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8EHQ-0406-003746

April 14, 2006

CONTAIN NO CBI
AR 226-3631

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



Re: TSCA 8(e) Substantial Risk Notice: Sulfonate-based and Carboxylic-based
Fluorochemicals, Docket Nos. 8EHQ-1180-374; 8EHQ-0381-0394;
8EHQ-0598-373 - Results from Analysis of Groundwater in the vicinity of former
Oakdale Disposal Site

To whom it may concern:

3M is submitting this notice to supplement its previous submissions on sulfonyl and carboxylic-based fluorochemicals. 3M has recently received the enclosed analytical report (3M Environmental Laboratory Report No. 06-0151) for groundwater samples collected in the vicinity of the former Oakdale Disposal Site (Site) in Oakdale, MN.

From the late 1940's until 1960, a variety of industrial and municipal wastes were disposed of at a property known as the Abresch site in Oakdale, MN. This location is also known as the Oakdale Disposal Site and underwent an extensive site remediation/cleanup in 1983-84. In 1985, a pump-out system was installed to remove and contain solvent-impacted shallow groundwater at the Site. This system has been in continuous operation since installation.

3M has been leading a study, with involvement from the Minnesota Pollution Control Agency (MPCA) and Minnesota Department of Health (MDH), to characterize the presence of fluorochemicals at the Site. In an early phase of this assessment, existing on-site and near site monitoring and pump-out wells were monitored for fluorochemicals. Results from this work are provided in Exygen Study No. P0001400, Interim Report #1, enclosed. A map depicting the locations of these wells is also enclosed as Figure 1. The results from this early work were used as a basis for locating nine additional monitoring wells. Four of these monitoring points are on-site and five are in close proximity to the Site. The location of these wells is provided on Figure 2, enclosed. Results from the first round of sampling for these wells are contained in the aforementioned 3M Environmental Laboratory Report (No. 06-0151).

The new wells were constructed to sample from four different geological strata: alluvium, Platteville limestone, St. Peter sandstone and the Prairie du Chien group. These are depicted on Figure 3 for the Site. Results for four of the five offsite wells are below the limit of quantitation

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(0.025 ng/mL) for all analytes. Levels in the fifth offsite well are significantly less than 1 ng/mL (ppb) for each analyte.

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

If you have any questions, please do not hesitate to contact Gary Hohenstein at (651) 778-5150.

Sincerely,

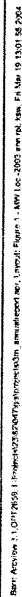
A handwritten signature in cursive script that reads "Katherine E. Reed". The signature is written in dark ink and is positioned above the printed name and title.

Katherine E. Reed
Staff Vice President
Environmental, Health and Safety Operations

Enclosures

000002

AR 226-3632



1000 0 1000 2000 Feet

LEGEND

- Platteville Well

- St. Peter Well

Piume of Isopropyl Ether in Platteville - 2003

Plume of Isopropyl Ether in Platteville - 1984

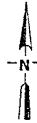


Figure 1

FC MONITORING PROGRAM WELLS

Oakdale Disposal Site
Oakdale, Minnesota



600 0 600 1200 Feet

LEGEND

-  Basal Alluvium Well
 Pump-out Well
 Water Table Well
 Surface Water Sampling Location
 Inferred Limits of Abresch Disposal Site Area
 Former Pump-Out Well, Converted to a Monitoring Well

Source: Digital Aerial Photos, Metropolitan Council, 2000 & Barr Engineering Co.

