

3M Company St. Paul, Minnesota

Perfluorooctanoic Acid (PFOA) Site Related Environmental Assessment Program

3M and Dyneon LLC

Decatur, Alabama

Status Report July 25, 2007 to August 2008





Perfluorooctanoic Acid (PFOA) Site Related Environmental Assessment Program 3M Decatur, Alabama Site

Status Report July 25, 2007 to August 2008

Prepared for

3M Company and Dyneon LLC

Ву

WESTON SOLUTIONS, INC. West Chester, Pennsylvania 19380

W.O. No. 02181.129.081.0001



TABLE OF CONTENTS

Section	
Section	Page
1.	BACKGROUND1-1
2.	SUMMARY OF ACTIVITIES
	SUMMARY OF ACTIVITIES2-1



1. BACKGROUND

In accordance with the *Phase 2 Work Plan for Sampling Environmental Media* (WESTON, 2004) and the Memorandum of Understanding (MOU, 2004) with EPA, 3M and Dyneon LLC are undertaking a Perfluorooctanoic Acid (PFOA) Site-Related Assessment Program at their Decatur, Alabama facility. A series of 11 Quarterly Status Reports were submitted over the interval between the effective date of the MOU of October 4, 2004 through the completion of Phase 2 monitoring on July 25, 2007. The Data Assessment Report, the Screening Level Human Exposure Assessment Report and the Future Data Needs Report for Phase 2 were submitted on January 15, 2008. These three documents were reviewed by a Peer Consultation Panel formed by Menzie-Cura & Associates followed by a meeting on April 16 and 17, 2008, in Decatur, Alabama, to complete the peer consultation process. The *Report on the Peer Consultation for a PFOA Site-Related Environmental Assessment Program for the 3M Property Located in Decatur, Alabama* (PFOA Peer Consultation Panel Report), dated June 1, 2008, has been issued (Menzie-Cura & Associates, 2008).

On June 18, 2008, EPA acknowledged the receipt of the PFOA Peer Consultation Panel Report and extended the review period to July 25, 2008, in accordance with the provisions of the MOU in a letter from Jim Willis to Michael Santoro of 3M. On July 24, 2008, EPA notified 3M of an additional extension of the review period to August 29, 2008. Upon completion of the EPA review, 3M and EPA will meet to discuss Phase 3 activities, including the scope of the Phase 3 data collection efforts necessary to satisfy the Charge in the MOU and the preparation of the Phase 3 Work Plan.

While the MOU has requirements for submitting quarterly status reports for Phase 2 and Phase 3 of the PFOA Site-Related Assessment Program, the interim period between the completion of Phase 2 activities and the commencement of Phase 3 activities was not addressed in terms of reporting. As discussed in the July 25, 2007 *Quarterly Status Report*, a new Good Laboratory Practice (GLP) Protocol P0003267 was developed for the analysis of environmental media samples collected after the Phase 2 effort. Additional



sampling and analysis of PFOA has been performed under P0003267 on a variety of media since the completion of the analytical component of Phase 2 monitoring on April 25, 2007. The purpose of this activity was to complete the commitments under the Letter of Intent (LOI) and to further characterize the marsh and drainageway areas west of the site. This report provides a summary of field and other activities completed and analytical data finalized between July 25, 2007 and August 2008. These data will be incorporated into the analytical data collected during Phase 3 activities and into the revised screening level human exposure assessment. As required by the MOU, 3M will submit quarterly status reports summarizing the progress of Phase 3 of the PFOA Site-Related Assessment Program after the Phase 3 Work Plan is finalized. For brevity, this report will be referred to as the August 2008 Status Report.



2. SUMMARY OF ACTIVITIES

The referenced *Phase 2 Work Plan* and MOU were finalized on October 25, 2004, and field activities were initiated in accordance with the *Phase 2 Work Plan*. This report represents the status report that covers the July 25, 2007 to August 2008 timeframe and includes a description of activities completed under the *Phase 2 Work Plan* since the July 25, 2007 *Quarterly Status Report*, the completion of the *Data Assessment Report*, *Screening Level Human Exposure Assessment Report* and the *Future Data Needs Report*, and the conclusion of the Peer Consultation process. Details are as follows:

- Off-site fish, clam, surface water and sediment sampling and analysis: Fish, clam, surface water and sediment samples collected from locations in the Tennessee River in December, 2006 to complete the Letter of Intent (LOI) commitment and on-site surface water and sediment samples in the Avenue A drainageway have been analyzed for PFOA under the P0003267 Good Laboratory Practices (GLP) study protocol. In addition, off-site sampling of sediments in the off-site marsh and associated drainageway locations upgradient and downgradient of the marsh and in a nearby isolated pond was performed in August 2007 and analysis for PFOA in these samples has been completed. A description of the off-site marsh and drainageway system sediment sampling activities and analytical results are provided in the attached Aquatic Sampling Technical Progress Report.
- Off-site groundwater monitoring well sampling and analysis: Off-site sampling of groundwater at the 600 series wells was performed in September 2007. Additional sampling of the 605R and 605L wells was performed in April 2008. Analysis for PFOA in these samples has been completed and the results are provided in the attached Groundwater Sampling Technical Progress Report.



ATTACHMENT 1

AQUATIC SAMPLING TECHNICAL PROGRESS REPORT

August 2008



AQUATIC SAMPLING TECHNICAL PROGRESS REPORT 3M AND DYNEON DECATUR, ALABAMA

In December 2006, fish, clam, sediment and surface water sampling was performed at six locations (reaches) in the Tennessee River and Bakers Creek to fulfill the Letter of Intent (LOI) and Phase 2 commitments. This included sampling at on-site surface water and sediment locations in the Avenue A drainageway. Complete details of December 2006 sample collection activities were provided in the January 2007 *Quarterly Status Report*. In addition, off-site sampling of sediments in the off-site marsh and associated drainageway locations upgradient and downgradient of the marsh and in a nearby isolated pond was performed in August 2007. PFOA analyses of the 2006 and 2007 samples were performed by the MPI (formerly Exygen) laboratory under the P0003267 Good Laboratory Practices (GLP) protocol.

In addition, samples were collected from the off-site marsh and its surrounding drainageway in August 2007 to expand the characterization of sediment PFOA concentrations in this area.

Tennessee River / Bakers Creek Sediment Results

Sediment sampling in the Tennessee River/Bakers Creek was performed at six locations, including the three LOI locations. Six on-site sediment samples were also collected from the Avenue A drainageway (designated as DAA in this study). The sediment samples were collected with surface water sample locations.

The three LOI sediment sampling locations were located upstream of the facility at river mile 307.5 (LOC-3; designated as DL3 in this study), across the river from the facility at river mile 301 (LOC-2; designated as DL2 in this study), and downstream of the facility at the mouth of Fox Creek (approximately river mile 296; LOC-1; designated as DL1 in this study). Additional locations that were sampled include a location farther downstream on the Tennessee River at the mouth of Mallard Creek at approximately river mile 293 (designated DMC), the cove and mouth of Bakers Creek in the vicinity of the facility's Wastewater Treatment Plant outfall (designated



DOU) and Bakers Creek upstream of the facility's outfall (designated DBC). Analytical data on sediment PFOA concentrations are tabulated in Table 1 and shown in Figure 1.

Tennessee River / Bakers Creek Surface Water Results

Surface water sampling was performed in conjunction with the December 2006 sediment sampling described above. A single surface water sample was collected at each of the six locations in the Tennessee River and Bakers Creek and at two locations in the Avenue A drainageway. Analytical data on surface water PFOA concentrations are provided in Table 2 and shown in Figure 2. The surface water and sediment laboratory data package for PFOA (Interim Report 1) is provided in an appendix to this attachment.

Tennessee River / Bakers Creek Fish and Clam Results

Largemouth bass (*Micropterus salmoides*) and channel catfish (*Ictalurus punctatus*) were collected using electrofishing and trotlining methods from sampling reaches associated with the sediment and surface water sampling locations described above. Asiatic clams (*Corbicula fluminea*) were collected from each of the six fish sampling reaches by towing a weighted benthic dredge. A tabulation of the results is contained in Tables 3 and 4 and shown in Figure 3. The fish and clam analytical data package for PFOA (Interim Report 3) is provided in an appendix to this attachment.

Off-Site Marsh and Drainageway Sediment Sampling and Results

Sediment samples were collected from the off-site marsh and its surrounding drainageway in August 2007. Figure 4 depicts the sampling locations. Samples from the off-site marsh (locations EP01 through EP14) were collected using a Geoprobe® rig. Sediment samples were collected from the 0 to 1 ft below ground surface (bgs) interval at all sample locations; from the 2.5 to 3 ft bgs interval at locations EP02 through EP05, EP07, EP09 through EP11 and EP13; and from the 4.5 to 5 ft bgs interval at locations EP02, EP03, EP07, EP09 and EP10.

Sediment samples were also collected from drainageways surrounding the off-site marsh. Five samples were collected upstream of the off-site marsh (DU01 through DU05) from the 0 to 1 ft bgs interval. Samples were collected downstream (DS01 and DS02) from the 0 to 1 ft bgs and



2.5 to 3 ft bgs intervals. Two samples (WP01 and WP02) from the 0 to 1 ft bgs interval were also collected from an isolated pond not connected to the marsh and located southwest of the marsh. Sediment samples from the drainageways and southwest pond were collected using a shovel and hand auger, which were decontaminated between sampling locations.

The analytical results for the off-site marsh and surrounding drainageways sediment samples are tabulated in Table 5 and are shown in Figure 4.



Table 1 Tennessee River and Avenue A Sediment PFOA Concentrations

December 2006

Sample ID DAA-SD-LOC001-0-061215	Sample Area	Sample Location	Average PFOA (ppb, ng/g)
DAA-SD-LOC002-0-061215	1	Location 001	3.93
DAA-SD-LOC003-0-061215	DAA	Location 002	3.00
DAA-SD-LOC004-0-061215		Location 003	113
DAA-SD-LOC005-0-061215		Location 004	556
DAA-SD-LOC006-0-061215		Location 005	48.3
DBC-SD-LOC001-0-061214		Location 006	44.7
DBC-SD-LOC002-0-061214	DD c	Location 001	5.15
DBC-SD-LOC003-0-061214	DBC	Location 002	4.65
DL1-SD-LOC001-0-061213		Location 003	3.71
DL1-SD-LOC002-0-061213	D	Location 001	0.536
DL1-SD-LOC003-0-061213	DL1	Location 002	0.782
DL2-SD-LOC001-0-061214		Location 003	ND
DL2-SD-LOC002-0-061214	DIA	Location 001	ND
DL2-SD-LOC003-0-061214	DL2	Location 002	ND
DL3-SD-LOC001-0-061214		Location 003	ND
DL3-SD-LOC002-0-061214	DI a	Location 001	ND
DL3-SD-LOC003-0-061214	DL3	Location 002	ND
DMC-SD-LOC001-0-061213		Location 003	ND
DMC-SD-LOC002-0-061213	Die F	Location 001	0.505
DMC-SD-LOC003-0-061213	DMC	Location 002	0.629
DOU-SD-LOC0001-0-061213		Location 003	0.703
DOU-SD-LOC002-0-061213	DOI!	Location 001	39.3
DOU-SD-LOC003-0-061213	DOU [Location 002	8.63
133 0 001213		Location 003	8.88

Concentrations reported on a dry weight basis.

