604	77.4	0.819	0.755	92.1	0.818	0.775	94.8
090901 BGF ECF LCS 1				0.520	0.492	94.7			
090901 BGF ECF LCS 2				0.520	0.501	96.4			
090901 BGF ECF LCS 3				0.520	0.506	97.3			
Average			80.6			98.9			102
%RSD			8.1			7.9			7.0
Sample ID	PFDA			PFUnA			PFDoA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.0497	0.0534	108	0.0503	0.0552	110	0.0506	0.0498	98.4
090901 BGF LCS Low 2	0.0497	0.0527	106	0.0503	0.0503	99.9	0.0506	0.0519	103
090901 BGF LCS Low 3	0.0497	0.0420	84.5	0.0503	0.0556	110	0.0506	0.0510	101
090901 BGF LCS Mid 1	0.150	0.137	91.5	0.152	0.135	89.1	0.152	0.152	100
090901 BGF LCS Mid 2	0.150	0.133	88.6	0.152	0.158	104	0.152	0.161	106
090901 BGF LCS Mid 3	0.150	0.145	96.8	0.152	0.150	98.7	0.152	0.154	101
090901 BGF LCS High 1	0.789	0.808	102	0.799	0.905	113	0.802	0.656	81.8
090901 BGF LCS High 2	0.789	0.741	93.9	0.799	0.884	111	0.802	0.810	101
090901 BGF LCS High 3	0.789	0.787	99.7	0.799	0.880	110	0.802	0.762	95.1
Average			96.8			105			98.6
%RSD			8.2			7.5			7.1

(1) LCS recovery exceeded 100±30%.

Table 23. LCS Recoveries for Extraction Batch 1 Using Extracted Matrix-Matched Calibration: PFBS, PFHS, and FOSA.

Sample ID	PFBS			PFHS			FOSA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.0496	0.0518	104	0.0516	0.0472	91.5	0.0492	0.0473	96.1
090826 BGF LCS Low 2	0.0496	0.0581	117	0.0516	0.0559	108	0.0492	0.0496	101
090826 BGF LCS Low 3	0.0496	0.0473	95.4	0.0516	0.0519	101	0.0492	0.0469	95.3
090826 BGF LCS Mid 1	0.150	0.122	81.0	0.156	0.151	96.6	0.148	0.141	95.1
090826 BGF LCS Mid 2	0.150	0.142	94.8	0.156	0.167	107	0.148	0.158	107
090826 BGF LCS Mid 3	0.150	0.140	93.4	0.156	0.144	92.4	0.148	0.139	94.2
090826 BGF LCS High 1	0.788	0.805	102	0.819	0.819	100	0.781	0.756	96.8
090826 BGF LCS High 2	0.788	0.711	90.3	0.819	0.708	86.5	0.781	0.782	100
090826 BGF LCS High 3	0.788	0.781	99.2	0.819	0.852	104	0.781	0.777	99.5
Average			97.5			98.6			98.3
%RSD			10			7.5			4.1

Table 24. LCS Recoveries for Extraction Batch 2 Using Extracted Matrix-Matched Calibration: PFBS, PFHS, and FOSA.

Sample ID	PFBS			PFHS			FOSA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.0496	0.0477	96.2	0.0516	0.0438	84.9	0.0492	0.0558	113
090828 BGF LCS Low 2	0.0496	0.0474	95.6	0.0516	0.0469	90.8	0.0492	0.0573	116
090828 BGF LCS Low 3	0.0496	0.0390	78.6	0.0516	0.0541	105	0.0492	0.0582	118
090828 BGF LCS Mid 1	0.150	0.147	97.7	0.156	0.139	89.1	0.148	0.119	80.4
090828 BGF LCS Mid 2	0.150	0.162	108	0.156	0.148	94.6	0.148	0.147	99.3
090828 BGF LCS Mid 3	0.150	0.156	104	0.156	0.183	117	0.148	0.159	107
090828 BGF LCS High 1	0.788	0.823	104	0.819	0.873	107	0.781	0.967	126
090828 BGF LCS High 2	0.788	0.713	90.5	0.819	0.925	113	0.781	1.00	128
090828 BGF LCS High 3	0.788	0.809	103	0.819	0.860	105	0.781	0.750	96.0
Average			97.5			101			109
%RSD			9.1			11			14

Table 25. LCS Recoveries for Extraction Batch 3 Using Extracted Matrix-Matched Calibration: PFBS, PFHS, and FOSA.