05P-0198-10

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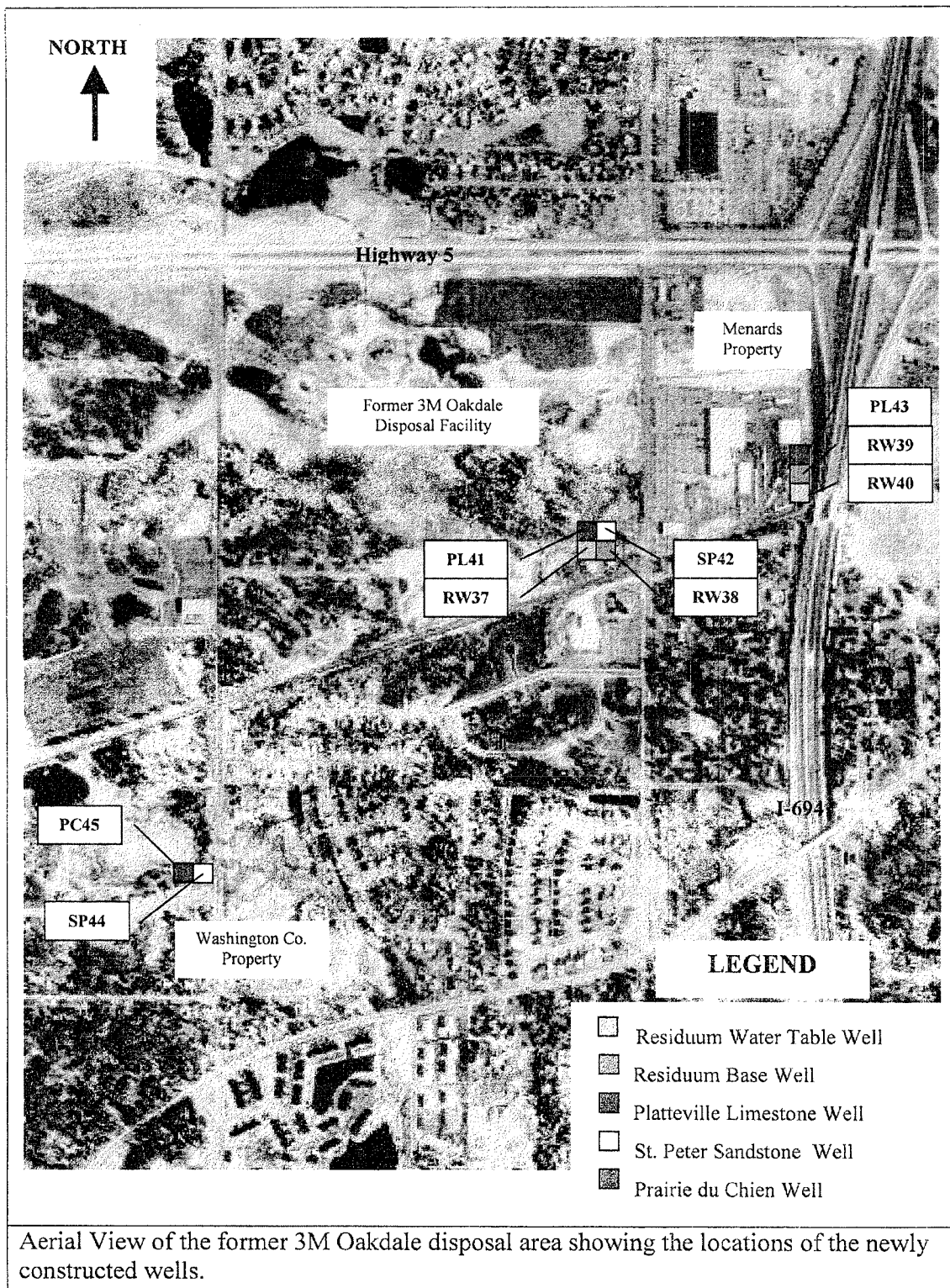
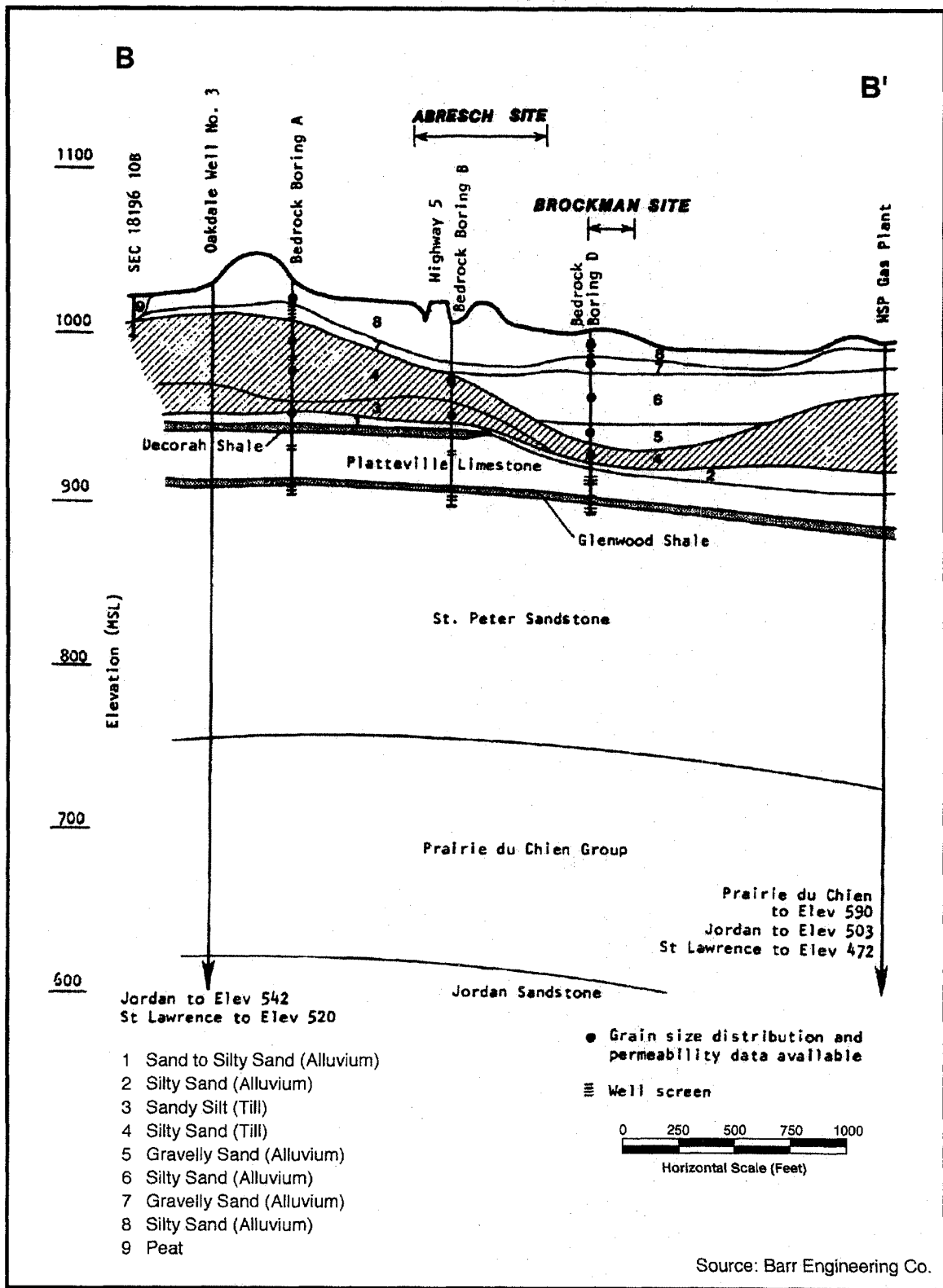