ND = Not detected at or above the acceptable LOQ of 0.2 ng/g.



Table 2 Tennessee River and Avenue A Surface Water PFOA Concentrations December 2006

Sample ID DL3-SW-LOC001-0-061214	Sample Area	Sample Locations	Average PFOA (ppb, ng/mL)
DL2-SW-LOC001-0-061214	DL3	Location 001	ND
DBC-SW LOC001-0-061214	DL2	Location 001	ND
DBC-SW-LOC001-0-061214	DBC	Location 001	ND
DOU-SW-LOC001-0-061213	DOU	Location 001	
DL1-SW-LOC001-0-061213	DL1	Location 001	3.54
DMC-SW-LOC001-0-061213	DMC		0.0764
DAA-SW-LOC002-0-061215	21110	Location 001	0.0511
DAA-SW-LOC005-0-061215	DAA	Location 002	1.32
1 0 0 0 0 0 0 0 12 13		Location 005	86.4

ND = Not detected at or above the acceptable LOQ of 0.025 ng/mL.



Table 3 Fish Fillet and Whole Body PFOA Concentrations December 2006

Sample ID	Specie	pic I	ype Average PFOA
DL3-F02-IPF001-0-06121	Upriver LO	C-3 (DL3)	(ppb, ng/g)
DI 3-F02 IPF002 0 06121	1		ND
DL3-F02-IPF002-0-06121	2		ND
DL3-F02-IPF003-0-06121	2	Fillet	ND
DL3-F02-IPF004-0-06121	2	2 met	ND
DL3-F02-IPF005-0-06121	2 Channe	1	ND
DL3-F02-IPW001-0-06121	2 catfish		ND
DL3-F02-IPW002-0-06121	2		ND
DL3-F02-IPW003-0-06121	2	Whole bod	ND ND
DL3-F02-IPW004-0-061212	2	Whole Boo	- 1
DL3-F02-IPW005-0-061212	2		ND
DL3-F02-MSF001-0-06120	7		ND
DL3-F02-MSF002-0-061203	7		0.469
DL3-F02-MSF003-0-061207	7	Fillet	ND
DL3-F02-MSF004-0-061207	,	rillet	0.370
DL3-F02-MSF005-0-061207	Largemout	h	0.295
DL3-F02-MSW001-0-061207	7 1 1	"	ND
DL3-F02-MSW002-0-061207	7	1	ND
DL3-F02-MSW003-0-061202	,	1871	0.386
DL3-F02-MSW004-0-061207	.	Whole body	' ND
DL3-F02-MSW005-0-061207	į		ND
	Cross River LOC	2 (DY2)	ND
222-102-11-001-0-061200	Tarrer Ede.	·2 (DL2)	
DL2-F02-IPF002-0-061209			ND
DL2-F02-IPF003-0-061209			ND
DL2-F02-IPF004-0-061209		Fillet	ND
DL2-F02-IPF005-0-061209	Channel		ND
DL2-F02-IPW001-0-061209	catfish		ND
DL2-F02-IPW002-0-061200	Cathisn		ND
DL2-F02-IPW003-0-061209			ND
DL2-F02-IPW004-0-061200		Whole body	ND
DL2-F02-IPW005-0-061209	ĺ		ND
-	 	ļ	ND
-	1		-
-		_	-
•		Fillet	
	Largemouth		_
DL2-F02-MSW001-0-061211			
DL2-F02-MSW002-0-061211	bass		0.377
-			0.550
-		Whole body	_
-			_
	1		

Fish tissue concentrations reported on a wet weight basis.

ND = Not detected at or above 0.2 ng/g.

NR = Not reported due to quality control issues.



Table 3 Fish Fillet and Whole Body PFOA Concentrations (cont.) December 2006

Sample ID	Specie	p.c 1 y	pe Average PFO
DBC-F02-IPF001-0-06121	Bakers Cree	k (DBC)	(PP0, 11g/g)
DBC-F02-IPF002-0-06121	1		1.42
DBC-F02-IPF003-0-06121	1		0.470
DBC-F02-IPF004-0-06121	1	Fillet	0.402
DBC-F02-IPF005-0-06121	1		0.432
DBC-F02-IPW001-0-06121	-	1	0.743
DBC-F02-IPW002-0-06121	l catfish		1.14
DBC-F02-IPW003-0-06121	1		0.640
DBC-F02-IPW004-0-06121	!	Whole body	2.01
DBC-F02-IPW005-0-061211		1	ND
DBC-F02-MSF001-0-06120	<u> </u>		1.48
DBC-F02-MSF002-0-061207			0.919
DBC-F02-MSF003-0-061207	1		0.354
DBC-F02-MSF004-0-061207		Fillet	0.334
DBC-F02-MSF005-0-061207	1		ND
DBC-F02-MSW001-0-061207		h	0.307
DBC-F02-MSW002-0-061207			2.89
DBC-F02-MSW003-0-061207		}	2.70
DBC-F02-MSW004-0-061207		Whole body	1.30
DBC-F02-MSW005-0-061207	1		1.56
32 M3 W 003-0-061207			1.74
DOU-F02-IPF001-0-061212	reek Mouth Nea	r Outfall (DOU)	1.74
DOU-F02-IPF002-0-061212			0.307
DOU-F02-IPF003-0-061212	1		0.533
DOU-F02-IPF004-0-061212		Fillet	0.333
DOU-F02-IPF005-0-061211			0.491
DOU-F02-IPW001-0-061212	Channel	1	1
DOU-F02-IPW002-0-061212	catfish		NR 0.627
DOU-F02-IPW003-0-061212		1	0.875
DOU-F02-IPW004-0-061212		Whole body	1.76
DOU-F02-IPW005-0-061212	1		ND
DOU-F02-MSF001-0-061209			
DOU-F02-MSF002-0-061212			1.11
DOU-F02-MSF003-0-061212			1.01
DOU-F02-MSF004-0-061212		Fillet	0.539
DOU-F02 MSF005 0 061212			0.560
DOU-F02-MSF005-0-061212	Largemouth		0.543
DOU-F02-MSW001-0-061209	bass		0.383
DOU-F02-MSW002-0-061212	į		1.14
DOU-F02-MSW003-0-061212		Whole body	3.05
DOU-F02-MSW004-0-061212 DOU-F02-MSW005-0-061212	l	body	1.24
h tissue concentrations reported of	i	-	1.07

ND = Not detected at or above 0.2 ng/g.

NR = Not reported due to quality control issues.



Table 3 Fish Fillet and Whole Body PFOA Concentrations (cont.) December 2006

Sample ID		Specie	- 1	Sample Ty	pe	Average PF((ppb, ng/g)	
DLI-F02-IPF001-0-06121	$\frac{Fa}{2}$	x Creek LO	C-1 (1	DL1)		(ppu, ng/g)	<u>'</u> _
DLI-F02-IPF002-0-06121	2		T		T	ND	
DLI-F02-IPF003-0-06121	2				İ	ND ND	
DLI-F02-IPF004-0-061212	2			Fillet	- 1		
DLI-F02-IPF005-0-061212	2					ND	
DL1-F02-IPW001-0-06121	2	Channel	1			ND	
DL1-F02-IPW002-0-06121	2	catfish			-+	ND 0.306	
DL1-F02-IPW003-0-061212	2					0.306	
DI 1-F02 IPW004 2 2 2 2 2	2			Whole body	,	0.336	
DL1-F02-IPW004-0-061215	5			body		0.310	
DL1-F02-IPW005-0-061215	5	,				0.264	
DLI-F02-MSF001-0-061209	9					0.525	_
DLI-F02-MSF002-0-061209)				-	ND	
DLI-F02-MSF003-0-061209)		- 1	Fillet		ND	
DLI-F02-MSF004-0-061209	- 1			- met	-	ND	
DL1-F02-MSF005-0-061209		Largemout	h			0.201	
DL1-F02-MSW001-0-061209) [bass	<u> </u>			ND	
DL1-F02-MSW002-0-061209)					0.339	
DL1-F02-MSW003-0-061209	1		,	Whole body	1	0.399	
DL1-F02-MSW004-0-061209	1			whole body		0.405	
DL1-F02-MSW005-0-061209						0.331	
DMC F02 TD	nrive	r Mallard C	reek (DMC)	Ь_	0.490	
	T		T	ome)			
DMC-F02-IPF002-0-061212			1			ND	ı
DMC-F02-IPF003-0-061212				Fillet		0.265	ı
DMC-F02-IPF004-0-061212	1		1	1 met		0.304	ı
DMC-F02-IPF005-0-061212		Channel				ND	ı
DMC-F02-IPW001-0-061212	7	catfish	-		 	ND	J
DMC-F02-IPW002-0-061212				j		ND	l
DMC-F02-IPW003-0-061212	1		w	hole body		ND	ı
DMC-F02-IPW004-0-061212	1		"	more body		ND	ı
DMC-F02-IPW005-0-061212	1					ND	ı
DMC-F02-MSF001-0-061209			 			ND	
DMC-F02-MSF002-0-061209				1		ND	
DMC-F02-MSF003-0-061209	1			E:II-4		0.254	
DMC-F02-MSF004-0-061212	1			Fillet		0.221	
DMC-F02-MSF005-0-061212	La	gemouth		1		ND	
DMC-F02-MSW001-0-061209		bass				ND	
DMC-F02-MSW002-0-061200				1		ND	
DMC-F02-MSW003-0-061200			% % 7 ×	_, , _		0.570	
JMC-F02-MSW004-0-061212			wh	ole body		0.423	
OMC-F02-MSW005-0-061212				j		0.841	
th tissue concentrations reported of the Not detected at or above 0.2				ļ		0.502	

ND = Not detected at or above 0.2 ng/g.

NR = Not reported due to quality control issues.



