UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 4
ATLANTA FEDERAL CENTER
61 FORSYTH STREET
ATLANTA, GEORGIA 30303-8960

OCT 13 2009

MEMORANDUM

SUBJECT: Dalton Utilities Submittal – Analytical Report: Fluorochemical Characterization of Aqueous and Solid Samples; MPI Report No. L0018099; dated June 29, 2009

FROM: Connie L. Roberts CLR
Special Assistant to the Director
Water Protection Division

TO: Note to File

Attached is a summary of the document titled: "Analytical Report for Fluorochemical Characterization of Aqueous and Solid Samples, MPI Report No. L0018099" dated June 29, 2009 submitted to EPA Region 4 by Dalton Utilities on July 20, 2009. This information was submitted as part of Dalton Utilities' response to EPA's May 20, 2009, Section 308 of the Clean Water Act request addressed to Mr. Don Cope, President and CEO of Dalton Utilities.

The summary provides analytical results for water and solid samples taken by Dalton Utilities at its Loopers Bend Wastewater Land Application Site, Dalton, Georgia. Groundwater, wastewater effluent, surface water, soils, compost and sludge samples were analyzed as part of this investigation.

The following abbreviations are used in the document:

- MW = monitoring well [used for groundwater sampling; monitoring wells are not used as drinking water sources]
- SP = sprayfield [used for wastewater effluent sampling]
- River = Conasauga River or Holly Creek [used for surface water sampling; Holly Creek is tributary to the Conasauga River]

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Analytical Report

Fluorochemical Characterization of Aqueous and Solid Samples

MPI Report No. L0018099

Testing Laboratory

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1 Introduction

Results are reported for the analysis of water and solid samples received at MPI Research from Dalton Utilities. The MPI Research study number assigned to the project is L0018099. Table I lists the target analytes quantitated for the samples.

Table I. Target Analytes for Quantitation

Compound Name	Acronym
Perfluorobutyric Acid	C4 Acid
Perfluoropentanoic Acid	C5 Acid
Perfluorohexanoic Acid	C6 Acid
Perfluoroheptanoic Acid	C7 Acid
Perfluorooctanoic Acid	C8 Acid
Perfluorononanoic Acid	C9 Acid
Perfluorodecanoic Acid	C10 Acid
Perfluoroundecanoic Acid	C11 Acid
Perfluorododecanoic Acid	C12 Acid
Perfluorotridecanoic Acid	C13 Acid
Perfluorotetradecanoic Acid	C14 Acid
Perfluorobutanesulfonate	C4 Sulfonate or PFBS
Perfluorohexanesulfonate	C6 Sulfonate or PFHS
Perfluorooctanesulfonate	C8 Sulfonate or PFOS
Perfluorooctanesulfonamide	FOSA

2 Sample Receipt

A total of sixty samples were received from David White at Dalton Utilities for this study. The samples were collected between May 26, 2009 and May 28, 2009. The samples arrived on May 29, 2009 via FED Ex and were logged in under MPI Research login number L0018099. The shipment was received cooled with wet ice. The samples were stored refrigerated from receipt until analysis. Chain-of-custody information is presented in Attachment A.

3 Methods - Analytical and Preparatory

3.1 Water Sample Preparation

Ten milliliters of sample was transferred into a 50 mL centrifuge tube. Samples designated as lab spikes were fortified appropriately with analyte and surrogate. All samples were fortified with a 50 µL portion of a 100 ng/mL surrogate spiking solution containing PFOA (m+4). Ten milliliters of acetonitrile was added to the sample. After shaking, the sample was sonicated for approximately 2 hours then centrifuged at 3000 rpm for ~10 minutes. A 1 mL portion of the

supernatant was transferred to an autosampler vial and fortified with 20 µL of a 25 ng/mL internal standard solution. The samples were then analyzed using electrospray LC/MS/MS.

3.2 Solid Sample Preparation

One gram of solid was measured into a 50 mL centrifuge tube. Samples designated as lab spikes were fortified appropriately with analyte and surrogate. All samples were fortified with a 40 µL portion of a 100 ng/mL surrogate spiking solution of PFOA (m+4). Eight milliliters of 80:20 acetonitrile: water was added to the sample. After shaking, the sample was sonicated for approximately 2 hours then centrifuged at 3000 rpm for 10 minutes. A 1 mL portion of the supernatant was transferred to an autosampler vial and fortified with 20 µL of a 25 ng/mL internal standard solution. The samples were then analyzed using electrospray LC/MS/MS.

3.3 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (HPLC), an aliquot of extract is injected and passed through a liquid-phase chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. Following HPLC separation, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against calibration standards.

An HP1100 system interfaced to an Applied Biosystems API 5000 LC/MS/MS was used to analyze the sample extracts for quantitation. A gradient elution through a Phenomenex Luna 3µ C8(2) Mercury, 20 x 4.0 mm column was used for separation.

The following gradient was performed:

Mobile Phase (A): 2mM Ammonium Acetate in Water
Mobile Phase (B): Methanol

Time	%A	%B
0.0	90	10
0.5	90	10
2.0	10	90
5.0	10	90
5.1	0	100
6.0	0	100
6.1	90	10
10.0	90	10

The following parameters were used for operation of the mass spectrometer:

Parameter	Setting
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	213→169 (C4 Acid)
	263→219 (C5 Acid)
	313→269 (C6 Acid)
	363→319 (C7 Acid)
	413→369 (C8 Acid)
	463→419 (C9 Acid)
	513→469 (C10 Acid)
	563→519 (C11 Acid)
	613→569 (C12 Acid)
	663→619 (C13 Acid)
	713→669 (C14 Acid)
	299→80 (PFBS)
	399→80 (PFHS)
	499→80 (PFOS)
	498→78 (FOSA)
	415→370 (Internal Std. ^{13}C PFOA (m+2))
	417→372 (Surrogate ^{13}C PFOA (m+4))
Gas Temperature	450°C