Figure 2

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05P-0185-2

Figure 3

GEOLOGIC CROSS SECTION B-B'
OAKDALE DISPOSAL SITE, MN

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Final Report

Analysis of PFOA, PFOS, PFHS, and PFBS
From Newly Constructed Wells at the Oakdale, Minnesota
Disposal Site

Laboratory Request Number: E06-0151

Method Requirement: 3M Method ETS-8-154.1 (modified)

Testing Laboratory

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Certificate #2052-01

The testing reported herein meet the requirements of ISO/IEC 17025-1999 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Certificate #2052-01. Testing that complies with this International Standard also operate in accordance with ISO 9001/ISO 9002 (1994).

3M Environmental Laboratory

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3M Technical Lead: Michelle Malinsky, Ph.D.

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Analytical Report E06-0151

Ground Water Samples from Newly Constructed Wells near the Oakdale, Minnesota Disposal Site
March 13, 2006

1 Introduction/Summary

The 3M Environmental Laboratory extracted and analyzed ground water samples collected by Weston Solutions, Inc. personnel on March 2-3, 2006 from newly constructed wells on and/or near the Oakdale, Minnesota 3M disposal site. Samples were returned to the 3M Environmental Laboratory for analysis of perfluorooctanoate (PFOA), perfluorooctane sulfonate (PFOS), perfluorohexane sulfonate (PFHS), and perfluorobutane sulfonate (PFBS) under laboratory project number E06-0151 using 3M Environmental Laboratory Method ETS 8-154.1 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extractions and High Performance Liquid Chromatography/Mass Spectrometry".