Sample ID	PFBS			PFHS			FOSA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.0496	0.0468	94.3	0.0516	0.0400	77.6	0.0492	0.0580	118
090901 BGF LCS Low 2	0.0496	0.0588	119	0.0516	0.0289	(1)56.1	0.0492	0.0506	103
090901 BGF LCS Low 3	0.0496	0.0386	77.7	0.0516	0.0342	(1)66.2	0.0492	0.0519	105
090901 BGF LCS Mid 1	0.150	0.154	103	0.156	0.144	92.0	0.148	0.136	92.1
090901 BGF LCS Mid 2	0.150	0.172	115	0.156	0.138	88.5	0.148	0.157	106
090901 BGF LCS Mid 3	0.150	0.172	115	0.156	0.132	84.8	0.148	0.139	93.6
090901 BGF LCS High 1	0.788	0.807	102	0.819	0.835	102	0.781	0.835	107
090901 BGF LCS High 2	0.788	0.833	106	0.819	0.773	94.4	0.781	0.922	118
090901 BGF LCS High 3	0.788	0.771	97.8	0.819	0.891	109	0.781	0.846	108
Average			103			85.6			106
%RSD			12			20			8.5

(1) LCS recovery exceeded 100±30%.

Table 26. LCS Recoveries for Extraction Batch 1 Using Extracted Matrix-Matched Calibration: Surrogates.

Sample ID	[1,2- ¹³ C ₂]PFOA			[¹⁸ O ₂]PFOS		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.104	0.102	97.8	0.104	0.101	97.5
090826 BGF LCS Low 2	0.104	0.113	109	0.104	0.109	104
090826 BGF LCS Low 3	0.104	0.102	98.1	0.104	0.124	119
090826 BGF LCS Mid 1	0.104	0.113	109	0.104	0.117	113
090826 BGF LCS Mid 2	0.104	0.111	107	0.104	0.105	101
090826 BGF LCS Mid 3	0.104	0.107	103	0.104	0.100	96.2
090826 BGF LCS High 1	0.104	0.115	110	0.104	0.097	93.3
090826 BGF LCS High 2	0.104	0.111	106	0.104	0.0968	93.1
090826 BGF LCS High 3	0.104	0.106	101	0.104	0.106	101
090826 BGF ECF LCS 1	0.104	0.126	121	0.104	0.110	106
090826 BGF ECF LCS 2	0.104	0.125	120	0.104	0.112	107
090826 BGF ECF LCS 3	0.104	0.124	119	0.104	0.109	105
090826 BGF PFOS LCS 1 @100 ng/g	0.104	0.111	107	0.104	0.0914	87.9
090826 BGF PFOS LCS 2 @100 ng/g	0.104	0.106	102	0.104	0.104	99.6
090826 BGF PFOS LCS 3 @100 ng/g	0.104	0.116	111	0.104	0.108	104
Average			108			102
%RSD			6.8			7.8

Table 27. LCS Recoveries for Extraction Batch 2 Using Extracted Matrix-Matched Calibration: Surrogates.

Sample ID	[1,2- ¹³ C ₂]PFOA			[¹⁸ O ₂]PFOS		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.104	0.127	122	0.104	0.111	106
090828 BGF LCS Low 2	0.104	0.0918	88.2	0.104	0.113	109
090828 BGF LCS Low 3	0.104	0.127	123	0.104	0.119	114
090828 BGF LCS Mid 1	0.104	0.0936	90.0	0.104	0.106	102
090828 BGF LCS Mid 2	0.104	0.115	111	0.104	0.102	98.5
090828 BGF LCS Mid 3	0.104	0.0969	93.2	0.104	0.0975	93.7
090828 BGF LCS High 1	0.104	0.128	123	0.104	0.108	102
090828 BGF LCS High 2	0.104	0.124	119	0.104	0.112	107
090828 BGF LCS High 3	0.104	0.111	107	0.104	0.111	107
090828 BGF ECF LCS 1	0.104	0.0961	92.4	0.104	0.122	118
090828 BGF ECF LCS 2	0.104	0.0866	83.2	0.104	0.115	110
090828 BGF ECF LCS 3	0.104	0.112	108	0.104	0.126	121
090828 BGF PFOS LCS 1 @100 ng/g	0.104	0.108	104	0.104	0.119	114
090828 BGF PFOS LCS 2 @100 ng/g	0.104	0.117	112	0.104	0.111	107
090828 BGF PFOS LCS 3 @100 ng/g	0.104	0.118	113	0.104	0.122	117
Average			106			108
%RSD			13			7.0

Table 28. LCS Recoveries for Extraction Batch 3 Using Extracted Matrix-Matched Calibration: Surrogates.