4 Analysis by LCMSMS

4.1 Calibration

For the water sample analysis, a 9-point calibration curve was analyzed throughout the analytical sequence for all compounds of interest. The calibration points were prepared at 0.0125, 0.025, 0.050, 0.100, 0.250, 0.500, 1.0, 2.5 and 5.0 ng/mL (ppb) each containing 0.5 ng/mL ^{13}C-PFOA (m+2). For the solid sample analysis, an 8-point calibration curve was analyzed throughout the analytical sequence for all compounds of interest. The calibration points were prepared at 0.025, 0.050, 0.100, 0.250, 0.500, 1.0, 2.5 and 5.0 ng/mL (ppb) each containing 0.5 ng/mL ^{13}C-PFOA (m+2). Standard preparation details can be found in Attachment D.

The ratio of the analyte concentration to the IS concentration versus the ratio of the analyte instrument response (area) to the IS response (area) was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r^2 \geq 0.985$.

For the results reported here, calibration criteria were met. The calibration curves are included in the raw data in Attachment C.

4.2 Surrogates

^{13}C labeled-perfluorooctanoic acid (^{13}C PFOA (m+4)) is used as a surrogate for the water and solid samples.

^{13}C PFOA (m+4) recoveries can be found in Attachment B.

4.3 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to laboratory reagents and/or controls. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range. Laboratory control spike recoveries are given in Attachment B.

4.4 Matrix Spikes

Seven matrix spikes, five for water and two for solids, were prepared by adding a known concentration of the target analyte to a sample. Matrix spikes are used to assess method accuracy in the matrix. The matrix spikes should show recoveries between 70-130%. For the results reported here, the matrix spikes were within the acceptable range with the exceptions of:

L18099-19 (MW M10) Spk C at 0.5 ng/mL for C5 Acid, C7 Acid, C8 Acid, C10 Acid and C13 Acid, which gave high recoveries after two separate preparations.

L18099-32 (MW D6) Spk D at 0.5 ng/mL for C13 Acid, which gave high recoveries after two separate preparations.

L18099-41 (SP CA15) Spk C at 0.5 ng/mL for C6 Acid, and C8 Acid, which gave high recoveries after two separate preparations.

L18099-57 (River R1) Spk D at 0.5 ng/mL for C9 Acid, C12 Acid, and C13 Acid, which gave high recoveries after two separate preparations.

L18099-2 (AC 6 Soil) Spk C at 5.0 ng/mL for C11 Acid, which gave high recoveries after two separate preparations.

4.5 Laboratory Duplicates

Five water samples and two solid samples were prepared in duplicate and analyzed. Duplicate results are given along with the sample results in Attachment B.

5 Data Summary

Please see Attachment B for a detailed listing of the analytical results. For the water samples the results are reported in parts per billion (ng/mL) on an as-received basis. For the solid samples, the results are reported in parts per billion (ng/g), on a dry-weight basis.

6 Data/Sample Retention

Samples are disposed of 60 days after the report is issued unless otherwise specified by the project manager. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by MPI Research. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.

7 Attachments

7.1 Attachment A: Chain of Custody

7.2 Attachment B: Analytical Results

7.3 Attachment C: Raw Analytical Data for Water

7.4 Attachment D: Raw Analytical Data for Solids

8 Signatures

Mark Neeley, Research Chemist Associate II — 6-26-09 Date

Karen Risha, Manager, Analytical — 06/29/09 Date

Other Lab Members Contributing to Data:
Sarah Coghlan
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Analytical Report

Summary of Fluorochemical Residues in Water Samples

Sample ID	C4 Acid Perfluorobutyric Acid Analyte Found (ng/mL, ppb)	C5 Acid Perfluoropentanoic Acid Analyte Found (ng/mL, ppb)	C6 Acid Perfluorohexanoic Acid Analyte Found (ng/mL, ppb)	C7 Acid Perfluoroheptanoic Acid Analyte Found (ng/mL, ppb)	C8 Acid Perfluorooctanoic Acid Analyte Found (ng/mL, ppb)
MW M10	ND	0.0373	0.0498	ND	ND
MW M10*	ND	0.0586	0.0614	NQ	ND
MW M11	0.173	0.427	0.574	0.154	0.287
MW M9	1.01	4.58	2.44	0.564	0.512
MW D2	0.275	0.909	0.713	0.472	1.10
MW M1	0.0993	0.411	0.426	0.295	0.604
MW U1	ND	0.0278	NQ	ND	ND
MW D3	0.251	0.664	0.380	0.161	0.228
MW D1	0.0950	0.434	0.487	0.387	1.21
MW M5	0.529	1.90	1.98	1.35	2.87
MW M6A	0.0515	0.171	0.148	0.145	0.331
MW M17	1.38	3.10	2.36	2.01	4.40
MW M14	0.681	3.28	3.01	1.50	2.44
MW M13	0.931	3.62	3.42	2.45	4.41
MW D6	0.550	1.92	1.680	1.13	2.73
MW D6*	0.447	1.68	1.640	1.01	2.60
MW D4	0.759	2.49	2.54	1.95	4.16
MW M3	0.0318	0.139	0.0613	ND	ND
MW M7	0.414	1.82	1.69	0.906	1.71
MW M2	0.959	2.13	0.397	NQ	ND
MW M8	0.381	0.885	0.868	0.421	1.08
MW M12	0.749	2.68	2.28	1.51	2.97
SP AC5	0.566	1.26	0.746	0.309	0.479
SP BA2	0.665	1.37	0.811	0.328	0.469
SP BA2*	0.742	1.47	0.963	0.341	0.539
SP CA15	0.684	1.45	0.723	0.261	0.509
SP CA15*	0.805	1.58	1.04	0.306	0.658
SP AC2	0.609	1.40	0.797	0.305	0.591
SP AC15	0.710	1.53	0.964	0.312	0.592
SP AC4	0.646	1.48	0.994	0.331	0.702
SP AC14	0.755	1.67	1.17	0.349	0.754
SP CB14A	0.869	1.77	0.935	0.314	0.692
SP CB12	0.751	1.43	0.749	0.274	0.531
SP BB9	0.785	1.50	0.763	0.260	0.568
SP BB13	0.761	1.52	0.767	0.270	0.565
SP CA2	0.776	1.43	0.709	0.275	0.527
SP CB3	0.903	1.69	0.878	0.316	0.615
SP BB12	0.998	1.80	1.13	0.358	0.755
SP BA4	0.839	1.57	0.824	0.305	0.566
MW D11	ND	ND	ND	ND	ND
MW D9	0.774	2.02	1.79	1.44	3.21
MW M4	0.530	2.50	2.25	1.48	3.89
River R1	ND	ND	ND	ND	ND
River R1*	ND	ND	ND	ND	ND
River R2	0.0494	0.184	0.188	0.112	0.358
River R3	ND	0.0386	ND	ND	0.0310
River R4	0.0468	0.195	0.170	0.0822	0.266