The 3M Environmental Laboratory prepared sets of sample containers for nine sampling locations. Each sample set consisted of a field sample, field sample duplicate, low field spike (1.0 ng/mL) and high field spike (10 ng/mL). Each empty container was marked with a "fill to here" line which corresponded to a final volume of 450 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the four target analytes prior to being sent to the field for sample collection.

All samples were extracted on March 3, 2006 and analyzed on March 3, 2006 and on March 6, 2006. Table 1 below summarizes the sample results. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



Certificate #2052-01

The testing reported herein meet the requirements of ISO/IEC 17025-1999 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Certificate #2052-01. Testing that complies with this International Standard also operate in accordance with ISO 9001/ISO 9002 (1994).

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Table 1. Sample Results Summary.

3M LIMS ID	Sample Description	¹⁹ PFOA Concentration (ng/mL)	²⁹ PFOS Concentration (ng/mL)	²⁹ PFHS Concentration (ng/mL)	²⁹ PFBS Concentration (ng/mL)
E06-0151-082	OKMN GWS RW40 0 060302	<0.0250	<0.0249	<0.0252	<0.0397
E06-0151-083	OKMN GWS RW40 DB 060302	<0.0250	<0.0249	<0.0252	<0.0397
Average		<0.0250	<0.0249	<0.0252	<0.0397
%RPD Sample/Sample Dup		NA	NA	NA	NA
E06-0151-086	OKMN GWS SP42 0 060302	<0.0250	<0.0249	<0.0252	<0.0397
E06-0151-087	OKMN GWS SP42 DB 060302	<0.0250	<0.0249	<0.0252	<0.0397
Average		<0.0250	<0.0249	<0.0252	<0.0397
%RPD Sample/Sample Dup		NA	NA	NA	NA
E06-0151-090	OKMN GWS PL43 0 060302	<0.0250	<0.0249	<0.0252	<0.0397
E06-0151-091	OKMN GWS PL43 DB 060302	<0.0250	<0.0249	<0.0252	<0.0397
Average		<0.0250	<0.0249	<0.0252	<0.0397
%RPD Sample/Sample Dup		NA	NA	NA	NA
E06-0151-094	OKMN GWS RW39 0 060302	<0.0250	<0.0249	<0.0252	<0.0397
E06-0151-095	OKMN GWS RW39 DB 060302	<0.0250	<0.0249	<0.0252	<0.0397
Average		<0.0250	<0.0249	<0.0252	<0.0397
%RPD Sample/Sample Dup		NA	NA	NA	NA
E06-0151-098	OKMN GWS PC45 0 060303	0.592	0.390	0.0649	<0.0397
E06-0151-098 ⁽¹⁾	OKMN GWS PC45 0 060303	0.522	0.464	0.0635	<0.0397
E06-0151-099	OKMN GWS PC45 DB 060303	0.514	0.449	0.0547	<0.0397
Average		0.543	0.434	0.0610	<0.0397
%RSD Sample/Sample Dup/Lab Dup		7.9	9.0	9.1	NA
E06-0151-102	OKMN GWS SP44 0 060303	<0.0250	<0.0249	<0.0252	<0.0397
E06-0151-103	OKMN GWS SP44 DB 060303	<0.0250	<0.0249	<0.0252	<0.0397
Average		<0.0250	<0.0249	<0.0252	<0.0397
%RPD Sample/Sample Dup		NA	NA	NA	NA
E06-0151-106	OKMN GWS RW38 0 060303	0.0525	0.187	<0.0252	<0.0397
E06-0151-107	OKMN GWS RW38 DB 060303	0.0556	0.195	<0.0252	<0.0397
Average		0.0540	0.191	<0.0252	<0.0397
%RPD Sample/Sample Dup		5.7	4.2	NA	NA
E06-0151-110	OKMN GWS RW37 0 060303	0.312	2.47	<0.0252	<0.0397
E06-0151-110 ⁽¹⁾	OKMN GWS RW37 0 060303	0.282	2.51	<0.0252	<0.0397
E06-0151-111	OKMN GWS RW37 DB 060303	0.298	2.56	<0.0252	<0.0397
Average		0.297	2.51	<0.0252	<0.0397
%RSD Sample/Sample Dup/Lab Dup		5.0	1.8	NA	NA
E06-0151-114	OKMN GWS PL41 0 060303	0.0766	0.0268	<0.0252	<0.0397
E06-0151-115	OKMN GWS PL41 DB 060303	0.0796	<0.0249	<0.0252	<0.0397
Average		0.0781	⁽¹⁾ 0.0268	<0.0252	<0.0397
%RPD Sample/Sample Dup		3.8	NA	NA	NA