Sample ID	[1,2- ¹³ C ₂]PFOA			[¹⁸ O ₂]PFOS		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.104	0.109	105	0.104	0.0946	91.0
090901 BGF LCS Low 2	0.104	0.114	110	0.104	0.116	112
090901 BGF LCS Low 3	0.104	0.115	111	0.104	0.118	114
090901 BGF LCS Mid 1	0.104	0.106	102	0.104	0.126	121
090901 BGF LCS Mid 2	0.104	0.113	109	0.104	0.124	120
090901 BGF LCS Mid 3	0.104	0.103	98.8	0.104	0.105	101
090901 BGF LCS High 1	0.104	0.105	101	0.104	0.115	110
090901 BGF LCS High 2	0.104	0.125	120	0.104	0.106	102
090901 BGF LCS High 3	0.104	0.0970	93.3	0.104	0.112	107
090901 BGF ECF LCS 1	0.104	0.119	115	0.104	0.102	98.0
090901 BGF ECF LCS 2	0.104	0.112	108	0.104	0.123	118
090901 BGF ECF LCS 3	0.104	0.105	101	0.104	0.104	99.8
090901 BGF PFOS LCS 1 @100 ng/g	0.104	0.104	100	0.104	0.128	123
090901 BGF PFOS LCS 2 @100 ng/g	0.104	0.105	101	0.104	0.116	112
090901 BGF PFOS LCS 3 @100 ng/g	0.104	0.114	110	0.104	0.113	109
Average			106			109
%RSD			6.6			8.6

10.2 Lab Control Spike Recoveries by Unextracted Acetonitrile Calibration using IS Quantitation.

Table 29. LCS Recoveries for Extraction Batch 1 Using Unextracted Acetonitrile Calibration: PFBA, PFPeA, PFHxA.

Sample ID	PFBA (solvent curve quant)			PFPeA (solvent curve quant)			PFHxA (solvent curve quant)		
	⁽¹⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	⁽²⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Std. Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
D90826 BGF LCS Low 1	0.544	0.554	102	0.0686	0.0821	120	0.0502	0.0502	100
D90826 BGF LCS Low 2	0.531	0.549	103	0.0682	0.0848	124	0.0502	0.0496	98.8
D90826 BGF LCS Low 3	0.532	0.548	103	0.0682	0.0835	122	0.0502	0.0529	105
D90826 BGF LCS Mid 1	0.653	0.636	97.4	0.175	0.211	121	0.151	0.158	105
D90826 BGF LCS Mid 2	0.625	0.632	101	0.174	0.220	126	0.151	0.163	108
D90826 BGF LCS Mid 3	0.631	0.606	96.1	0.175	0.214	123	0.151	0.148	97.9
D90826 BGF LCS High 1	1.33	1.34	101	0.854	1.13	132	0.797	0.810	102
D90826 BGF LCS High 2	1.32	1.27	96.6	0.853	1.04	122	0.797	0.715	89.7
D90826 BGF LCS High 3	1.33	1.32	99.0	0.854	1.13	132	0.797	0.803	101
Average			100			125			101
%RSD			2.7			3.8			5.3

(1) Spike level of PFBA corrected for endogenous concentration of the bluegill fillet control matrix as determined by quantitation of the matrix blanks by the using the unextracted acetonitrile calibration (PFBA Conc = 4.61 ng/g).

(2) Spike level of PFPeA corrected for endogenous concentration of the bluegill fillet control matrix as determined by quantitation of the matrix blanks by the using the unextracted acetonitrile calibration (PFPeA Conc = 0.148 ng/g).

(3) LCS recovery exceeded 100±30%.

Table 30. LCS Recoveries for Extraction Batch 2 Using Unextracted Acetonitrile Calibration: PFBA, PFPeA, PFHxA.

Sample ID	PFBA (solvent curve quant)			PFPeA (solvent curve quant)			PFHxA (solvent curve quant)		
	⁽¹⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	⁽²⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Std. Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.521	0.498	95.6	0.128	0.106	83.1	0.0502	0.0609	121
090828 BGF LCS Low 2	0.540	0.528	97.8	0.128	0.0974	76.4	0.0502	0.0525	105
090828 BGF LCS Low 3	0.503	0.468	93.1	0.128	0.0990	77.6	0.0502	0.0640	127
090828 BGF LCS Mid 1	0.640	0.595	92.9	0.385	0.249	⁽³⁾ 64.7	0.151	0.156	103
090828 BGF LCS Mid 2	0.633	0.727	115	0.385	0.473	123	0.151	0.166	110
090828 BGF LCS Mid 3	0.646	0.635	98.4	0.385	0.256	⁽³⁾ 66.5	0.151	0.152	101
090828 BGF LCS High 1	1.30	1.28	98.2	2.03	1.24	⁽³⁾ 61.2	0.797	0.791	99.3
090828 BGF LCS High 2	1.32	1.32	99.7	2.03	1.31	⁽³⁾ 64.7	0.797	0.792	99.4
090828 BGF LCS High 3	1.31	1.16	88.3	2.03	1.15	⁽³⁾ 56.8	0.797	0.731	91.7
Average			97.6			74.9			106
%RSD			7.6			⁽⁴⁾ 27			11

(1) Spike level of PFBA corrected for endogenous concentration of the bluegill fillet control matrix as determined by quantitation of the matrix blanks by the using the unextracted acetonitrile calibration (PFBA Conc = 4.61 ng/g).