*Laboratory Duplicate
ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).
NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).

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Analytical Report

Summary of Fluorochemical Residues in Water Samples (continued)

Sample ID	C9 Acid Perfluorononanoic Acid Analyte Found (ng/mL, ppb)	C10 Acid Perfluorodecanoic Acid Analyte Found (ng/mL, ppb)	C11 Acid Perfluoroundecanoic Acid Analyte Found (ng/mL, ppb)	C12 Acid Perfluorododecanoic Acid Analyte Found (ng/mL, ppb)	C13 Acid Perfluorotridecanoic Acid Analyte Found (ng/mL, ppb)
MW M10	ND	ND	ND	ND	ND
MW M10*	ND	ND	ND	ND	ND
MW M11	0.0308	ND	ND	ND	ND
MW M9	ND	ND	ND	ND	ND
MW D2	0.131	0.0825	ND	ND	ND
MW M1	ND	ND	ND	ND	ND
MW U1	ND	ND	ND	ND	ND
MW D3	ND	ND	ND	ND	ND
MW D1	0.104	0.128	ND	ND	ND
MW M5	0.449	0.0578	ND	ND	ND
MW M6A	0.0730	NQ	ND	ND	ND
MW M17	1.17	0.856	0.137	ND	ND
MW M14	0.129	ND	ND	ND	ND
MW M13	0.811	0.120	ND	ND	ND
MW D6	0.575	0.211	ND	ND	ND
MW D6*	0.497	0.190	ND	ND	ND
MW D4	0.543	0.0966	ND	ND	ND
MW M3	ND	ND	ND	ND	ND
MW M7	0.0940	0.0978	ND	ND	ND
MW M2	ND	ND	ND	ND	ND
MW M8	0.108	0.106	ND	ND	ND
MW M12	0.452	0.167	ND	ND	ND
SP AC5	0.104	0.227	0.109	ND	ND
SP BA2	0.0652	0.162	0.107	ND	ND
SP BA2*	0.0764	0.173	0.117	ND	ND
SP CA15	0.113	0.200	0.118	ND	ND
SP CA15*	0.120	0.225	0.141	ND	ND
SP AC2	0.127	0.246	0.164	ND	NQ
SP AC15	0.0832	0.152	0.105	ND	ND
SP AC4	0.126	0.267	0.187	ND	NQ
SP AC14	0.137	0.233	0.172	ND	ND
SP CB14A	0.119	0.218	0.151	ND	ND
SP CB12	0.118	0.192	0.114	ND	ND
SP BB9	0.0964	0.180	0.107	ND	ND
SP BB13	0.103	0.194	0.119	ND	ND
SP CA2	0.107	0.194	0.106	ND	ND
SP CB3	0.108	0.186	0.108	ND	ND
SP BB12	0.132	0.254	0.159	ND	ND
SP BA4	0.108	0.208	0.118	ND	ND
MW D11	ND	ND	ND	ND	ND
MW D9	0.604	0.103	ND	ND	ND
MW M4	0.333	0.217	ND	ND	ND
River R1	ND	ND	ND	ND	ND
River R1*	ND	ND	ND	ND	ND
River R2	0.0646	0.0807	ND	ND	ND
River R3	ND	ND	ND	ND	ND
River R4	0.0504	0.0763	ND	ND	ND

*Laboratory Duplicate
ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).
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Analytical Report

Summary of Fluorochemical Residues in Water Samples (continued)