Table 4 Asiatic Clam PFOA Concentrations December 2006

Sample ID	Location	Average PFOA (ppb, ng/g)
DL3-102-CFW001-0-061219 DL2-102-CFW001-0-061219 DBC-102-CFW001-0-061219 DOU-102-CFW001-0-061219 DL1-102-CFW001-0-061219 DMC-102-CFW001-0-061219	Upriver LOC-3 (DL3) Cross River LOC-2 (DL2) Bakers Creek (DBC) Bakers Creek Mouth Near Outfall (DOU) Fox Creek LOC-1 (DL1) Downriver Mallard Creek (DMC)	0.219 0.360 0.898 0.845 0.221 ND

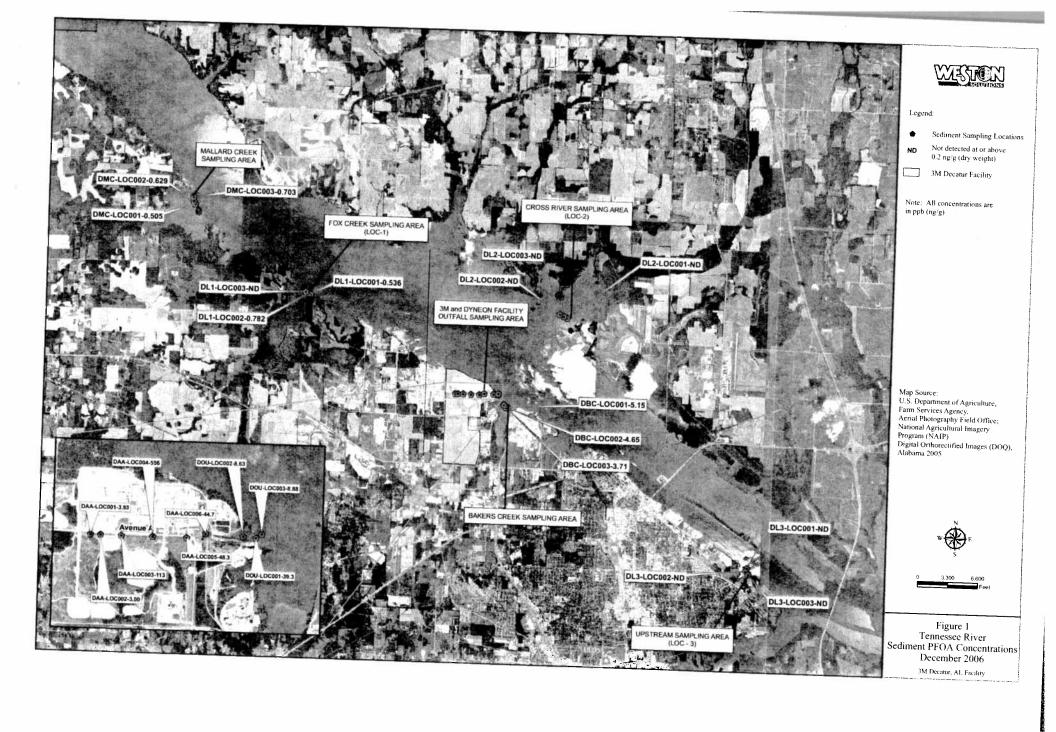
Clam tissue concentrations reported on a wet weight basis. ND = Not detected at or above 0.2 ng/g.

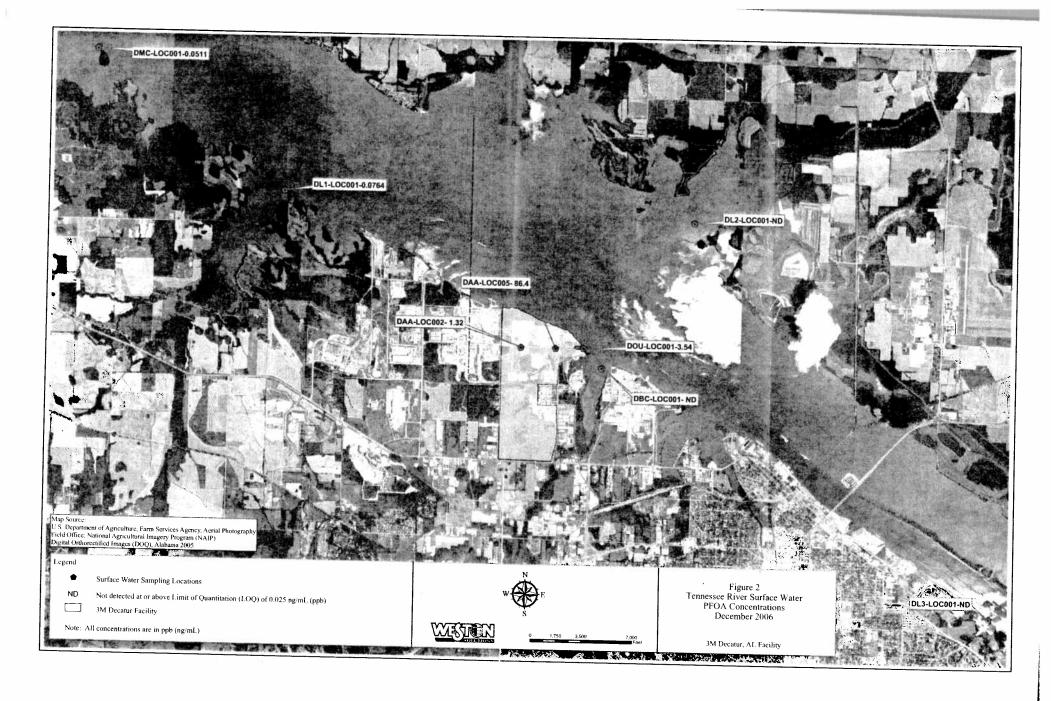


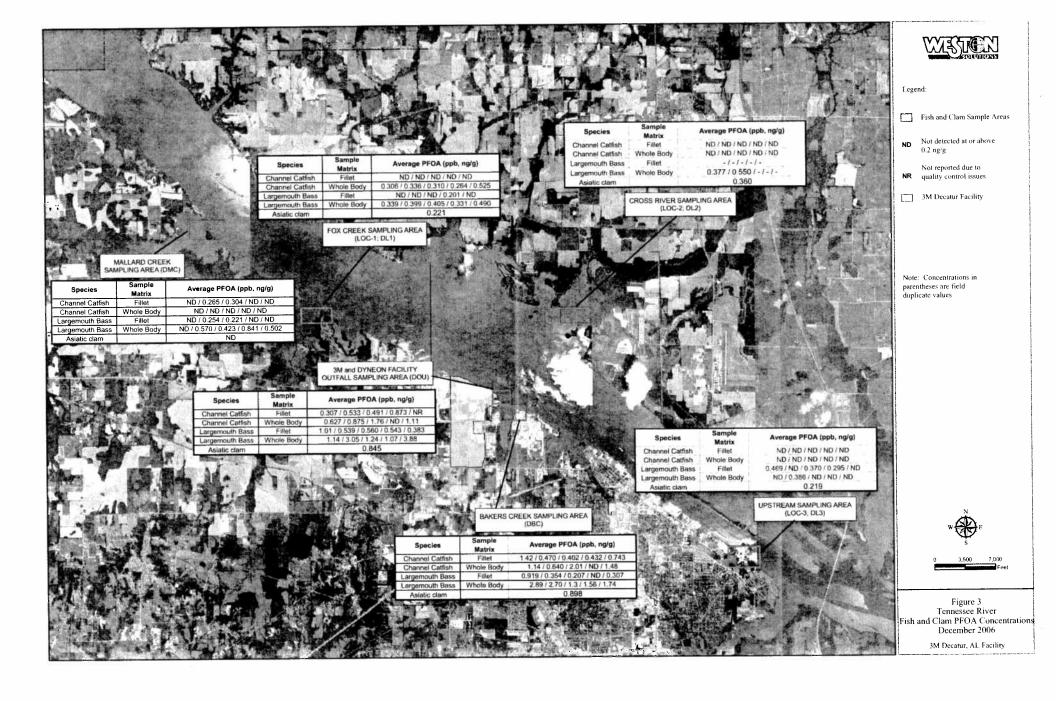
Table 5 Off-Site Marsh Sediment PFOA Concentrations
August 2007

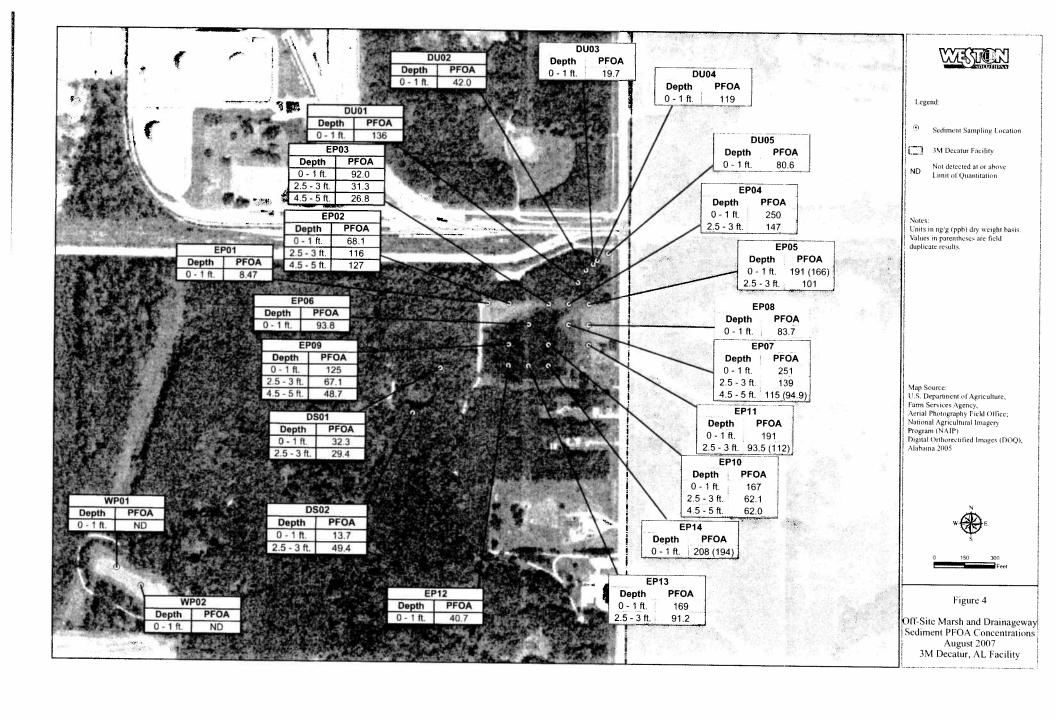
	Sample ID	Samp		epth	Average PF	$\hat{\Omega}$
- 1	DAL SD DS01 0 0010	Locati	(~5°)		(ppb, ng/g	ол ()
ı	DAL SD DS01 0 0010	DS01	0 - 1 ft		32.3	_
ı	DAL SD DS02 0 0010	JI	2.5 - 3 1	t.	29.4	
ı	DAL SD DS02 0 0030	⊢ DS02	0 - 1 ft		13.7	
	DAL SD DU01 0 0010	<u>' </u>	2.5 - 3 f	t.	49.4	_
ı	DAL SD DU02 0 0010		0 - 1 ft.		136	
F	DAL SD DU03 0 0010		0 - 1 ft.		42.0	
F	DAL SD DU04 0 0010		0 - 1 ft.		19.7	
	DAL SD DU05 0 0010		0 - 1 ft.		119	_
	DAL SD EP01 0 0010		0 - 1 ft.		80.6	
	DAL SD EP02 0 0010	EP01	0 - 1 ft.		8.47	
	DAL SD EP02 0 0030	-	0 - 1 ft.		68.1	
	DAL SD EP02 0 0045	EP02	2.5 - 3 ft.		116	
	DAL SD EP03 0 0010	 	4.5 - 5 ft.		127	
	DAL SD EP03 0 0030	-	0 - 1 ft.		92.0	
	DAL SD EP03 0 0045	EP03	2.5 - 3 ft.		31.3	
	DAL SD EP04 0 0010	 	4.5 - 5 ft.		26.8	
	DAL SD EP04 0 0030	EP04	0 - 1 ft.		250	_
	DAL SD EP05 0 0010		2.5 - 3 ft.		147	
	DAL SD EP05 0 0030	EP05	0 - 1 ft.		191 (166)	┪
I	OAL SD EP06 0 0010		2.5 - 3 ft.		101	\dashv
D	OAL SD EP07 0 0010	EP06	0 - 1 ft.		93.8	\dashv
D	AL SD EP07 0 0030	Enon	0 - 1 ft.		251	┨
D	AL SD EP07 0 0045	EP07	2.5 - 3 ft.		139	┪
D	AL SD EP08 0 0010	EDOO	4.5 - 5 ft.		115 (94.9)	1
D.	AL SD EP09 0 0010	EP08	0 - 1 ft.		83.7	┨
D,	AL SD EP09 0 0030	Thee	0 - 1 ft.		125	1
D	AL SD EP09 0 0045	EP09	2.5 - 3 ft.		67.1	1
\mathbf{D}_{ℓ}	AL SD EP10 0 0010		4.5 - 5 ft.		48.7	1
DA	AL SD EP10 0 0030	EDIO	0 - 1 ft.		167	1
DA	AL SD EP10 0 0045	EP10	2.5 - 3 ft.		62.1	1
DA	AL SD EP11 0 0010		4.5 - 5 ft.		62.0	ł
DA	L SD EP11 0 0030	EP11	0 - 1 ft.		191	1
DÀ	L SD EP12 0 0010		2.5 - 3 ft.	9	93.5 (112)	1
DA	L SD EP13 0 0010	EP12	0 - 1 ft.		40.7	
DA	L SD EP13 0 0030	EP13	0 - 1 ft.		169	
DA	L SD EP14 0 0010		2.5 - 3 ft.		91.2	
DAI	SD WP01 0 0010	EP14	0 - 1 ft.	2	08 (194)	
DAI	CID IXIDaa	WP01 WP02	0 - 1 ft.		ND	
	~ *** *** ** *** *** *** *** *** *** *	W P02 1	0 - 1 ft.		ND	

ND = Not detected at or above Limit of Quantitation.
Values in parentheses are field duplicate results.