(2) Spike level of PFPeA corrected for endogenous concentration of the bluegill fillet control matrix as determined by quantitation of the matrix blanks by the using the unextracted acetonitrile calibration (PFPeA Conc = 0.275 ng/g).

(3) LCS recovery exceeded 100±30%.

(4) %RSD exceeded ±20%.

Table 31. LCS Recoveries for Extraction Batch 3 Using Unextracted Acetonitrile Calibration: PFBA, PFPeA, PFHxA.

Sample ID	PFBA (solvent curve quant)			PFPeA (solvent curve quant)			PFHxA (solvent curve quant)		
	⁽¹⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	⁽²⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	⁽³⁾ Corrected Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.528	0.486	92.0	0.0635	0.0586	89.1	0.0549	0.0566	103
090901 BGF LCS Low 2	0.561	0.542	96.6	0.0644	0.0574	89.2	0.0644	0.0574	89.2
090901 BGF LCS Low 3	0.571	0.507	88.8	0.0646	0.0538	83.2	0.0646	0.0538	83.2
090901 BGF LCS Mid 1	0.677	0.691	102	0.168	0.158	94.3	0.168	0.158	94.3
090901 BGF LCS Mid 2	0.649	0.633	97.5	0.167	0.165	98.9	0.167	0.165	98.9
090901 BGF LCS Mid 3	0.663	0.657	99.1	0.167	0.169	101	0.167	0.169	101
090901 BGF LCS High 1	1.36	1.34	98.3	0.824	0.792	96.1	0.824	0.792	96.1
090901 BGF LCS High 2	1.35	1.38	102	0.824	0.847	103	0.824	0.847	103
090901 BGF LCS High 3	1.35	1.26	93.7	0.823	0.767	93.2	0.823	0.767	93.2
Average			96.7			94.2			95.8
%RSD			4.6			6.7			6.9

(1) Spike level of PFBA corrected for endogenous concentration of the bluegill fillet control matrix as determined by quantitation of the matrix blanks by the using the unextracted acetonitrile calibration (PFBA Conc = 4.91 ng/g).

(2) Spike level of PFPeA corrected for endogenous concentration of the bluegill fillet control matrix as determined by quantitation of the matrix blanks by the using the unextracted acetonitrile calibration (PFPeA Conc = 0.129 ng/g).

(3) Spike level of PFHxA corrected for endogenous concentration of the bluegill fillet control matrix as determined by quantitation of the matrix blanks by the using the unextracted acetonitrile calibration (PFHxA Conc = 0.0482 ng/g).

Table 32. LCS Recoveries for Extraction Batch 1 Using Unextracted Acetonitrile Calibration: PFCAs (C7-C12).

Sample ID	PFHpA			PFOA			PFNA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.0492	0.0502	102	0.0516	0.0455	88.1	0.0515	0.0455	88.3
090826 BGF LCS Low 2	0.0492	0.0540	110	0.0516	0.0536	104	0.0515	0.0440	85.5
090826 BGF LCS Low 3	0.0492	0.0485	98.6	0.0516	0.0524	102	0.0515	0.0437	84.8
090826 BGF LCS Mid 1	0.148	0.146	98.8	0.156	0.153	98.2	0.155	0.148	95.2
090826 BGF LCS Mid 2	0.148	0.169	114	0.156	0.170	109	0.155	0.163	105
090826 BGF LCS Mid 3	0.148	0.147	99.6	0.156	0.150	96.2	0.155	0.150	96.5
090826 BGF LCS High 1	0.781	0.797	102	0.819	0.815	100	0.818	0.906	111
090826 BGF LCS High 2	0.781	0.735	94.1	0.819	0.730	89.1	0.818	0.818	100
090826 BGF LCS High 3	0.781	0.720	92.3	0.819	0.708	86.4	0.818	0.816	99.7
090826 BGF ECF LCS 1				0.520	0.520	99.9			
090826 BGF ECF LCS 2				0.520	0.531	102			
090826 BGF ECF LCS 3				0.520	0.546	105			
Average			101			97.0			96.2
%RSD			6.9			8.0			9.2
Sample ID	PFDA			PFUnA			PFDoA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.0497	0.0437	87.9	0.0503	0.0554	110	0.0506	0.0485	95.8
090826 BGF LCS Low 2	0.0497	0.0452	90.9	0.0503	0.0549	109	0.0506	0.0438	86.6
090826 BGF LCS Low 3	0.0497	0.0435	87.5	0.0503	0.0587	117	0.0506	0.0483	95.4
090826 BGF LCS Mid 1	0.150	0.142	94.4	0.152	0.156	103	0.152	0.155	102
090826 BGF LCS Mid 2	0.150	0.149	99.1	0.152	0.149	97.8	0.152	0.160	105
090826 BGF LCS Mid 3	0.150	0.157	104	0.152	0.152	100	0.152	0.148	97.2
090826 BGF LCS High 1	0.789	0.817	104	0.799	0.844	106	0.802	0.774	96.5
090826 BGF LCS High 2	0.789	0.724	91.8	0.799	0.716	89.6	0.802	0.769	95.9
090826 BGF LCS High 3	0.789	0.803	102	0.799	0.804	101	0.802	0.779	97.1
Average			95.7			104			96.8
%RSD			7.0			7.7			5.2