Sample ID	C14 Acid Perfluorotetradecanoic Acid Analyte Found (ng/mL, ppb)	PFBS Perfluorobutanesulfonate Analyte Found (ng/mL, ppb)	PFHS Perfluorohexanesulfonate Analyte Found (ng/mL, ppb)	PFOS Perfluorooctanesulfonate Analyte Found (ng/mL, ppb)	FOSA Perfluorooctane sulfonamide Analyte Found (ng/mL, ppb)
MW M10	ND	0.145	ND	ND	ND
MW M10*	ND	0.188	ND	ND	ND
MW M11	ND	0.227	0.0362	0.152	ND
MW M9	ND	0.282	0.108	ND	ND
MW D2	ND	0.749	0.155	1.07	0.0429
MW M1	ND	0.180	0.159	0.451	ND
MW U1	ND	ND	ND	ND	ND
MW D3	ND	0.281	0.0381	0.105	ND
MW D1	ND	0.588	0.263	1.98	ND
MW M5	ND	2.19	0.841	2.52	ND
MW M6A	ND	0.454	NQ	0.127	ND
MW M17	ND	19.4	0.219	2.31	0.134
MW M14	ND	0.696	0.719	0.753	ND
MW M13	ND	2.49	1.00	2.18	ND
MW D6	ND	1.57	0.337	1.93	0.0842
MW D6*	ND	1.42	0.279	1.83	0.0742
MW D4	ND	4.36	0.958	3.35	ND
MW M3	ND	ND	ND	ND	ND
MW M7	ND	0.406	0.361	0.986	ND
MW M2	ND	NQ	ND	ND	ND
MW M8	ND	4.79	0.0695	0.479	ND
MW M12	ND	1.62	0.515	2.06	ND
SP AC5	ND	1.24	ND	0.287	0.0265
SP BA2	ND	1.10	NQ	0.236	NQ
SP BA2*	ND	1.23	0.0290	0.259	0.0277
SP CA15	ND	2.23	0.0594	0.289	0.0321
SP CA15*	ND	2.40	0.0682	0.348	0.0385
SP AC2	ND	1.38	0.0301	0.350	0.0487
SP AC15	ND	1.59	0.0390	0.272	0.0259
SP AC4	ND	1.59	0.0336	0.387	0.0565
SP AC14	ND	1.77	0.0430	0.380	0.0513
SP CB14A	ND	3.86	0.0417	0.336	0.0414
SP CB12	ND	3.21	0.0686	0.281	0.0269
SP BB9	ND	3.01	0.0439	0.275	0.0264
SP BB13	ND	2.92	0.0508	0.280	0.0291
SP CA2	ND	3.19	0.0690	0.277	0.0274
SP CB3	ND	3.66	0.0464	0.276	0.0253
SP BB12	ND	3.99	0.0833	0.382	0.0413
SP BA4	ND	3.18	0.0697	0.292	0.0319
MW D11	ND	ND	ND	NQ	ND
MW D9	ND	5.11	0.531	2.94	ND
MW M4	ND	0.641	1.00	5.15	0.0329
River R1	ND	NQ	ND	NQ	ND
River R1*	ND	NQ	ND	NQ	ND
River R2	ND	0.319	0.0484	0.665	0.0575
River R3	ND	NQ	ND	0.0477	ND
River R4	ND	0.295	0.0368	0.601	0.0442

*Laboratory Duplicate
ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).
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Recovery Summary of Fluorochemical Residues in Water Samples

Sample Description	Amount Spiked (ng/mL)	C4 Acid Amt Found in Sample (ng/mL)	C4 Acid Amount Recovered (ng/mL)	C4 Acid Recovery (%)	C5 Acid Amt Found in Sample (ng/mL)	C5 Acid Amount Recovered (ng/mL)	C5 Acid Recovery (%)	C6 Acid Amt Found in Sample (ng/mL)	C6 Acid Amount Recovered (ng/mL)	C6 Acid Recovery (%)	C7 Acid Amt Found in Sample (ng/mL)	C7 Acid Amount Recovered (ng/mL)	C7 Acid Recovery (%)
Reagent Spike A (061809A) 0.05 ng/mL	0.05	ND	0.0541	108	ND	0.0581	116	ND	0.0600	120	ND	0.0582	116
Reagent Spike B (061809A) 0.5 ng/mL	0.5	ND	0.408	82	ND	0.410	82	ND	0.393	79	ND	0.468	94
Reagent Spike A (061809B) 0.05 ng/mL	0.05	ND	0.0557	111	ND	0.0480	96	ND	0.0646	129	ND	0.0469	94
Reagent Spike B (061809B) 0.5 ng/mL	0.5	ND	0.395	79	ND	0.398	80	ND	0.363	73	ND	0.463	93
Reagent Spike A (062309A) 0.05 ng/mL	0.05	--	--	--	--	--	--	ND	0.0531	106	--	--	--
Reagent Spike B (062309A) 0.5 ng/mL	0.5	--	--	--	--	--	--	ND	0.498	99	--	--	--
MW M18 Matrix Spike (L18099-19 Spk C, 0.5 ng/mL Lab Spike)	0.5	ND	0.589	118	0.0373	0.733	139^	0.0498	0.664	129	ND	0.780	156^
MW D6 Matrix Spike (L18099-32 Spk D, 0.5 ng/mL Lab Spike)	0.5	0.550	0.923	75	1.92	2.33	82	1.68	2.14	92	1.13	1.65	104
SP BA2 Matrix Spike (L18099-40 Spk E, 0.5 ng/mL Lab Spike)	0.5	0.665	1.03	73	1.37	1.81	88	0.811	1.25	88	0.328	0.899	114
SP CA15 Matrix Spike (L18099-41 Spk C, 0.5 ng/mL Lab Spike)	0.5	0.684	1.16	95	1.45	2.02	114	0.723	1.46	147^	0.261	0.903	128
River R1 Matrix Spike (L18099-57 Spk D, 0.5 ng/mL Lab Spike)	0.5	ND	0.495	99	ND	0.581	116	ND	0.523	105	ND	0.644	129