APPENDIX TO ATTACHMENT 1 AQUATIC SAMPLING TECHNICAL PROGRESS REPORT

Hillian carding and

INTERIM REPORT 1 – ANALYSIS OF DECATUR SURFACE WATER AND SEDIMENT SAMPLES

The second The street The second second

INTERIM REPORT #1 - Analysis of Decatur Surface Water and Sediment Samples

STUDY TITLE

Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Decatur Monitoring Program

DATA REQUIREMENTS

EPA TSCA Good Laboratory Practice Standards 40 CFR 792

STUDY DIRECTOR

Jaisimha Kesari P.E., DEE Weston Solutions, Inc. 1400 Weston Way West Chester, PA 19380 Phone: 610-701-3761

INTERIM REPORT COMPLETION DATE

February 28, 2008

TESTING FACILITY

MPI Research, Inc. 3058 Research Drive State College, PA 16801 Phone: 814-272-1039

STUDY SPONSOR

3M Company 3M Building 42-02-E-27 St. Paul, MN 55144 Phone: 651-778-5200

PROJECT

MPI Research Study Number: 0137.0219 ExyLIMS Protocol Number: P0003267

Total Pages: 101

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

ExyLIMS Protocol Number P0003267, entitled "Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Decatur Monitoring Program," conducted for 3M Company, is being performed in compliance with EPA TSCA Good Laboratory Practice Standards 40 CFR 792 by MPI Research, Inc, with the following exception:

The primary standard SP0008065 was not characterized according to EPA TSCA GLP 40 CFR 792.

Karen Risha

Principal Investigator MPI Research, Inc.

02/28/08 Date

Jaisimha Kesari P.E., DEE

Study Director

Weston Solutions, Inc.

3/3/08 Date /

Michael A. Saptoro

Sponsor Representative

3M Company

3/6/0B

QUALITY ASSURANCE STATEMENT

MPI Research's Quality Assurance Unit reviewed ExyLIMS Protocol Number P0003267, entitled, "Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Decatur Monitoring Program". All reviewed phases were inspected for conduct according to MPI Research's Standard Operating Procedures, the Study Protocol, the Study Method, and all applicable Good Laboratory Practice Standards. All findings were reported to the MPI Principal Investigator, Management and to the Study Director.

Phase	Date Inspected	Date Reported to Principal <u>Investigator</u>	Date Reported to MPI Management	Date Reported to Study Director
Sample Preparation	07/02/07	07/02/07	07/02/07	07/02/07
Raw Data and Draft Report Review	08/08/07	08/13/07	08/13/07	08/13/07
Raw Data and Final Report Review	12/10/07	12/11/07	12/11/07	12/11/07

Lynann Porter $\frac{\partial -\partial 8 - C}{\partial a_{1}}$

Quality Assurance Research Group Leader, Quality Assurance Unit

Note: All in-lab inspections and the protocol review will be documented in the QA statement for the final analytical report at the conclusion of the study. This QA statement involves only the review of the interim report and associated raw data.

CERTIFICATION OF AUTHENTICITY

This interim report, for ExyLIMS Protocol Number P0003267, is a true and complete representation of the raw data.

Submitted by: MPI Research, Inc. 3058 Research Drive State College, PA 16801 (814) 272-1039 Principal Investigator, MPI: Manager Analytical MPI Research, Inc. MPI Research Facility Management: Kevin Lloyd General Manager, Analytical Sciences MPI Research, Inc. Study Difector, Weston Solutions, Inc. Jaisimha Kesari P.E., DEE Weston Solutions, Inc. Sponsor Representative, 3M Company: Michael A. Santoro

MPI Research

3M Company

Director of Regulatory Affairs

Page 4 of 101

STUDY IDENTIFICATION

Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Decatur Monitoring Program

PROTOCOL NUMBER:

P0003267

MPI STUDY NUMBER:

0137.0219

TYPE OF STUDY:

Residue

SAMPLE MATRIX:

Surface Water and Sediment

TEST SUBSTANCES:

Perfluorooctanoic Acid (PFOA)

SPONSOR:

3M Company

3M Building 42-02-E-27

St. Paul, MN 55144

STUDY DIRECTOR:

Jaisimha Kesari P.E., DEE

Weston Solutions, Inc. 1400 Weston Way

West Chester, PA 19380

STUDY MONITOR:

Michael A. Santoro

3M Company

3M Building 0236-01-B-10

St. Paul, MN 55144

TESTING FACILITY:

MPI Research, Inc.

3058 Research Drive

State College, PA 16801

ANALYTICAL PHASE

TIMETABLE:

Study Initiation Date:

06/14/07

Interim Analytical Start Date:

06/19/07

Interim Analytical Termination Date: 11/09/07

Interim Report Completion Date:

02/28/08

PROJECT PERSONNEL

The Study Director for this project is Jaisimha Kesari at Weston Solutions, Inc. The following personnel from MPI Research, Inc. were associated with various phases of this interim portion of the study:

Name	<u>Title</u>
Karen Risha	Manager Analytical, Principal Investigator
Christine Edwards	Project Leader, Industrial Analysis
Krista Gallant	Research Chemist Associate 1
Ellen Dashem	Research Chemist Associate 1
Stacey Orso	Research Chemist Associate 1
Nancy Saxton	Research Chemist Associate 1
Mark Ammerman	Project Leader, Sample Control
Eric Edwards	Sample Custodian 2

TABLE OF CONTENTS

TITLE PAGE	ıge
GOOD LABORATORY PRACTICE COMPLIANCE CONTRIBUTION	1
QUALITY ASSURANCE STATEMENT	2
CERTIFICATION OF AUTHENTIQUES	3
STUDY IDENTIFICATION	.4
PROJECT PERSONNEI	5
TABLE OF CONTENTS	.6
LIST OF TARLES	7
LIST OF FIGURES	. 8
LIST OF APPENDICES	.9
1.0 SUMMARY	10
2.0 OBJECTIVE	1
3.0 INTRODUCTION	2
4.0 ANALYTICAL TEST SAMOLES	2
5.0 REFERENCE MATERIAL	2
6.0 DESCRIPTION OF ANALYTICAL ACCUMENTS	3
6.1 Extraction Procedure for Symfon W.	3
6.2 Extraction Procedure for Sediment	4
6.3 Preparation of Standards and Fortification 2	4
6.4 Chromatography 14	4
6.5 Instrument Sensitivity	5
6.6 Description of 1 C/MS/MS I	5
6.7 Quantitation and Example Calculation	5
.0 EXPERIMENTAL DESIGN	5
.0 RESULTS	3
.0 CONCLUSION)
0.0 RETENTION OF DATA AND SAMPLES)
20	

Interim Report #1 - Analysis of Decatur Surface Water and Sediment Samples

MPI Study No.: 0137.0219 ExyLIMS Protocol No.: P0003267

LIST OF TABLES

Table I.	Summary of PFOA in Surface Water Samples	Page
Table II.	Summary of PFOA in Re-Extracted Surface Water Samples	23
Table III.	Summary of PFOA in Sediment Samples	24
Table IV.	Matrix Spike Recovery Summary of PFOA in Surface Water Samples	25
Table V.	Matrix Spike Recovery Summary of PFOA in Re-Extracted Surface Wat	
Table VI.	Matrix Spike Recovery Summary of PFOA in Sediment Samples	26
Table VII.	Total Percent Solids for Sediment Samples	21

LIST OF FIGURES

Figure 1	Typical Non-Extracted Calibration Curve for PFOA in 50:50 Non-Extracted Calibration Curve for PFOA in 50:50	<u>Page</u>
Figure 2	Non-Extracted Standards of PFOA in 50:50 Acctonitrile: Water, 0.0125 ng/mL and 0.025 ng/mL, Respectively	
Figure 3.	PFOA in a Reagent Blank, a 0.25 ng/mL Fortified Reagent Spike A, and a 2.5 ng/mL Fortified Reagent Spike B, Respectively	
Figure 4.	Chromatogram Representing a Surface Water Sample Analyzed for PFOA (ExyLIMS ID: C0227212, Data Set: 062007A)	
Figure 5.	PFOA in a Control Blank, a 2.0 ng/g Fortified Control Spike A, and a 20 ng/g Fortified Control Spike B, Respectively	
Figure 6.	Chromatogram Representing a Sediment Sample Analyzed for PFOA (ExyLIMS ID: C0226986, Data Set: 062507G)	
Figure 7.	Typical Non-Extracted Calibration Curve for PFOA Confirmation Ion in	
Figure 8.	Non-Extracted Standards of PFOA Confirmation Ion in 50:50 Acetonitrile: Water, 0.0125 ng/mL and 0.025 ng/mL, Respectively	
Figure 9.	PFOA Confirmation Ion in a Reagent Blank, a 0.25 ng/mL Fortified Reagent Spike A, and a 2.5 ng/mL Fortified Reagent Spike B,	
Figure 10.	Chromatogram Representing a Surface Water Sample Analyzed for PFOA Confirmation Ion (ExyLIMS ID: C0227212, Data Set: 062007A)	
	Spike A, and a 20 ng/g Fortified Control Spike B. Respectively.	
Figure 12.	Chromatogram Representing a Sediment Sample Analyzed for PFOA Confirmation Ion (ExyLIMS ID: C0226986, Data Set: 062507G)	

Interim Report #1 - Analysis of Decatur Surface Water and Sediment Samples

MPI Study No.: 0137.0219 ExyLIMS Protocol No.: P0003267

LIST OF APPENDICES

	<u>Page</u>
Appendix A	Study Protocol45

1.0 SUMMARY

MPI Research, Inc. successfully extracted and analyzed surface water and sediment samples for the determination of perfluorooctanoic acid (PFOA) according to 3M Environmental Laboratory Method ETS-8-012 (V0003400) (Appendix A, pg. 60).