Table 33. LCS Recoveries for Extraction Batch 2 Using Unextracted Acetonitrile Calibration: PFCAs (C7-C12).

Sample ID	PFHpA			PFOA			PFNA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.0492	0.0494	100	0.0516	0.0785	(1)152	0.0515	0.0429	83.2
090828 BGF LCS Low 2	0.0492	0.0344	(1)69.9	0.0516	0.0684	129	0.0515	0.0433	84.0
090828 BGF LCS Low 3	0.0492	0.0381	77.4	0.0516	0.0562	109	0.0515	0.0525	102
090828 BGF LCS Mid 1	0.148	0.131	88.8	0.156	0.134	86.1	0.155	0.153	98.7
090828 BGF LCS Mid 2	0.148	0.165	112	0.156	0.182	116	0.155	0.165	106
090828 BGF LCS Mid 3	0.148	0.123	82.8	0.156	0.149	95.7	0.155	0.163	105
090828 BGF LCS High 1	0.781	0.844	108	0.819	0.941	115	0.818	0.871	106
090828 BGF LCS High 2	0.781	0.858	110	0.819	0.814	99.4	0.818	0.814	99.5
090828 BGF LCS High 3	0.781	0.654	83.7	0.819	0.718	87.7	0.818	0.826	101
090828 BGF ECF LCS 1				0.520	0.414	79.6			
090828 BGF ECF LCS 2				0.520	0.387	74.4			
090828 BGF ECF LCS 3				0.520	0.492	94.5			
Average			92.5			110			98.4
%RSD			17			19			8.9
Sample ID	PFDA			PFUnA			PFDoA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.0492	0.0494	100	0.0503	0.0617	123	0.0506	0.0532	105
090828 BGF LCS Low 2	0.0492	0.0344	69.9	0.0503	0.0749	(1)149	0.0506	0.0480	94.9
090828 BGF LCS Low 3	0.0492	0.0381	77.4	0.0503	0.0734	(1)146	0.0506	0.0502	98.3
090828 BGF LCS Mid 1	0.148	0.131	88.8	0.152	0.135	88.9	0.152	0.160	105
090828 BGF LCS Mid 2	0.148	0.165	112	0.152	0.174	115	0.152	0.140	91.8
090828 BGF LCS Mid 3	0.148	0.123	82.8	0.152	0.158	104	0.152	0.171	113
090828 BGF LCS High 1	0.781	0.844	108	0.799	0.833	104	0.802	0.907	113
090828 BGF LCS High 2	0.781	0.858	110	0.799	0.987	124	0.802	0.833	104
090828 BGF LCS High 3	0.781	0.654	83.7	0.799	0.779	97.4	0.802	0.813	101
Average			92.5			117			103
%RSD			17			18			7.0

(1) LCS Recovery exceeded 100±30%.

Table 34. LCS Recoveries for Extraction Batch 2 Using Unextracted Acetonitrile Calibration: PFCAs (C7-C12).