Sample Description	Amount Spiked (ng/mL)	C8 Acid Amt Found in Sample (ng/mL)	C8 Acid Amount Recovered (ng/mL)	C8 Acid Recovery (%)	C9 Acid Amt Found in Sample (ng/mL)	C9 Acid Amount Recovered (ng/mL)	C9 Acid Recovery (%)	C10 Acid Amt Found in Sample (ng/mL)	C10 Acid Amount Recovered (ng/mL)	C10 Acid Recovery (%)	C11 Acid Amt Found in Sample (ng/mL)	C11 Acid Amount Recovered (ng/mL)	C11 Acid Recovery (%)
Reagent Spike A (061809A) 0.05 ng/mL	0.05	ND	0.0569	114	ND	0.0612	122	ND	0.0486	97	ND	0.0602	120
Reagent Spike B (061809A) 0.5 ng/mL	0.5	ND	0.395	79	ND	0.433	87	ND	0.435	87	ND	0.398	80
Reagent Spike A (061809B) 0.05 ng/mL	0.05	ND	0.0533	107	ND	0.0468	94	ND	0.0581	116	ND	0.0575	115
Reagent Spike B (061809B) 0.5 ng/mL	0.5	ND	0.397	79	ND	0.403	81	ND	0.417	83	ND	0.368	74
Reagent Spike A (062309A) 0.05 ng/mL	0.05	ND	0.0443	89	--	--	--	--	--	--	--	--	--
Reagent Spike B (062309A) 0.5 ng/mL	0.5	ND	0.589	118	--	--	--	--	--	--	--	--	--
MW M18 Matrix Spike (L18099-19 Spk C, 0.5 ng/mL Lab Spike)	0.5	ND	0.654	131^	ND	0.607	121	ND	0.686	137^	ND	0.600	120
MW D6 Matrix Spike (L18099-32 Spk D, 0.5 ng/mL Lab Spike)	0.5	2.73	3.32	118	0.575	1.15	115	0.211	0.830	124	ND	0.505	101
SP BA2 Matrix Spike (L18099-40 Spk E, 0.5 ng/mL Lab Spike)	0.5	0.469	0.94	94	0.0652	0.555	98	0.182	0.744	116	0.107	0.585	96
SP CA15 Matrix Spike (L18099-41 Spk C, 0.5 ng/mL Lab Spike)	0.5	0.509	1.18	134^	0.113	0.671	112	0.200	0.765	113	0.118	0.728	122
River R1 Matrix Spike (L18099-57 Spk D, 0.5 ng/mL Lab Spike)	0.5	ND	0.610	122	ND	0.704	141^	ND	0.627	125	ND	0.620	124

ND = Not detected = Response is below the LOD of 0.0125 ng/mL.
NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL.
**Analysis not required.
^ Confirmation analysis was performed for the out of range recovery. The second analysis confirmed the high recovery, a matrix effect is suspected to be the cause.

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Recovery Summary of Fluorochemical Residues in Water Samples (continued)

Sample Description	C12 Acid Amount Spiked (ng/mL)	C12 Acid Amt Found in Sample (ng/mL)	C12 Acid Amount Recovered (ng/mL)	C12 Acid Recovery (%)	C13 Acid Amt Found in Sample (ng/mL)	C13 Acid Amount Recovered (ng/mL)	C13 Acid Recovery (%)	C14 Acid Amt Found in Sample (ng/mL)	C14 Acid Amount Recovered (ng/mL)	C14 Acid Recovery (%)	PFBS Amt Found in Sample (ng/mL)	PFBS Amount Recovered (ng/mL)	PFBS Recovery (%)
Reagent Spike A (061809A) 0.05 ng/mL	0.05	ND	0.0574	115	ND	0.0513	103	ND	0.0647	129	ND	0.0546	109
Reagent Spike B (061809A) 0.5 ng/mL	0.5	ND	0.395	79	ND	0.427	85	ND	0.408	82	ND	0.442	88
Reagent Spike A (061809B) 0.05 ng/mL	0.05	ND	0.0606	121	ND	0.0580	116	ND	0.0518	104	ND	0.0652	130
Reagent Spike B (061809B) 0.5 ng/mL	0.5	ND	0.366	73	ND	0.390	78	ND	0.384	77	ND	0.393	79
Reagent Spike A (062309A) 0.05 ng/mL	0.05	--	--	--	--	--	--	--	--	--	ND	0.041	82
Reagent Spike B (062309A) 0.5 ng/mL	0.5	--	--	--	--	--	--	--	--	--	ND	0.516	103
MW M16 Matrix Spike (L18099-19 Spk C, 0.5 ng/mL Lab Spike)	0.5	ND	0.641	128	ND	0.725	145^	ND	0.603	121	0.145	0.718	115
MW D6 Matrix Spike (L18099-32 Spk D, 0.5 ng/mL Lab Spike)	0.5	ND	0.577	115	ND	0.716	143^	ND	0.597	119	1.57	1.93	72
SP BA2 Matrix Spike (L18099-40 Spk E, 0.5 ng/mL Lab Spike)	0.5	ND	0.484	97	ND	0.607	121	ND	0.477	95	1.10	1.53	86
SP CA16 Matrix Spike (L18099-41 Spk C, 0.5 ng/mL Lab Spike)	0.5	ND	0.566	113	ND	0.572	114	ND	0.515	103	2.23	2.86	126
River R1 Matrix Spike (L18099-67 Spk D, 0.5 ng/mL Lab Spike)	0.5	ND	0.672	134^	ND	0.708	142^	ND	0.622	124	NQ	0.598	120