Table I. The nominal LOQ for the method for surface water samples are listed in Table I. The nominal LOQ for the method for surface water samples was 0.025 ng/mL. The limit of quantitation (LOQ) for the analyte in the sediment samples are listed in Tables III. The target LOQ for the method for sediment samples was 0.20 ng/g. After evaluation of the reagent blanks (method blanks) used for the analysis, the LOQ was determined. In some cases, the LOQ was raised due to the evaluation. A discussion of the process used to evaluate the reagent blanks can be found in section 6.4 of the report. In instances where raising the LOQ resulted in a non-detected sample result, the sample was re-extracted to obtain a lower LOQ. The LOQ for the analyte in the re-extracted surface water samples are listed in Table II. The nominal LOQ for the method for re-extracted surface water samples was 0.025 ng/mL.

Analytical results and assessed accuracies for the analysis of PFOA found in the surface water samples are summarized in Table I. Fortification recoveries for PFOA in the surface water samples are detailed in Table IV. The average percent recovery \pm standard deviation for PFOA in the surface water samples was $96 \pm 17\%$. Analytical results and assessed accuracies for the analysis of PFOA found in the re-extracted surface water samples are summarized in Table II. Fortification recoveries for PFOA in the re-extracted surface water samples are detailed in Table V. The average percent recovery \pm standard deviation for PFOA in the surface water samples was $102 \pm 2\%$. Analytical results and assessed accuracies for the analysis of PFOA found in the sediment samples are summarized in Table III. Fortification recoveries for PFOA in the sediment samples are detailed in Table VI. The average percent recovery \pm standard deviation for PFOA in the sediment samples are detailed in Table VI. The average percent recovery \pm standard deviation for PFOA in the sediment samples are detailed in Table VI. The average percent recovery \pm standard deviation for PFOA in the sediment samples was $106 \pm 12\%$.

The assessed accuracy for the majority of the samples reported is +/- 30%. The accuracies were assessed for each sample by reviewing the matrix spike whose spiking level most closely matches the endogenous concentration found in the sample. Several surface water samples had raised LOQ values due to the reagent blank evaluation. In instances where the LOQ was raised and the sample result was non-detected, the sample was re-extracted to obtain quantitative results. In instances where the LOQ was raised for a quantitated sample, an expanded assessed accuracy of +/- 50% is being reported.

Total percent solid results for the sediment samples are detailed in Table VII.

2.0 OBJECTIVE

The objective of the analytical part of this study was to determine levels of perfluorooctanoic acid (PFOA) in surface water and sediment according to Protocol P0003267 (Appendix A).

3.0 INTRODUCTION

This report details the results of the analysis for the determination of PFOA in surface water and sediment using the 3M Environmental Laboratory analytical method ETS-8-012.1 (V0003400) entitled, "Method of Analysis for the Determination of Perfluorobutanoic Acid (PFBA), Perfluoropentanoic Acid (PFPeA), Perfluorohexanoic Acid (PFHA), Perfluorohexanoic Acid (PFHA), Perfluorooctanoic Acid (PFOA), Perfluorononanoic Acid (PFNA), Perfluorodecanoic Acid (PFDA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Water, Soil and Sediment by LC/MS/MS."

The study was initiated on June 14, 2007, when the study director signed protocol number P0003267. The analytical start date for this interim report was June 19, 2007, and the analytical termination date for this interim report was November 9, 2007.

4.0 ANALYTICAL TEST SAMPLES

A total of sixty-four samples (ExyLIMS ID C0226986 – C0227003, C0227200 – C0227234, and C0227532 – C0227542, from login ID L00010412), forty surface waters and twenty-four sediments, were received on wet ice on December 20, 2006 from Charles Young at Weston Solutions, Inc. The forty surface water samples represented three rinse blanks, one trip blank, two associated trip blank field spikes, and eight surface water sites with their associated field spikes. All samples were logged in by MPI personnel and placed in refrigerated storage.

Sample identification (ID) codes for the surface water and sediment samples are of the form Dxx-Sx-LOCxxx-x(x)-06012xx and are composed of the strings described below:

The first string defines the sampling area where D indicates the Decatur, Alabama general study area and L3 = LOI Location 3 at Mallard Point Park, L2 = LOI Location 2 at Swan Creek mouth, L1 = LOI Location 1 at Fox Creek mouth, MC = Mallard Creek mouth, BC = Bakers Creek mouth, OU = 3M outfall cove, and AA = Avenue A drainage.

The second string defines the sample matrix where SW = surface water and SD = sediment.

Interim Report #1 - Analysis of Decatur Surface Water and Sediment Samples

MPI Study No.: 0137.0219 ExyLIMS Protocol No.: P0003267

The third string indicates the specific sampling location.

The fourth string describes the sample aliquot where 0 = primary sample volume, DB = duplicate sample volume, LS = low spike, MS = mid spike, HS = high spike, and RB = equipment rinseate blank.

The final string is the sample collection date in YYMMDD format.

Sample log-in and chain of custody information is located in the raw data package associated with this interim report. Storage records will be kept at MPI Research, Inc. (State College).

5.0 REFERENCE MATERIAL

The requisition information, lot, purity, and expiration date for the reference material used in this study is listed below. The reference material was stored refrigerated.

Compound	ExyLIMS Inventory No.	Supplier	Lot#	Purity (%)	Expiration Date	Received
PFOA	SP0008065	Oakwood Products, Inc.	Y16G	98	No Definitive Expiration	<u>Date</u> 09/08/06

The molecular structure of the standard is given below:

PFOA

Chemical Name: Perfluorooctanoic acid

Molecular Weight: 414

Transitions Monitored: 413 → 369

 $413 \rightarrow 219$

Structure:

$$F \xrightarrow{F} F \xrightarrow{F} F \xrightarrow{F} O$$

$$F \xrightarrow{F} F \xrightarrow{F} F \xrightarrow{F} F$$

$$OH$$

6.0 DESCRIPTION OF ANALYTICAL METHOD

The 3M Environmental Laboratory analytical method ETS-8-012.1 (V0003400) entitled, "Method of Analysis for the Determination of Perfluorobutanoic Acid (PFBA), Perfluoropentanoic Acid (PFPeA), Perfluorohexanoic Acid (PFHA), Perfluorohexanoic

Acid (PFHpA), Perfluorooctanoic Acid (PFOA), Perfluorononanoic Acid (PFNA), Perfluorodecanoic Acid (PFDA), Perfluoroundecanoic Acid (PFDA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Water, Soil and Sediment by LC/MS/MS" was used for the sample analysis in this study.

6.1 Extraction Procedure for Surface Water

A 10 mL aliquot of the water sample was used for the extraction procedure. The sample was measured into a 50 mL polypropylene centrifuge tube. The appropriate samples were fortified and 10 mL of acetonitrile was added. The samples were capped tightly and shaken. The samples were placed into an ultrasonic bath at room temperature for ~2 hours. The samples were then centrifuged at ~3000 rpm for 10 minutes. A portion of the supernate was then transferred to an autosampler vial. Each sample was analyzed by LC/MS/MS electrospray.

6.2 Extraction Procedure for Sediment

A 1 gram aliquot of the soil sample was used for the extraction procedure. The sample was weighed into a 15 mL polypropylene centrifuge tube. The appropriate samples were fortified and 8 mL of 80:20 acetonitrile:water was added. The samples were capped tightly and shaken. The samples were placed into an ultrasonic bath at room temperature for ~2 hours. The samples were then centrifuged at ~3000 rpm for 10 minutes. A portion of the supernate was then transferred to an autosampler vial. Each sample was analyzed by LC/MS/MS electrospray.

6.3 Preparation of Standards and Fortification Solutions

A stock standard solution was prepared as specified in the method. The stock standard solution was prepared at a concentration of 10,000 $\mu g/mL$ by dissolving 1.0 g of the standard (corrected for purity and salt content, if necessary) in acetonitrile. From that solution, a 1000 $\mu g/mL$ fortification standard solution was prepared by taking 10 mL of the stock and bringing the volume up to 100 mL with acetonitrile. By taking 10 mL of the 1000 µg/mL fortification standard and bringing the volume up to 100 mL with acetonitrile, a 100 $\mu g/mL$ fortification standard was prepared. By taking 10 mL of the 100 µg/mL fortification standard and bringing the volume up to 100 mL with acetonitrile, a 10 $\mu g/mL$ fortification standard was prepared. By taking 10 mL of the 10 $\mu g/mL$ fortification standard and bringing the volume up to 100 mL with acetonitrile, a 1.0 μg/mL fortification standard was prepared. By taking 10 mL of the 1.0 µg/mL fortification standard and bringing the volume up to 100 mL with acetonitrile, a 0.1 $\mu g/mL$ fortification standard was prepared. By taking 10 mL of the 0.1 $\mu g/mL$ fortification standard and bringing the volume up to 100 mL with acetonitrile, a 0.01 μg/mL fortification standard was prepared.

A set of external calibration standards were prepared in 50:50 acetonitrile:water. The following concentrations were prepared:

Conc. of Fort.	Aliquot	Final Volume	Eight Co. C.
Solution	Volume	of	Final Conc. of
(ng/mL)	(mL)	Solution (mL)	Calibration Std.
100	5.0	100	(ng/mL)
100	2.5	100	5.0
100	1.0	100	2.5 1.0
5.0	10	100	0.50
2.5	10	100	0.30
1.0	10	100	0.10
0.5	10	100	0.05
0.25	10	100	0.025

The stock standard solution and the 1000 $\mu g/mL$ standard solution were stored in a freezer (-20° \pm 5°C) when not in use. All other fortification and calibration standard solutions were stored in a refrigerator (4° \pm 2°C) when not in use. Documentation of standard preparation is located in the raw data package associated with this interim report.

6.4 Chromatography

Quantification of the analyte was accomplished by LC/MS/MS electrospray. The retention time of PFOA was 3.9 minutes. Method blanks prepared for each data set were used to determine the LOQ. In instances where there were no peaks in the method blanks, the LOQ was determined by the concentration of the lowest standard injected in the analytical run that met the 70–130% recovery range of its known value. In instances where there were peaks detected in the method blanks, the blanks were evaluated. If the average of the responses of all the method blanks was less than 50 % of the response of the lowest standard meeting the recovery criteria, then the LOQ was determined by the lowest standard. If the average of the responses of all the method blanks was greater than 50 % of the response of the lowest standard meeting the recovery criteria, then the LOQ was raised to the standard that met the less than 50 % criteria.

6.5 Instrument Sensitivity

The smallest standard amount injected during the chromatographic run had a concentration of 0.0125 ng/mI. for the surface water samples, and a concentration of 0.025 ng/mL for the sediment samples.