Sample ID	PFHpA			PFOA			PFNA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.0492	0.0387	78.8	0.0516	0.0489	94.8	0.0515	0.0515	100
090901 BGF LCS Low 2	0.0492	0.0296	59.2	0.0516	0.0481	93.3	0.0515	0.0469	91.0
090901 BGF LCS Low 3	0.0492	0.0347	70.5	0.0516	0.0557	108	0.0515	0.0518	101
090901 BGF LCS Mid 1	0.148	0.107	72.6	0.156	0.139	89.0	0.155	0.146	94.0
090901 BGF LCS Mid 2	0.148	0.118	79.7	0.156	0.148	94.8	0.155	0.169	109
090901 BGF LCS Mid 3	0.148	0.106	71.7	0.156	0.145	92.9	0.155	0.157	101
090901 BGF LCS High 1	0.781	0.553	70.8	0.819	0.786	96.0	0.818	0.888	109
090901 BGF LCS High 2	0.781	0.644	82.5	0.819	0.886	108	0.818	0.904	111
090901 BGF LCS High 3	0.781	0.562	72.0	0.819	0.718	87.6	0.818	0.773	94.5
090901 BGF ECF LCS 1				0.520	0.470	90.4			
090901 BGF ECF LCS 2				0.520	0.479	92.0			
090901 BGF ECF LCS 3				0.520	0.483	92.9			
Average			73.2			96.0			101
%RSD			9.0			7.6			7.2
Sample ID	PFDA			PFUnA			PFDoA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.0497	0.0543	109	0.0503	0.0591	117	0.0506	0.0496	98.1
090901 BGF LCS Low 2	0.0497	0.0535	108	0.0503	0.0545	108	0.0506	0.0520	103
090901 BGF LCS Low 3	0.0497	0.0424	85.4	0.0503	0.0594	118	0.0506	0.0510	101
090901 BGF LCS Mid 1	0.150	0.141	94.0	0.152	0.133	87.3	0.152	0.167	110
090901 BGF LCS Mid 2	0.150	0.137	91.0	0.152	0.153	101	0.152	0.178	117
090901 BGF LCS Mid 3	0.150	0.149	99.5	0.152	0.146	96.1	0.152	0.169	111
090901 BGF LCS High 1	0.789	0.839	106	0.799	0.845	106	0.802	0.746	93.1
090901 BGF LCS High 2	0.789	0.769	97.4	0.799	0.825	103	0.802	0.923	115
090901 BGF LCS High 3	0.789	0.816	103	0.799	0.822	103	0.802	0.868	108
Average			99.3			104			106
%RSD			8.1			9.2			7.5

(1) LCS recovery exceeded 100±30%.

Table 35. LCS Recoveries for Extraction Batch 1 Using Unextracted Acetonitrile Calibration: PFBS, PFHS, and FOSA.

Sample ID	PFBS			PFHS			FOSA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.0496	0.0408	82.2	0.0516	0.0437	84.7	0.0492	0.0455	92.4
090826 BGF LCS Low 2	0.0496	0.0473	95.3	0.0516	0.0529	103	0.0492	0.0478	97.2
090826 BGF LCS Low 3	0.0496	0.0362	73.0	0.0516	0.0486	94.2	0.0492	0.0451	91.7
090826 BGF LCS Mid 1	0.150	0.112	75.0	0.156	0.153	98.2	0.148	0.140	94.6
090826 BGF LCS Mid 2	0.150	0.134	89.1	0.156	0.171	109	0.148	0.157	106
090826 BGF LCS Mid 3	0.150	0.131	87.6	0.156	0.146	93.7	0.148	0.139	93.7
090826 BGF LCS High 1	0.788	0.813	103	0.819	0.859	105	0.781	0.764	97.8
090826 BGF LCS High 2	0.788	0.717	90.9	0.819	0.743	90.7	0.781	0.790	101
090826 BGF LCS High 3	0.788	0.788	100	0.819	0.894	109	0.781	0.785	101
Average			88.5			98.6			97.3
%RSD			12			8.6			4.9

Table 36. LCS Recoveries for Extraction Batch 2 Using Unextracted Acetonitrile Calibration: PFBS, PFHS, and FOSA.

Sample ID	PFBS			PFHS			FOSA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.0496	0.0390	78.5	0.0516	0.0398	77.2	0.0492	0.0632	129
090828 BGF LCS Low 2	0.0496	0.0387	78.0	0.0516	0.0425	82.3	0.0492	0.0849	(1)132
090828 BGF LCS Low 3	0.0496	0.0310	(1)62.5	0.0516	0.0488	94.5	0.0492	0.0658	(1)134
090828 BGF LCS Mid 1	0.150	0.128	85.6	0.156	0.123	78.9	0.148	0.133	89.7
090828 BGF LCS Mid 2	0.150	0.143	95.0	0.156	0.131	83.7	0.148	0.164	111
090828 BGF LCS Mid 3	0.150	0.137	91.4	0.156	0.162	104	0.148	0.176	119
090828 BGF LCS High 1	0.788	0.745	94.6	0.819	0.777	94.9	0.781	1.09	(1)140
090828 BGF LCS High 2	0.788	0.645	81.8	0.819	0.825	101	0.781	1.11	(1)142
090828 BGF LCS High 3	0.788	0.732	92.9	0.819	0.766	93.5	0.781	0.828	106
			84.5			90.0			123
			13			11			14

(1) LCS recovery exceeded 100±30%.

Table 37. LCS Recoveries for Extraction Batch 3 Using Unextracted Acetonitrile Calibration: PFBS, PFHS, and FOSA.