Sample Description	PFHS Amount Spiked (ng/mL)	PFHS Amt Found in Sample (ng/mL)	PFHS Amount Recovered (ng/mL)	PFHS Recovery (%)	PFOS Amt Found in Sample (ng/mL)	PFOS Amount Recovered (ng/mL)	PFOS Recovery (%)	FOSA Amt Found in Sample (ng/mL)	FOSA Amount Recovered (ng/mL)	FOSA Recovery (%)
Reagent Spike A (061809A) 0.05 ng/mL	0.05	ND	0.0568	114	ND	0.0514	103	ND	0.0541	108
Reagent Spike B (061809A) 0.5 ng/mL	0.5	ND	0.416	83	ND	0.407	81	ND	0.440	88
Reagent Spike A (061809B) 0.05 ng/mL	0.05	ND	0.0541	108	ND	0.0584	117	ND	0.0497	99
Reagent Spike B (061809B) 0.5 ng/mL	0.5	ND	0.413	83	ND	0.398	80	ND	0.401	80
Reagent Spike A (062309A) 0.05 ng/mL	0.05	--	--	--	ND	0.0432	86	--	--	--
Reagent Spike B (062309A) 0.5 ng/mL	0.5	--	--	--	ND	0.545	109	--	--	--
MW M16 Matrix Spike (L18099-19 Spk C, 0.5 ng/mL Lab Spike)	0.5	ND	0.575	115	ND	0.585	117	ND	0.609	122
MW D6 Matrix Spike (L18099-32 Spk D, 0.5 ng/mL Lab Spike)	0.5	0.337	0.783	89	1.93	2.47	108	0.0842	0.625	108
SP BA2 Matrix Spike (L18099-40 Spk E, 0.5 ng/mL Lab Spike)	0.5	NQ	0.415	83	0.236	0.632	79	NQ	0.470	94
SP CA16 Matrix Spike (L18099-41 Spk C, 0.5 ng/mL Lab Spike)	0.5	0.0594	0.515	91	0.289	0.779	98	0.0321	0.553	104
River R1 Matrix Spike (L18099-67 Spk D, 0.5 ng/mL Lab Spike)	0.5	ND	0.595	119	NQ	0.581	112	ND	0.650	130

ND = Not detected = Response is below the LOD of 0.0125 ng/mL.
NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL.
**Analysis not required.
^ Confirmation analysis was performed for the out of range recovery. The second analysis confirmed the high recovery, a matrix effect is suspected to be the cause.

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Recovery Summary of ^{13}C PFOA (m+4) in Water Samples

Client Sample ID	MPI Sample ID	Amount Spiked (ng/mL, ppb)	Amount Recovered (ng/mL, ppb)	Recovery (%)
NA	Reagent Control (061809A)	0.50	0.616	123
NA	Reagent Spike A (061809A)	0.05	0.0585	117
NA	Reagent Spike B (061809A)	0.50	0.442	88
NA	Reagent Control (061809B)	0.50	0.669	134
NA	Reagent Spike A (061809B)	0.05	0.0497	99
NA	Reagent Spike B (061809B)	0.50	0.452	90
MW M10 Spike C	L18099-19 Spike C	0.50	0.638	128
MW M10	L18099-19	0.50	0.597	119
MW M10*	L18099-19 DUP	0.50	0.595	119
MW M11	L18099-20	0.50	0.693	139
MW M9	L18099-21	0.50	0.613	123
MW D2	L18099-22	0.50	0.640	128
MW M1	L18099-23	0.50	0.595	119
MW U1	L18099-24	0.50	0.602	120
MW D3	L18099-25	0.50	0.562	112
MW D1	L18099-26	0.50	0.630	126
MW M5	L18099-27	0.50	0.636	127
MW M6A	L18099-28	0.50	0.619	124
MW M17	L18099-29	0.50	0.603	121
MW M14	L18099-30	0.50	0.640	128
MW M13	L18099-31	0.50	0.664	133
MW D6 Spike D	L18099-32 Spike D	0.50	0.625	125
MW D6	L18099-32	0.50	0.673	135
MW D6*	L18099-32 DUP	0.50	0.550	110
MW D4	L18099-33	0.50	0.535	107
MW M3	L18099-34	0.50	0.574	115
MW M7	L18099-35	0.50	0.533	107
MW M2	L18099-36	0.50	0.522	104
MW M8	L18099-37	0.50	0.561	112
MW M12	L18099-38	0.50	0.580	116
SP AC5	L18099-39	0.50	0.529	106
SP BA2 Spike E	L18099-40 Spike E	0.50	0.586	117
SP BA2	L18099-40	0.50	0.558	112
SP BA2*	L18099-40 DUP	0.50	0.590	118
SP CA15 Spike C	L18099-41 Spike C	0.50	0.619	124
SP CA15	L18099-41	0.50	0.615	123
SP CA15*	L18099-41 DUP	0.50	0.540	108
SP AC2	L18099-42	0.50	0.565	113
SP AC15	L18099-43	0.50	0.648	100
SP AC4	L18099-44	0.50	0.564	113
SP AC14	L18099-45	0.50	0.579	116
SP CB14A	L18099-46	0.50	0.586	117
SP CB12	L18099-47	0.50	0.577	115
SP BB9	L18099-48	0.50	0.551	110
SP BB13	L18099-49	0.50	0.517	103
SP CA2	L18099-50	0.50	0.579	116
SP CB3	L18099-51	0.50	0.680	136
SP BB12	L18099-52	0.50	0.647	129
SP BA4	L18099-53	0.50	0.611	122
MW D11	L18099-54	0.50	0.606	121
MW D9	L18099-55	0.50	0.645	129
MW M4	L18099-56	0.50	0.623	125
River R1 Spike D	L18099-57 Spike D	0.50	0.730	146
River R1	L18099-57	0.50	0.604	121
River R1*	L18099-57 DUP	0.50	0.689	138
River R2	L18099-58	0.50	0.595	119
River R3	L18099-59	0.50	0.628	126
River R4	L18099-60	0.50	0.628	126