6.6 Description of LC/MS/MS Instruments and Operating Conditions

Instruments:

API 5000 Biomolecular Mass Analyzer

Interface:

SCIEX Turbo Ion Spray Liquid Introduction Interface

Computer:

DELL Precision 360

DELL OptiPlex GX400

Software:

PE SCIEX Analyst 1.4.1

HPLC:

Hewlett Packard (HP) Series 1200 Hewlett Packard (HP) Series 1100

HP Quat Pump

HP Vacuum Degasser

HP Autosampler HP Column Oven

HPLC Column: Phenomenex Luna C8 (2) Mercury, 2cm x 4 mm, 3μm

Column Temp.: ~35° C Injection Vol.:

 $10 \mu L$

Mobile Phase (A): 2 mM Ammonium Acetate in water

Mobile Phase (B): Methanol

Gradient:

Time (min)	<u>% A</u>	<u>% B</u>
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

Total run time: ~10 min

Flow Rate:

0.75 mL/min

Ions monitored:

TOTAL INCIDIO	u.		
<u>Analyte</u>	Mode	Transition	Retention Time
		<u>Monitored</u>	(min)
PFOA	negative	$413 \rightarrow 369$	~3.9 min.
PFOA			
Confirmation	negative	413 → 219	~3.9 min.
Ion			

6.7 Quantitation and Example Calculation

Ten microliters of sample or calibration standard was injected into the LC/MS/MS. The peak area was measured and the standard curve was generated (using 1/x fit weighted linear regression) by Analyst software using eight or nine concentrations of standards. The concentration was determined from the following equations.

Interim Report #1 - Analysis of Decatur Surface Water and Sediment Samples

MPI Study No.: 0137.0219 ExyLIMS Protocol No.: P0003267

Equation 1 calculated the amount of analyte found (in ng/mL, based on peak area) using the standard curve (linear regression parameters) generated by the Analyst software

Equation 1:

Analyte found $(ng/mL) = (Peak area - intercept) \times EDF \times PEDF$ slope

Where: EDF = Extraction Dilution Factor, factor by which the sample volume was diluted during the extraction (EDF =2 for water samples and EDF =1 for soil samples).

PEDF = Post Extraction Dilution Factor, factor by which the final volume was diluted, if necessary.

For the sediment samples, equation 2 was used to convert the amount of analyte found in ng/mL to ng/g (ppb).

Equation 2:

Analyte found (ppb) = [analyte found (ng/mL) x volume extracted (8 mL)] sample weight (1 g)

Equation 3 was then used to calculate the amount of analyte found in ppb based on dry

Equation 3:

Analyte found (ppb) dry weight = analyte found (ppb) x [100% / total solids(%)]

NOTE: Total solids (%) = [dry weight (g) / wet weight (g)] \times 100%

For samples fortified with known amounts of analyte prior to extraction, Equation 4 was used to calculate the percent recovery.

Equation 4:

For water samples:

Recovery (%) =

(total analyte found (ng/mL) - average analyte in sample (ng/mL)) ×100% analyte added (ng/mL)

For sediment samples (based on wet weight):

Recovery (%) =

(total analyte found (ng/g) - average analyte in sample (ng/g)) ×100% analyte added (ng/g)

Interim Report #1 - Analysis of Decatur Surface Water and Sediment Samples

MPI Study No.: 0137.0219 ExyLIMS Protocol No.: P0003267

An example of a calculation using an actual sample follows:

Sediment sample Exygen ID: C0226989 Spike C (Set: 062507H), fortified at 2.0 ng/g with PFOA where:

peak area 48526 intercept 3170 slope 167000 extraction dilution factor 1 post extraction dilution factor 1 ng/g PFOA added (fort level) 2.0 ng/g average amt in corresponding sample = ND (not detected) total percent solid 48.16 %

From equation 1:

Analyte found (ng/mL) = $[48526 - 3170] \times 1 \times 1$ 167000= 0.272 ng/mL

From equation 2:

Analyte found, wet weight (ng/g) = $(0.272 \text{ ng/mL} \times 8 \text{ mL})$ = 2.18 ng/g

From equation 3:

Analyte found (ng/g, ppb) dry weight = $2.18 \text{ ng/g} \times [100\% / 48.16\%]$

= 4.53 ng/g

From equation 4:

% Recovery = $\frac{(2.18 \text{ ng/g})}{2.0 \text{ ng/g}} \times 100\%$ = $\frac{109\%}{2.0 \text{ ng/g}}$

NOTE: Numbers may differ slightly from raw data due to rounding.

7.0 EXPERIMENTAL DESIGN

For water samples designated as field matrix spikes, PFOA was added at a known concentration to the bottles in the laboratory before being shipped to the field. The samples were filled to a 200 mL volumetric fill line in the field. For the sediment samples designated as laboratory matrix spikes, PFOA was added to the samples after they were aliquotted in the laboratory, before the extraction solvent was added to the samples.

The surface water samples were initially extracted in three sets. The sets included four reagent blanks (method blanks), three reagent blanks fortified at one lower level and three reagent blanks fortified at one higher level of known concentrations. The first two sets contained three surface water sample sites. The last set contained two sample sites, three equipment rinseate blanks, and one trip blank and associated trip blank field spikes. For each site, a sample, a field duplicate and a range of two to three matrix field spikes were collected and extracted.

Two water sample sites were re-extracted in one set. The set included three reagent blanks (method blanks), three reagent blanks fortified at one lower level and three reagent blanks fortified at one higher level of known concentrations. For each of the two water sites in the set, a sample, a field duplicate and two matrix field spikes were re-extracted.

The sediment samples were extracted in eight sets. Each set included four control blanks (method blanks), three control blanks fortified at one lower level and three control blanks fortified at one higher level of known concentrations. All eight sets contained three sample sites. For each sample site, a sample, a laboratory replicate, and three laboratory matrix spikes were prepared and extracted.

8.0 RESULTS

The limit of quantitation (LOQ) for the analyte in the surface water samples are listed in Table I. The nominal LOQ for the method for surface water samples was 0.025 ng/mL. The limit of quantitation (LOQ) for the analyte in the sediment samples are listed in Tables III. The target LOQ for the method for sediment samples was 0.20 ng/g. After evaluation of the reagent blanks (method blanks) used for the analysis, the LOQ was determined. In some cases, the LOQ was raised due to the evaluation. A discussion of the process used to evaluate the reagent blanks can be found in section 6.4 of the report. In instances where raising the LOQ resulted in a non-detected sample result, the sample was re-extracted to obtain a lower LOQ. The LOQ for the analyte in the re-extracted surface water samples are listed in Table II. The nominal LOQ for the method for re-extracted surface water samples was 0.025 ng/mL.

Analytical results and assessed accuracies for the analysis of PFOA found in the surface water samples are summarized in **Table I**. Fortification recoveries for PFOA in the surface water samples are detailed in **Table IV**. The average percent recovery \pm standard deviation for PFOA in the surface water samples was $96 \pm 17\%$. Analytical results and assessed accuracies for the analysis of PFOA found in the re-extracted surface water samples are summarized in **Table II**. Fortification recoveries for PFOA in the re-extracted surface water samples are detailed in **Table V**. The average percent recovery \pm standard deviation for PFOA in the surface water samples was $102 \pm 2\%$. Analytical results and assessed accuracies for the analysis of PFOA found in the sediment samples are summarized in **Table III**. Fortification recoveries for PFOA in the sediment samples

are detailed in Table VI. The average percent recovery \pm standard deviation for PFOA in the sediment samples was $106 \pm 12\%$.

The assessed accuracy for the majority of the samples reported is +/- 30%. The accuracies were assessed for each sample by reviewing the matrix spike whose spiking level most closely matches the endogenous concentration found in the sample. Several surface water samples had raised LOQ values due to the reagent blank evaluation. In instances where the LOQ was raised and the sample result was non-detected, the sample was re-extracted to obtain quantitative results. In instances where the LOQ was raised for a quantitated sample, an expanded assessed accuracy of +/- 50% is being reported.

Total percent solid results for the sediment samples are detailed in Table VII.

9.0 CONCLUSION

The surface water and sediment samples were successfully extracted and analyzed for PFOA according 3M Environmental Laboratory analytical method ETS-8-012.1 (V0003400).

10.0 <u>RETENTION OF DATA AND SAMPLES</u>

All original paper data generated by MPI Research, Inc. (State College) that pertains to this interim report will be shipped to the study director. This does not include facility-specific raw data such as instrument or temperature logs. Exact copies of all raw data, as well as a signed copy of the final analytical report and all original facility-specific raw data, will be retained in the MPI Research, Inc. (State College) archives for the period of time specified in EPA TSCA Good Laboratory Practice Standards 40 CFR 792.

MPI Research

Page 20 of 101

Interim Report #1 - Analysis of Decatur Surface Water and Sediment Samples

MPI Study No.: 0137.0219 ExyLIMS Protocol No.: P0003267

TABLES

Table I. Summary of PFOA in Surface Water Samples

C8 Acid PFOA

		Perfluorooctanoic Acid		
Exygen ID	Client Sample ID	Analyte Found (ppb, ng/mL)	Acceptable LOQ (ng/mL)	Assessed Accuracy (+/- %)
C0227200 C0227201	DL3-SW-LOC001-0-061214	ND	0.025	30
C0227201	DL3-SW-LOC001-DB-061214	ND	0.025	30
C0227204	DL2-SW-LOC001-0-061214	ND	0.025	30
C0227205	DL2-SW-LOC001-DB-061214	ND	0.025	30
C0227208	DBC-SW-LOC001-0-061214	ND	0.025	30
C0227209	DBC-SW-LOC001-DB-061214	ND	0.025	30
C0227212	DOU-SW-LOC001-0-061213	3.65	0.200	50
C0227213	DOU-SW-LOC001-DB-061213	3.43	0.200	50
C0227216	DL1-SW-LOC001-0-061213	NR*	-	
C0227217	DL1-SW-LOC001-DB-061213	NR*		•
C0227220	DMC-SW-LOC001-0-061213	NR*	_	
C0227221	DMC-SW-LOC001-DB-061213	NR*	•	
C0227224	DAA-SW-LOC002-0-061215	1.31	0.025	30
C0227225	DAA-SW-LOC002-DB-061215	1.32	0.025	30
C0227229	DOU-F02-IPF004-RB-061212	ND	0.025	30
C0227230	DL2-SW-LOC001-RB-061214	ND	0.025	30
C0227231	DAA-SD-LOC006-RB-061215	ND	0.025	30
C0227232	Trip Blank	ND	0.025	30
C0227538 C0227539	DAA-SW-LOC005-0-061215	86.0	0.025	30
00227539	DAA-SW-LOC005-DB-061215	86.7	0.025	30

ND = Not detected at or above the acceptable LOQ.

NR* = Not reported due to elevated LOQ; see Table II for re-extracted sample results.

Summary of PFOA in Re-Extracted Surface Water Samples Table II.

C8 Acid PFOA

		Perfluorooctanole Acid		
Exygen ID	Client Sample ID	Analyte Found (ppb, ng/mL)	Acceptable LOQ (ng/mL)	Assessed Accuracy
C0227216 C0227217	DL1-SW-LOC001-0-061213 DL1-SW-LOC001-DB-061213	0.0831 0.0697	0.050 0.050	30
C0227220 C0227221	DMC-SW-LOC001-0-061213 DMC-SW-LOC001-DB-061213	0.0511 ¹ ND ¹	0.050 0.050	30 30 30

ND = Not detected at or above the acceptable LOQ.