Sample ID	PFBS			PFHS			FOSA		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.0496	0.0412	83.0	0.0516	<0.0102	(1)N/A	0.0492	0.0538	109
090901 BGF LCS Low 2	0.0496	0.0530	107	0.0516	<0.0102	(1)N/A	0.0492	0.0465	94.5
090901 BGF LCS Low 3	0.0496	0.0331	(2)66.7	0.0516	<0.0102	(1)N/A	0.0492	0.0478	97.1
090901 BGF LCS Mid 1	0.150	0.146	97.6	0.156	0.104	(2)66.8	0.148	0.131	88.2
090901 BGF LCS Mid 2	0.150	0.164	109	0.156	0.0986	(2)63.2	0.148	0.151	102
090901 BGF LCS Mid 3	0.150	0.164	110	0.156	0.0925	(2)59.3	0.148	0.133	89.8
090901 BGF LCS High 1	0.788	0.787	99.9	0.819	0.815	99.5	0.781	0.818	105
090901 BGF LCS High 2	0.788	0.813	103	0.819	0.751	91.7	0.781	0.904	116
090901 BGF LCS High 3	0.788	0.752	95.4	0.819	0.872	107	0.781	0.829	106
			96.8			81.3			101
			14			(3)25			9.1

(1) Recovery not calculated. Sample result was below the LOQ.

(2) LCS recovery exceeded 100±30%.

(3) %RSD exceeded ±20%.

Table 38. LCS Recoveries for Extraction Batch 1 Using Unextracted Acetonitrile Calibration: Surrogates.

Sample ID	[1,2- ¹³ C ₂]PFOA			[¹⁸ O ₂]PFOS		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090826 BGF LCS Low 1	0.104	0.0947	91.1	0.104	0.104	100
090826 BGF LCS Low 2	0.104	0.105	101	0.104	0.112	108
090826 BGF LCS Low 3	0.104	0.095	91.3	0.104	0.128	123
090826 BGF LCS Mid 1	0.104	0.106	102	0.104	0.121	117
090826 BGF LCS Mid 2	0.104	0.103	99.2	0.104	0.108	104
090826 BGF LCS Mid 3	0.104	0.1	96.1	0.104	0.103	99
090826 BGF LCS High 1	0.104	0.107	103	0.104	0.0997	95.9
090826 BGF LCS High 2	0.104	0.103	98.1	0.104	0.0995	95.7
090826 BGF LCS High 3	0.104	0.0982	94.4	0.104	0.109	105
090826 BGF ECF LCS 1	0.104	0.117	113	0.104	0.114	110
090826 BGF ECF LCS 2	0.104	0.116	112	0.104	0.115	111
090826 BGF ECF LCS 3	0.104	0.116	111	0.104	0.112	108
090826 BGF PFOS LCS 1 @100 ng/g	0.104	0.103	99.2	0.104	0.0938	90.2
090826 BGF PFOS LCS 2 @100 ng/g	0.104	0.0991	95.2	0.104	0.107	103
090826 BGF PFOS LCS 3 @100 ng/g	0.104	0.108	104	0.104	0.112	107
Average			101			105
%RSD			6.9			8.1

Table 39. LCS Recoveries for Extraction Batch 2 Using Unextracted Acetonitrile Calibration: Surrogates.

Sample ID	[1,2- ¹³ C ₂]PFOA			[¹⁸ O ₂]PFOS		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090828 BGF LCS Low 1	0.104	0.121	116	0.104	0.0984	94.6
090828 BGF LCS Low 2	0.104	0.0878	84.4	0.104	0.101	96.9
090828 BGF LCS Low 3	0.104	0.121	116	0.104	0.106	102
090828 BGF LCS Mid 1	0.104	0.0895	86.1	0.104	0.0944	90.7
090828 BGF LCS Mid 2	0.104	0.110	106	0.104	0.0907	87.2
090828 BGF LCS Mid 3	0.104	0.0926	89.0	0.104	0.0860	82.7
090828 BGF LCS High 1	0.104	0.121	117	0.104	0.0940	90.4
090828 BGF LCS High 2	0.104	0.118	113	0.104	0.0982	95.4
090828 BGF LCS High 3	0.104	0.106	102	0.104	0.099	95.2
090828 BGF ECF LCS 1	0.104	0.0918	88.3	0.104	0.109	105
090828 BGF ECF LCS 2	0.104	0.0830	79.8	0.104	0.102	98.2
090828 BGF ECF LCS 3	0.104	0.107	116	0.104	0.113	109
090828 BGF PFOS LCS 1	1.104	0.103	84.4	0.104	0.106	102
090828 BGF PFOS LCS 2	2.104	0.111	116	0.104	0.0987	94.9
090828 BGF PFOS LCS 3	3.104	0.112	86.1	0.104	0.109	105
Average			100			96.6
%RSD			15			7.4

Table 40. LCS Recoveries for Extraction Batch 3 Using Unextracted Acetonitrile Calibration: Surrogates.