* Laboratory Duplicate

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Analytical Report

Summary of Fluorochemical Residues in Solid Samples

Sample ID	C4 Acid Perfluorobutyric Acid Analyte Found (µg/kg) Dry Weight	C5 Acid Perfluoropentanoic Acid Analyte Found (µg/kg) Dry Weight	C6 Acid Perfluorohexanoic Acid Analyte Found (µg/kg) Dry Weight	C7 Acid Perfluoroheptanoic Acid Analyte Found (µg/kg) Dry Weight	C8 Acid Perfluorooctanoic Acid Analyte Found (µg/kg) Dry Weight
Compost	712	408	559	499	4420
AC 6 Soil	1.36	4.20	2.88	1.58	6.83
AC 6 Soil*	1.63	4.38	3.42	1.68	7.75
BA 11 Soil	2.44	6.79	4.68	1.59	8.64
BA 12 Soil	6.30	7.97	7.31	2.72	14.3
BB 13 Soil	4.48	15.6	11.0	6.58	21.3
CA 5 Soil	4.60	11.1	6.37	2.89	16.9
CA 12 Soil	3.43	11.6	7.76	3.36	12.2
CB 4 Soil	3.71	10.0	4.89	1.34	5.34
CB 14A Soil	7.37	32.3	21.5	8.75	29.7
BB 9 Soil	1.15	5.48	3.88	1.62	8.46
CA 9B Soil	1.89	3.95	3.77	2.18	16.7
CB 13 Soil	3.27	10.2	9.22	4.46	17.7
AC 13 Soil	1.30	8.94	5.43	2.36	7.36
BA 5 Soil	4.86	13.4	7.96	6.08	37.0
BB 12 Soil	3.90	12.3	7.45	3.27	12.8
STP 2 Sludge	ND	224	157	ND	87.5
STP 2 Sludge*	ND	215	187	ND	81.3
STP 3 Sludge	ND	281	128	ND	68.2
STP 4 Sludge	152	415	190	33.8	134

*Laboratory Duplicate
ND = Not Detected = Response below the LOQ of 0.2 µg/kg (wet weight)

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Analytical Report

Summary of Fluorochemical Residues in Solid Samples (continued)

Sample ID	C9 Acid Perfluorononanoic Acid Analyte Found (µg/kg) Dry Weight	C10 Acid Perfluorodecanoic Acid Analyte Found (µg/kg) Dry Weight	C11 Acid Perfluoroundecanoic Acid Analyte Found (µg/kg) Dry Weight	C12 Acid Perfluorododecanoic Acid Analyte Found (µg/kg) Dry Weight	C13 Acid Perfluorotridecanoic Acid Analyte Found (µg/kg) Dry Weight
Compost	681	3160	1400	654	441
AC 6 Soil	3.89	20.1	60.2	44.2	44.0
AC 6 Soil*	4.36	19.2	53.9	41.9	47.0
BA 11 Soil	5.44	48.6	117	53.2	47.4
BA 12 Soil	9.89	33.8	37.1	10.5	9.99
BB 13 Soil	17.8	93.7	433	109	282
CA 5 Soil	8.54	48.8	52.4	34.6	23.0
CA 12 Soil	7.96	40.4	124	59.7	96.3
CB 4 Soil	3.26	22.3	81.9	18.4	24.9
CB 14A Soil	19.2	70.6	164	105	166
BB 9 Soil	3.90	24.3	43.3	25.2	35.0
CA 9B Soil	9.34	44.0	39.3	26.2	16.9
CB 13 Soil	11.4	46.5	132	52.1	93.3
AC 13 Soil	3.33	16.6	50.5	33.5	37.5
BA 5 Soil	8.06	53.2	14.8	27.6	4.96
BB 12 Soil	12.6	58.9	123	41.0	83.6
STP 2 Sludge	ND	ND	93.2	ND	ND
STP 2 Sludge*	ND	ND	66.7	ND	ND
STP 3 Sludge	ND	92.1	102	ND	49.2
STP 4 Sludge	47.6	208	347	74.0	195

*Laboratory Duplicate
ND = Not Detected = Response below the LOQ of 0.2 µg/kg (wet weight)

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Analytical Report

Summary of Fluorochemical Residues in Solid Samples (continued)

Sample ID	C14 Acid Perfluorotetradecanoic Acid Analyte Found (µg/kg) Dry Weight	PFBS Perfluorobutanesulfonate Analyte Found (µg/kg) Dry Weight	PFHS Perfluorohexanesulfonate Analyte Found (µg/kg) Dry Weight	PFOS Perfluorooctanesulfonate Analyte Found (µg/kg) Dry Weight	FOSA Perfluorooctane sulfonamid Analyte Found (µg/kg) Dry Weight
Compost	129	1370	72.3	2500	108
AC 6 Soil	22.7	4.56	0.589	67.7	188
AC 6 Soil*	25.4	5.06	0.706	64.8	176
BA 11 Soil	19.2	12.8	0.732	135	358
BA 12 Soil	4.29	7.85	1.24	174	12.5
BB 13 Soil	42.8	36.6	1.35	243	349
CA 5 Soil	15.3	15.8	1.98	288	323
CA 12 Soil	23.9	40.3	0.932	78.9	52.2
CB 4 Soil	5.51	9.35	0.509	37.7	242
CB 14A Soil	50.0	84.5	3.01	147	187
BB 9 Soil	13.1	15.9	0.893	85.7	49.3
CA 9B Soil	11.6	7.50	1.58	283	169
CB 13 Soil	37.5	15.3	2.00	144	166
AC 13 Soil	19.0	6.81	0.671	46.6	332
BA 5 Soil	6.83	1.87	1.99	178	32.6
BB 12 Soil	17.5	24.0	0.975	153	66.3
STP 2 Sludge	ND	74.3	ND	171	144
STP 2 Sludge*	ND	82.4	ND	136	94.4
STP 3 Sludge	ND	1290	ND	84.7	27.5
STP 4 Sludge	ND	1940	ND	170	58.0

*Laboratory Duplicate
ND = Not Detected = Response below the LOQ of 0.2 µg/kg (wet weight)

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Recovery Summary of Fluorochemical Residues in Solid Samples