Relative Percent Difference was not calculated due to the presence of a nondetect and resulting uncertainty

Table III. Summary of PFOA in Sediment Samples

C8 Acid PFOA

		н.	C8 Acid PFOA	
		Analyte Found	Accordable	
	Client	(ppb, ng/g)	Acceptable LOQ	Assessed Accuracy
Exygen ID	Sample ID	Dry Weight	(ng/g)	(+/- %)
C0228986	DL3-SD-LOC001-0-061214			
C0226986 Rep	DL3-SD-LOC001-0-061214*	ND ND	0.20	30
C0226987	DL3-SD-LOC002-0-061214	"	0.20	30
C0226987 Rep	DL3-SD-LOC002-0-061214*	ND	0.20	30
C0226988		ND	0.20	30
C0226988 Rep	DL3-SD-LOC003-0-061214 DL3-SD-LOC003-0-061214*	ND	0.20	30
C0226989		ND	0.20	30
C0226989 Rep	DL2-SD-LOC001-0-061214 DL2-SD-LOC001-0-061214*	ND	0.20	30
		ND	0.20	30
C0226990 C0226990 Rep	DL2-SD-LOC002-0-061214	ND	0.20	30
-•	DL2-SD-LOC002-0-061214*	ND	0.20	30
C0226991 C0226991 Rep	DL2-SD-LOC003-0-061214	ND	0.20	30
	DL2-SD-LOC003-0-061214*	NO	0.20	30
C0226992	DBC-SD-LOC001-0-061214	5.60	0.20	30
C0226992 Rep	DBC-SD-LOC001-0-061214*	4.70	0.20	30
C0226993	DBC-SD-LOC002-0-061214	4.98	0.20	30
C0226993 Rep	DBC-SD-LOC002-0-061214*	4.32	0.20	30
C0228994	DBC-SD-LOC003-0-061214	3.98	0.20	30
C0226994 Rep	DBC-SD-LOC003-0-061214*	3.44	0.20	30
C0226995	DOU-SD-LOC001-0-061213	23.9^	0.20	30
C0226995 Rep	DOU-SD-LOC001-0-061213*	54.6^	0.20	30
C0225996	DOU-SD-LOC002-0-061213	8.61	0.20	30
C0226996 Rep	DOU-SD-LOC002-0-061213*	8.65	0.20	30 30
C0226997	DOU-SD-LOC003-0-061213	8.59	0.20	
C0226997 Rep	DOU-SD-LOC003-0-061213*	9.16	0.20	30 30
C0228998	DL1-SD-LOC001-0-061213	0.586	0.20	
C0226998 Rep	DL1-SD-LOC001-0-061213*	0.485	0.20	30 30
C0226999	DL1-SD-LOC002-0-061213	1.104		
C0226999 Rep	DL1-SD-LOC002-0-081213*	0.463^	0,20 0.20	30 30
C0227000	DL1-SD-LOC003-0-081213	ND		
C0227000 Rep	DL1-SD-LOC003-0-061213*	I ND	0.20 0.20	30
C0227001	DMC-SD-LOC001-0-061213	ND¹		30
C0227001 Rep	DMC-SD-LOC001-0-061213*	0.505	0.20 0.20	30
C0227002	DMC-SD-LOC002-0-061213	D.602		30
C0227002 Rep	DMC-SD-LOC002-0-061213*	0.658	0.20 0.20	30
C0227003	DMC-SD-LOC003-0-061213			30
C0227003 Rep	DMC-SD-LOC003-0-061213*	0.532 ⁴ 0.873 ⁴	0.20	30
C0227532	DAA-SD-LOC005-0-061215		0.20	30
C0227532 Rep	DAA-SD-LOC006-0-061215*	43,3 46.1	0.20	30
C0227533	DAA-SD-LOC005-0-081215	[0.20	30
C0227533 Rep	DAA-SD-LOC005-0-061215*	45.3 51.2	0.20	30
C0227534	DAA-SD-LOC004-0-061215		0.20	30
C0227534 Rep	DAA-SD-LOC004-0-061215*	537	0.20	30
C0227535		574	0.20	30
C0227535 Rep	DAA-SD-LOC003-0-061215 DAA-SD-LOC003-0-061215*	116	0.20	30
C0227536	i	110	0.20	30
C0227536 Rep	DAA-SD-LOC002-0-061215 DAA-SD-LOC002-0-061215*	3.78*	0.20	30
C0227537		2.22 ⁿ	0.20	30
C0227537 Rep	DAA-SD-LOC001-0-081215 DAA-SD-LOC001-0-061215*	4.33	0.20	30
	- 0. 00 E0000 (-0-001215)	3.53	0.20	30

^{*}Laboratory Duplicate

[^]Relative Percent Difference > 30%

Relative Percent Difference was not calculated due to the presence of a nondetect and resulting uncertainty.

ND = Not detected at or above the acceptable LOQ.

Table IV. Matrix Spike Recovery Summary of PFOA in Surface Water Samples

C8 Acid PF(AC
-------------	----

	-	•	C8 Acid PFOA Perfluoroactanoic Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered	Recovery
DL3-SW-LOC001-LS-061214		1	(ng/mL)	(%)
(C0227202, 0 25 ppb Field Spike) DL3-SW-LOC001-HS-061214 (C0227203, 5.0 ppb Field Spike)	0.25	ND	0.204	82
	5.0	ND	NA	NA
DL2-SW-LOC001-LS-061214 (C0227206, 0.25 ppb Field Spike)	0.25	ND		naca.
DL2-SW-LOC001-HS-061214 (C0227207, 5.0 ppb Field Spike)	5.0		0.206	82
DBC-SW-LOC001-LS-061214 (C0227210, 0.25 ppb Field Spike)	0.5	ND	NA	NA
DBC-SW-LOC001-HS-051214	0.25	ND	0.302	121
(C0227211, 5.0 ppb Field Spike) DOU-SW-LOC001-LS-061213	5.0	ND	NA	NA
DOU-SW-LOC001-HS-061212	0.25	3.54	NA	NA
(C0227215, 5.0 ppb Field Spike)	5.0	3.54	7,52	
DL1-SW-LOC001-LS-061213 (C0227218, 0.26 ppb Field Spike)	0.25	NR*	- 100	80
DL1-SW-LOC001-HS-061213 (C0227219, 5.0 ppb Fleid Spike)	5.0		NR*	NR*
DMC-SW-LOC001-I S-081212	J.Q	NR*	NR*	NR*
(C0227222, 0.25 ppb Field Spike) DMC-SW-LOC001-HS-061213	0.25	NR*	NR*	NR*
(C0227223, 5.0 ppb Field Spike) DAA-SW-L OC002-LS-061215	5.0	NR*	NR*	NR*
(C0227226, 0.25 ppb Fleid Spike)	0.25	1.32	*	
DAA-SW-LOC002-MS-061215 (C0227227, 5.0 ppb Field Spike)	5.D	1.32	NA	NA
DAA-SW-LOC002-HS-061215 (C0227228, 100 ppb Field Spike)	100		5.72	88
Trip Blank Low Spike (C0227233, 0.25 ppb Fleid Spike)		1.32	NA	NA
Trip Blank High Spike	0.25	ND	0.290	116
(C0227234, 5.0 ppb Field Spike) DAA-SW-LOC005-LS-061215	5.0	ND	NA	NA
(C0227\$40, 0.25 ppb Fleid Splke)	0.25	86.4	N/A	· •
AA-SW-LOC005-MS-061215 (C0227641, 5.0 ppb Field Spike)	5.0	86.4	NA	NA
AA-SW-LOC005-HS-061215 (C0227842, 100 ppb Field Spike)	100		NA	NA
	100	86.4	192	106

Average: 96 Standard Deviation:

ND = Not detected at or above the acceptable LOQ reported in Table I.

NA = Not applicable. This matrix spike concentration was not used to assess the accuracy for this analyte.

NR* = Not reported due to elevated LOQ; see Table V for re-extracted matrix spike results.

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.

Table V. Matrix Spike Recovery Summary of PFOA in Re-Extracted Surface Water Samples

C8 Acid PFOA

			Perfluorooctanoic Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
DL1-SW-LOC001-LS-061213 (C0227218, 0.25 ppb Field Spike)	0.25	0.0764	0.334	
DL1-SW-LOC001-HS-061213 (C0227219, 5.0 ppb Field Spike)	5.0	0.0764	NA	103 NA
DMC-SW-LOC001-LS-061213 (G0227222, 0.25 ppb Field Spike)	0.25	0.0505		
DMC-SW-LOC001-HS-061213 (C0227223, 5.0 ppb Fleid Spike)	5.0	0.0505	0.302	101
		0.0000	NA	NA

Average: 102 Standard Deviation:

ND = Not detected at or above the acceptable LOQ reported in Table II.

NA = Not applicable. This matrix spike concentration was not used to assess the accuracy for this analyte.

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.

Table VI. Matrix Spike Recovery Summary of PFOA in Sediment Samples

			C8 Acid PFOA	
Sample Description	Amoun Spiked (ng/g)	in Sample	Perfluoroctanoic Admount Recovered	Recovery
DL3-SD-LOC001-0-061214 (C0228886 Spk C, 2.0 ppb Spike)		(ng/g) wet wt.	(ng/g) wet wt.	(%)
DL3-SD-LOC001-0-061214 (C0226986 Spk D, 40 ppb Spike)	2.0	ND	2.10	105
DL3-SD-LOC001-0-061214 (C0226986 Spk E, 800 ppb Spike)	800	ND	NA	NA
DL3-SD-LOC002-0-081214 (C0225987 Spk F, 2.0 ppb Spike)	500	ND	NA	NA
DL3-SD-LOC002-0-061214 (C0226887 Spk G, 40 ppb Spike)	2.0	ND	2.08	104
DL3-SD-LOC002-0-061214 (C0226987 Spk H, 800 ppb Spike)	40	ND	NA	NA
DL3-SD-LOC003-0-061214	800	ND	NA	N A
(C0226988 Spk I, 2.0 ppb Spike) DL3-SD-LOC003-0-061214 (C0226988 Spk J, 40 ppb Spike)	2.0	ND	1.91	96
DL3-SD-LOC003-0-061214 (C0226988 Spk K, 800 ppb Spike)	40	ND	NA	NA
DL2-SD-LOC001-D-061214	800	ND	NA	NA
(C0226989 Spk C, 2.0 ppb Spike) DL2-SD-LOC001-0-061214	2.0	ND	2.17	109
(C0225989 Spk D, 40 ppb Spkts) DL2-SD-LOC001-0-061214 (C0226889 Spk E, 800 ppb Spike)	40	ND	NA	· NA
DL2-SD-LOC002-0-061214	800	ND	NA	NA
(C0226990 Spk F, Z.0 ppb Spike) DL2-SD-LOC002-0-061214	2.0	ND	2.21	111
(C0228990 Spk G, 40 ppb Spike) DL2-SD-LOC002-0-061214	40	ND	NA	NA
(C0226960 Spk H, 800 ppb Spike) DL2-SD-LOC003-0-061214	800	ND	NA	NA
(C0226991 Spk I, 2.0 ppb Spike) DL2-SD-LOC003-0-061214	2.0	ND	2.04	102
(C0226991 Spk J, 40 ppb 8pike) DL2-SD-LOC003-0-061214	40	ND	NA	NA NA
C0226991 3pk K, 800 ppb \$pike) DBC-SD-LOC001-0-061214	800	ND	NA	NA.
C0226992 Spk C, 2.0 ppb Spike) DBC-SD-LOC001-0-061214	2.0	2.36	4.74	
C0226992 Spk D, 40 ppb Spike) BC-SD-LOC001-D-051214	40	2.36	NA	119 NA
20226992 Spk E, 600 ppb Spiks)	800	2.36	NA	NA NA

ND = Not detected at or above the acceptable LOQ reported in Table III.