(1) The analytical uncertainty for PFOA was 100±5.8% based on method accuracy and precision. See Section 3.7 for additional explanation. The limit of quantitation (LOQ) was 0.0250 ng/mL. Results and average values are rounded to three significant figures according to EPA rounding rules. %RPD values are rounded to two significant figures.

Because of rounding, values may vary slightly from those listed in the raw data. Samples with reported results less than the LOQ of 0.0250 ng/mL may or may not have PFOA present. Results less than the LOQ are not quantified.

- (2) The analytical uncertainty for PFOS was $100 \pm 3.7\%$ based on method accuracy and precision. See Section 3.7 for additional explanation. The limit of quantitation (LOQ) was 0.0249 ng/mL. Results and average values are rounded to three significant figures according to EPA rounding rules. %RPD values are rounded to two significant figures. Because of rounding, values may vary slightly from those listed in the raw data. Samples with reported results less than the LOQ of 0.0249 ng/mL may or may not have PFOS present. Results less than the LOQ are not quantified.
- (3) The analytical uncertainty for PFHS was $100 \pm 8.6\%$ based on method accuracy and precision. See Section 3.7 for additional explanation. The limit of quantitation (LOQ) was 0.0252 ng/mL. Results and average values are rounded to three significant figures according to EPA rounding rules. %RPD values are rounded to two significant figures. Because of rounding, values may vary slightly from those listed in the raw data. Samples with reported results less than the LOQ of 0.0252 ng/mL may or may not have PFHS present. Results less than the LOQ are not quantified.
- (4) The analytical uncertainty for PFBS was $100 \pm 6.6\%$ based on method accuracy and precision. See Section 3.7 for additional explanation. The limit of quantitation (LOQ) was 0.0397 ng/mL. Results and average values are rounded to three significant figures according to EPA rounding rules. %RPD values are rounded to two significant figures. Because of rounding, values may vary slightly from those listed in the raw data. Samples with reported results less than the LOQ of 0.0397 ng/mL may or may not have PFBS present. Results less than the LOQ are not quantified.
- (5) Sample results are for a laboratory extraction duplicate. The relative standard deviation (%RSD) is reported using all three results (sample, laboratory duplicate, and sample duplicate).
- (6) The average value listed is the concentration for the sample or sample duplicate that produced a value above the LOQ. A true average and %RPD between the sample and sample duplicate was not determined.

2 Methods - Analytical and Preparatory

2.1 Sample Collection

Samples were collected in Nalgene™ (low-density polyethylene) bottles prepared at the 3M Environmental Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest (PFOA, PFOS, PFHS, and PFBS). Collected sample bottles were returned to the laboratory at ambient conditions on March 3, 2006. Table 2 below details the bottles prepared and the corresponding spike levels for each sampling location. Samples were collected from nine individual sites. Table 3 provides the key code for the sample descriptions.

Table 2. Sample Collection and Spike Information.

Description	Nominal Final Volume (mL)	Final Concentration (ng/mL)			
		PFOA	PFOS	PFHS	PFBS
Sample	450	NA	NA	NA	NA
Sample Duplicate	450	NA	NA	NA	NA
Low Field Matrix Spike	450	0.999	0.997	1.01	0.992
High Field Matrix Spike	450	9.99	9.97	10.1	9.92
Trip Blank	450	NA	NA	NA	NA
Trip Blank Low Matrix Spike	450	0.999	0.997	1.01	0.992
Trip Blank High Matrix Spike	450	9.99	9.97	10.1	9.92

Table 3. Sample Description Key Code.