Sample Description	C4 Acid				C5 Acid			C6 Acid			C7 Acid		
	Amount Spiked* (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.05 ng/mL	0.05	ND	0.0364	73	ND	0.0454	91	ND	0.0542	108	ND	0.0444	89
Reagent Spike B 0.5 ng/mL	0.5	ND	0.441	88	ND	0.433	87	ND	0.437	87	ND	0.496	99
AC 6 Soil Matrix Spike (L18099-2 Spt C, 0.5 ng/mL Lab Spike)	0.5	0.122	0.611	98	0.377	0.742	73	0.259	0.761	100	0.142	0.598	91
AC 6 Soil Matrix Spike (L18099-2 Spt D, 5 ng/mL Lab Spike)	5.0	--	--	--	--	--	--	--	--	--	--	--	--
STP 2 Sludge Matrix Spike (L18099-16 Spt E, 0.5 ng/mL Lab Spike)	0.5	ND	0.356	71	0.0825	0.569	96	0.0648	0.813	110	ND	0.552	110

Sample Description	C8 Acid				C9 Acid			C10 Acid			C11 Acid		
	Amount Spiked* (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.05 ng/mL	0.05	ND	0.0492	98	ND	0.0619	124	ND	0.0541	108	ND	0.0538	108
Reagent Spike B 0.5 ng/mL	0.5	ND	0.466	93	ND	0.517	103	ND	0.461	92	ND	0.502	100
AC 6 Soil Matrix Spike (L18099-2 Spt C, 0.5 ng/mL Lab Spike)	0.5	0.613	1.26	129	0.349	0.916	113	1.81	2.36	110	--	--	--
AC 6 Soil Matrix Spike (L18099-2 Spt D, 5 ng/mL Lab Spike)	5.0	--	--	--	--	--	--	--	--	--	5.41	12.2	136^
STP 2 Sludge Matrix Spike (L18099-16 Spt E, 0.5 ng/mL Lab Spike)	0.5	0.0361	0.634	120	ND	0.504	101	ND	0.527	105	0.0384	0.562	105

Sample Description	C12 Acid				C13 Acid			C14 Acid			PFBS		
	Amount Spiked* (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.05 ng/mL	0.05	ND	0.0496	99	ND	0.0624	125	ND	0.0539	108	ND	0.0561	112
Reagent Spike B 0.5 ng/mL	0.5	ND	0.457	91	ND	0.470	94	ND	0.472	94	ND	0.408	82
AC 6 Soil Matrix Spike (L18099-2 Spt C, 0.5 ng/mL Lab Spike)	0.5	3.96	4.47	102	--	--	--	--	--	--	0.409	1.04	126
AC 6 Soil Matrix Spike (L18099-2 Spt D, 5 ng/mL Lab Spike)	5.0	--	--	--	3.95	9.50	111	2.04	7.94	118	--	--	--
STP 2 Sludge Matrix Spike (L18099-16 Spt E, 0.5 ng/mL Lab Spike)	0.5	ND	0.406	82	ND	0.541	108	ND	0.595	119	0.0308	0.581	112

Sample Description	PFHS				PFOS			FOSA		
	Amount Spiked* (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.05 ng/mL	0.05	ND	0.0564	113	ND	0.0507	101	ND	0.0506	101
Reagent Spike B 0.5 ng/mL	0.5	ND	0.460	92	ND	0.448	90	ND	0.480	96
AC 6 Soil Matrix Spike (L18099-2 Spt C, 0.5 ng/mL Lab Spike)	0.5	0.0529	0.603	110	--	--	--	--	--	--
AC 6 Soil Matrix Spike (L18099-2 Spt D, 5 ng/mL Lab Spike)	5.0	--	--	--	6.06	10.7	92	16.9	20.9	80
STP 2 Sludge Matrix Spike (L18099-16 Spt E, 0.5 ng/mL Lab Spike)	0.5	ND	0.492	98	0.0704	0.580	102	0.0595	0.522	93

ND = Not detected = Response less than 0.025 ng/mL.
*Spiking levels refer to the amount of analyte in the extracts.
**Analysis not required.
^ Confirmation analysis was performed for the out of range recovery. The second analysis confirmed the high recovery, a matrix effect is suspected to be the cause.

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Recovery Summary of ^{13}C PFOA (m+4) in Solid Samples

Client Sample ID	MPI Sample ID	Amount Spiked (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
NA	Reagent Control	0.50	0.832	166
NA	Reagent Spike A	0.05	0.0565	113
NA	Reagent Spike B	0.50	0.516	103
Compost	L18099-1	0.50	0.589	118
AC 6 Soil Matrix Spike	L18099-2 Spike C	0.50	0.733	147
AC 6 Soil Matrix Spike	L18099-2 Spike D	5.0	6.51	130
AC 6 Soil	L18099-2	0.50	0.650	130
AC 6 Soil*	L18099-2 DUP	0.50	0.670	134
BA 11 Soil	L18099-3	0.50	0.719	144
BA 12 Soil	L18099-4	0.50	0.726	145
BB 13 Soil	L18099-5	0.50	0.592	118
CA 5 Soil	L18099-6	0.50	0.656	131
CA 12 Soil	L18099-7	0.50	0.595	119
CB 4 Soil	L18099-8	0.50	0.634	127
CB 14A Soil	L18099-9	0.50	0.612	122
BB 9 Soil	L18099-10	0.50	0.643	129
CA 9B Soil	L18099-11	0.50	0.609	122
CB 13 Soil	L18099-12	0.50	0.616	123
AC 13 Soil	L18099-13	0.50	0.578	116
BA 5 Soil	L18099-14	0.50	0.612	122
BB 12 Soil	L18099-15	0.50	0.628	126
STP 2 Sludge Matrix Spike	L18099-16 Spike E	0.50	0.586	117
STP 2 Sludge	L18099-16	0.50	0.553	111
STP 2 Sludge*	L18099-16 DUP	0.50	0.568	114
STP 3 Sludge	L18099-17	0.50	0.564	113
STP 4 Sludge	L18099-18	0.50	0.605	121

*Laboratory Duplicate