NA = Not applicable. This matrix spike concentration not used to assess the accuracy for this analyte.

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.

Matrix Spike Recovery Summary of PFOA in Sediment Table VI. Samples (continued)

C8 Acid PFOA

		C8 Acid PFOA		
			Perfluorooctanoic Aci	d
Comple	Amount	Amt Found	Amount	
Sample Description	Spiked	in Sample	Recovered	Recovery
Description	(ng/g)	(ng/g)	(ng/g)	(%)
DBC-SD-LOC002-0-061214				
(C0226993 Spk F, 2.0 ppb Spike)	2.0	0.40		
	2.0	2.19	4.04	93
DBC-SD-LOC002-0-061214				
(C0226993 Spk G, 40 ppb Spike)	40	2.19	NA	NA
DBC-SD-LOC002-0-061214				, •• •
(C0226993 Spk H, 800 ppb Spike)	800	2.19	NA	
	}	2.10	NA	NA
DBC-SD-LOC003-0-061214	1			
(C0226994 Spk I, 2.0 ppb Spike)	2.0	1.60	3.53	67
DBC-SD-LOC003-0-061214	1		3.33	97
(C0226994 Spk J, 40 ppb Splke)	40			
	1 ***	1.60	NA	NA
DBC-SD-LOC003-0-061214	1			
(C0726994 Spk K, 800 ppb Spike)	800	1.60	NA	NA
DOLLED LOOPER & COLO.	ſ			****
DOU-SD-LOC001-0-061213				
(C0226995 Spk C, 2.0 ppb Spike)	2.0	26.5	NA	NA
DOU-SD-LOC001-0-061213	1			
(C0226995 Spk D, 40 ppb Spika)	40	26.5	67.5	409
DOU-SD-LOC001-0-061213	į.		u.,	103
(C0226985 Spk E, 800 ppb Spike)	800			
, and an of the chiral	000	26.5	NA	NA
DOU-SD-LOC002-0-061213	1	1		
(C0226996 Spk F, 2.0 ppb Spike)	2.0	4.00		
DOU-SD-LOC002-0-061213	1 ***	4.93	NA	NA
	1			
(C0226996 Spk G, 40 ppb Spike)	40	4.93	49.5	111
DOU-SD-LOC002-0-061213	1	ļ		
(C0226996 Spk H, 800 ppb Spike)	800	4.93	NA	NA
	1		IVA	INA
DOU-SD-LOC003-0-061213	1] .		
(C0226997 Spk I, 2.0 ppb Spike)	2.0	6.95	NA.	NA
DOU-SD-LOC003-0-061213	1		,	19/3
(C0226997 Spk J, 40 ppb Spike)	40	6.95	47.0	
DOU-SD-LOC003-0-061213	1	0.93	47.6	102
(C0Z26997 Spk K, 800 ppb Spike)				
(4-1-1-1-1 aby K; 600 bbn abike)	800	6.95	NA	NA
DL1-SD-LOC001-0-061213				
(C0226998 Spk C, 2.0 ppb Spike)	1 22			•
	2.0	0.286	2.58	115
DL1-SD-LOC001-0-061213]			
(C0226998 Spk D, 40 ppb Spike)	40	0.286	NA	NA
DL1-SD-LOC001-0-061213				(VA
(C0226998 Spk E, 800 ppb Spke)	800	0.286	A t a	4.4
,		0.200	NA	NA
DL1-SD-LOC002-0-061213	1			
(C0226999 Spk F, 2.0 ppb Spike)	2.0	0.352	2.59	440
DL1-SD-LOC002-0-061213		VL	2.40	112
(C0226999 Spk G, 40 ppb Spika)	40	0.050		
] ""	0.352	NA	NA
DL1-SD-LOC002-0-061213	1			
(CD226999 Spk H, 800 ppb Splks)	800	0.352	NA	NA
	L	I		• • • •

ND = Not detected at or above the acceptable LOQ reported in Table III.

NA = Not applicable. This matrix spike concentration not used to assess the accuracy for this analyte.

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.

Table VI. Matrix Spike Recovery Summary of PFOA in Sediment Samples (continued)

			C8 Acid PFOA Perfluorocctanoic Acid		
Sample Description	Amount Spiked (ng/g)	Amt Found in Sample	Amount Recovered	Recovery	
DL1-SD-LOC003-0-061213		<u>(ng/g)</u>	(ng/g)	(%)	
(C0227000 Spk I, 2.0 ppb Spike)		1	· · · · · · · · · · · · · · · · · · ·		
DL1-SD-LOC003-0-061213	2.0	ND	2.44	400	
(C0227000 Spk J, 40 ppb Spike)			,,	122	
	40	ND	NA		
DL1-SD-LOC003-0-061213 (C0227000 8pk K, 800 ppb Spike)			1973	NA	
(Total ood spik K, 800 ppb Spike)	800	ND	NA		
DMC-SD-LOC001-0-061213			NA	NA	
(C0227001 Spk C, 2.0 ppb Spike)	1				
DMC-SD-LOC001-0-061213	2.0	0.266	2.39	100	
(C0227001 Spk D, 40 ppb Spike)	1		-,	106	
DMC PD LOCAL TO THE SPIKE	40	0.266	NA		
DMC-SD-LOC001-0-061213 (C0227001 Spk E, 800 ppb Spike)		1	11/1	NA	
(spike)	800	0.266	N/A		
DMC-SD-LOC002-0-061213	1	}	NA	NA	
(C0227002 Spk F, 2.0 ppb Spike)	1 20				
DMC-SD-LOC002-0-061213	2.0	0.284	2.52	140	
(C0227002 Spk G, 40 ppb Spike)				112	
DMC-SD-LOC002-0-061213	40	0.284	NA		
(C0227002 Spk H, 800 ppb Spike)	}	}	.,,,	NA	
sou ppo Spike)	800	0.284	NA		
DMC-SD-LOC003-0-061213	1		NA	NA	
(C0227003 Spk J. 2.0 ppb Spike)	1 20				
DMC-SD-LOC003-0-061213	2.0	0.310	2.48	109	
(C0227003 Spk J, 40 ppb Spike)	1			108	
DMC-SD-LOC003-0-061213	40	0.310	NA		
(C0227003 Spk K, 800 ppb Spike)				NA	
	800	0.310	NA		
DAA-SD-LOC006-0-061215			103	NA	
(C0227532 Spk C, 2.0 ppb Splke)	2.0				
DAA-SD-LOC006-0-061215	20	32.6	NA	NA	
(C0227632 Spk D, 40 ppb Spike)	40			110	
DAA-SD-LOC006-0-061215	40	32.6	77.8	110	
(C0227632 Spk E, 600 ppb Spike)				113	
	800	32.6	NA	b 1 e	
DAA-SD-LOC005-0-081215	1			NA	
(C0227533 Spk F, 2.0 ppb Spike)	2.0	24.4			
DAA-SD-LOC005-0-061215		34.6	NA	NA	
(C0227533 Spk G, 40 ppb Spike)	40				
DAA-SD-LOC005-0-061215	70	34.6	69.6	88	
(C0227533 Spk H, 800 ppb Splke)	200			5 5	
	800	34.6	NA	\$1. 0	
DAA-SD-LOC004-0-061215	i i			NA	
(C0227634 Spk I, 2.0 ppb Spike)	2.0	150			
DAA-SD-LOC004-0-061215		169	NA	NA	
(C0227534 Spk J, 40 ppb Spike)	40	400		•	
DAA-SD-LOC004-0-061215	~	169	NA	NA	
C0227634 Spk K, 800 ppb Spike)	800			- 37 1	
	600 (169	731		

ND = Not detected at or above the acceptable LOQ reported in Table III.

NA = Not applicable. This matrix spike concentration not used to assess the accuracy for this analyte.

Note: Since this summary table shows rounded results, recovery values may vary elightly from the values in the raw data.

Table VI. Matrix Spike Recovery Summary of PFOA in Sediment Samples (continued)

C8 Acid	PFOA
---------	------

	C8 Acid PFOA Perfluorooctanoic Acid			
Sample Description	Amount Spiked (ng/g)	Amt Found in Sample (ng/g)	Amount Recovered (ng/g)	Recovery (%)
DAA-SD-LOC003-0-061215 (C0227535 Spk C, 2.0 ppb Spike)	2.0	50.3		
DAA-SD-LOC003-0-061215 (C0227535 Spk D, 40 ppb Spike)	40		NA	NA
DAA-SD-LOC003-0-061215		50.3	102	129
(C0227535 Spk E, 800 ppb Spike) DAA-SD-LOC002-0-061215	800	50.3	NA	NA
(C0227535 Spk F, 2.0 ppb Spike)	2.0	2.16	4.15	100
DAA-SD-LOC002-0-061215 (C0227535 Spk G, 40 ppt Spike)	40	2.16	NA	
DAA-SD-LOC002-0-061215 (C0227535 Spk H, 800 ppb Spike)	800	2.16	NA NA	NA NA
DAA-SD-LOC001-0-061215 (C0227536 Spk I, 2.0 ppb Spike)	2.0	2.20		
DAA-SD-LOC001-0-061215 (C0227836 Spk J. 40 ppb Spike)	40	2.20	4.65	123
DAA-SD-LOC001-0-061215 (C0227536 Spk K, 800 ppb Spike)	800		NA	NA
		2.20	NA Average:	NA 106
		Sta	andard Deviation	40

Standard Deviation: 12

ND = Not detected at or above the acceptable LOQ reported in Table III.

NA = Not applicable. This matrix spike concentration not used to assess the accuracy for this analyte.

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.

Table VII. Total Percent Solids for Sediment Samples

Exygen ID	Client Sample ID	Total Percent Solids (%)
C0226986	DL3-SD-LOC001-0-061214	65.16
C0226987	DL3-SD-LOC002-0-061214	43.41
C0226988	DL3-SD-LOC003-0-061214	46.81
C0226989	DL2-SD-LOC001-0-061214	48.16
C0226990	DL2-SD-LOC002-0-051214	46.49
C0226991	DL2-SD-LOC003-0-061214	47.72
C0226992	DBC-SD-LOC001-0-061214	45.84
C0226993	DBC-SD-LOC002-0-061214	47.17
C0226994	DBC-SD-LOC003-0-061214	43.10
C0226995	DOU-SD-LOC001-0-061213	67.54
C0226996	DOU-SD-LOC002-0-061213	57.12
C0226997	DOU-SD-LOC003-0-061213	78.34
C0226998	DL1-SD-LOC001-0-061213	53.47
C0226999	DL1-SD-LOC002-0-061213	45.02
C0227000	DL1-SD-LOC003-0-061213	78.98
C0227001	DMC-SD-LOC001-0-061213	65.71
C0227002	DMC-SD-LOC002-0-061213	45.17
C0227003	DMC-SD-LOC003-0-061213	44.17
C0227532	DAA-SD-LOC006-0-061215	72.89
00227533	DAA-SD-LOC005-0-061215	71.69
0227534	DAA-SD-LOC004-0-061215	30.49
0227535	DAA-SD-LOC003-0-061215	44.39
0227536	DAA-SD-LOC002-0-061215	71.91
0227537	DAA-SD-LOC001-0-061215	56.02