Sample ID	[1,2- ¹³ C ₂]PFOA			[¹⁸ O ₂]PFOS		
	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	% Recovery
090901 BGF LCS Low 1	0.104	0.104	99.9	0.104	0.0906	87.1
090901 BGF LCS Low 2	0.104	0.109	105	0.104	0.111	107
090901 BGF LCS Low 3	0.104	0.109	105	0.104	0.114	109
090901 BGF LCS Mid 1	0.104	0.101	97.2	0.104	0.121	116
090901 BGF LCS Mid 2	0.104	0.107	103	0.104	0.120	115
090901 BGF LCS Mid 3	0.104	0.0981	94.4	0.104	0.100	96.6
090901 BGF LCS High 1	0.104	0.101	96.8	0.104	0.110	106
090901 BGF LCS High 2	0.104	0.119	114	0.104	0.102	97.9
090901 BGF LCS High 3	0.104	0.0929	89.3	0.104	0.107	103
090901 BGF ECF LCS 1	0.104	0.113	109	0.104	0.0977	94
090901 BGF ECF LCS 2	0.104	0.106	102	0.104	0.118	113
090901 BGF ECF LCS 3	0.104	0.0999	96.0	0.104	0.0995	95.7
090901 BGF PFOS LCS 1	0.104	0.0988	95.0	0.104	0.123	118
090901 BGF PFOS LCS 2	0.104	0.100	96.2	0.104	0.111	107
090901 BGF PFOS LCS 3	0.104	0.108	104	0.104	0.108	104
Average			100			105
%RSD			6.4			8.6

10.3 Surrogate Recoveries in Spiked Blanks (Extracted Curve Calibration).

Extraction Batch 1					
Sample ID	Sample Description	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	%Rec	[¹⁸ O ₂]PFOS Spike Conc. (ng/mL) Calc. Conc. (ng/mL) %Rec
090826 BGF Matrix Blank-1	(with IS/surr)	0.104	0.113	109	0.104 0.112 108
090826 BGF Matrix Blank-2	(with IS/surr)	0.104	0.112	108	0.104 0.121 116
090826 Aqueous Blank-1	(with IS/surr)	0.104	0.0980	94.2	0.104 0.0810 77.9
090826 Aqueous Blank-2	(with IS/surr)	0.104	0.113	109	0.104 0.110 106
Acetonitrile Blank with IS & Surr	08 007-145	0.0998	0.104	104	0.0956 0.0995 104
Acetonitrile Blank with IS & Surr	08 007-145	0.0998	0.125	125	0.0956 0.0948 99.2

Extraction Batch 2					
Sample ID	Sample Description	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	%Rec	[¹⁸ O ₂]PFOS Spike Conc. (ng/mL) Calc. Conc. (ng/mL) %Rec
090826 BGF Matrix Blank-1	(with IS/surr)	0.104	0.114	110	0.104 0.101 97.1
090826 BGF Matrix Blank-2	(with IS/surr)	0.104	0.103	99.0	0.104 0.107 103
090826 Aqueous Blank-1	(with IS/surr)	0.104	0.103	99.0	0.104 0.0998 96.0
090826 Aqueous Blank-2	(with IS/surr)	0.104	0.102	98.1	0.104 0.118 113
Acetonitrile Blank with IS & Surr.	08 007-145	0.0998	0.0929	93.1	0.0956 0.109 114
Acetonitrile Blank with IS & Surr.	08 007-145	0.0998	0.0793	79.5	0.0956 0.125 131

Extraction Batch 3					
Sample ID	Sample Description	Spike Conc. (ng/mL)	Calc. Conc. (ng/mL)	%Rec	[¹⁸ O ₂]PFOS Spike Conc. (ng/mL) Calc. Conc. (ng/mL) %Rec
090901 BGF Matrix Blank-1	(with IS/surr)	0.104	0.102	98.1	0.104 0.105 101
090901 BGF Matrix Blank-2	(with IS/surr)	0.104	0.0990	95.2	0.104 0.107 103
090901 Aqueous Blank-1	(with IS/surr)	0.104	0.108	104	0.104 0.107 103
090901 Aqueous Blank-2	(with IS/surr)	0.104	0.108	104	0.104 0.104 100
Acetonitrile Blank with IS & Surrogate	08 007-145	0.0998	0.107	107	0.0956 0.101 106
Acetonitrile Blank with IS & Surrogate	08 007-145	0.0998	0.110	110	0.0956 0.106 111

(1) Surrogate recovery exceeded 100±30%.