Sample ID Code	Definition
RW	Residuum well
PL	Platteville Limestone well
SP	St. Peter Sandstone well
PC	Prairie du Chein well
OKMN	Oakdale Minnesota site
GW	Groundwater Sample
0	Sample
DB	Duplicate
LS	Low Spike
HS	High Spike

The ground water samples, sample duplicates, and field matrix spikes returned for analysis all had visible sediment present which "colored" the water sample when shaken to disperse the solids. All collected samples were centrifuged to remove particulate before extraction. See Section 2.2 for more information. Additionally, the samples associated with RW37 generated a fairly strong petroleum odor. The table below describes additional observations noted for each of the sample locations.

Table 4. Sample Observations.

Well Identification	Observations		
	Sediment Color	Sediment Amount	Other
RW37	Grey-brown	⁽¹⁾ Moderate	Strong petroleum odor present
RW38	Rust-brown	⁽²⁾ Light	NA
RW39	Rust-brown	⁽²⁾ Light	NA
RW40	Rust-brown	⁽¹⁾ Moderate	NA
PL41	Rust-brown	⁽¹⁾ Moderate	NA
SP42	Rust-brown	⁽¹⁾ Moderate	NA
PL43	Rust-brown	⁽¹⁾ Moderate	NA
SP44	Grey-brown	⁽¹⁾ Moderate	NA
PC45	Rust-brown	⁽²⁾ Light	NA

(1) Moderate: after centrifugation of a 40 mL aliquot, solids roughly the size of a pea remained after removal of the supernatant.

(2) Light: after centrifugation of a 40 mL aliquot, solids present, but minimal.

2.2 Extraction

All samples, calibration standards, and associated quality control samples were extracted using the procedure outlined in ETS-8-154.1 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extractions and High Performance Liquid Chromatography/Mass Spectrometry". Briefly, 40 mL of sample were loaded onto a pre-conditioned Waters C18 solid phase extraction (SPE) cartridge (Sep-Pak, 1.0 g, 6 cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol. This extraction procedure concentrates the samples by a factor of eight. (Initial volume = 40 mL, final volume = 5 mL). The calibration standards, lab control spikes, method blanks, and trip blank sample and spikes were extracted as described above. Because the collected ground water samples contained visibly significant amounts of sediment/particulate, the 40 mL aliquot was centrifuged at 3500 rpm for ten minutes for all samples, sample duplicates, low field matrix spikes and high field matrix spikes prior to extraction. After centrifugation, the supernatant was loaded onto the SPE cartridge for extraction similar to the calibration standards and laboratory quality control samples. The remaining solids were retained if further extraction was deemed necessary. ETS-8-154.1 does not describe sample centrifugation prior to extraction. This method modification was

necessary as the sediment completely clogged the SPE cartridges almost immediately preventing complete loading of the water sample. The final methanol sample extracts had a slight yellowish color. Recoveries of laboratory matrix spikes and field sample matrix spikes demonstrated that the centrifugation step did not significantly impact the method performance.

Samples were extracted and initially analyzed on March 3, 2006. The trip blank spikes for all four analytes produced recoveries of approximately 60%. Additionally, most field sample matrix spike recoveries were between 60-80%. Upon further review of the data, it was determined that an older standard was used to prepare the upper range of the calibration curve on March 3, 2006 and that this standard was not the same one used to prepare the collection bottle spikes. On March 6, 2006, a new extracted calibration curve was prepared using the same (newer) standard used to prepare the collection bottle spikes. Method blanks and lab control spikes at 1 ng/mL and 10 ng/mL were also prepared with the new curve. Additionally, the trip blank and trip blank field matrix spikes along with two samples (E06-0151-098 and E06-0151-0110) were also re-extracted. Furthermore, low and high lab matrix spikes at 1 and 10 ng/mL were prepared for the two samples selected for re-extraction. For the lab matrix spiked samples, an appropriate amount of spiking standard was added to the 40 mL sample aliquot before it was centrifuged to remove the solids. Analysis of selected calibration points from the first curve (March 3, 2006) against the new curve (March 6, 2006) demonstrated that a systematic bias of 20-30% was present in the first analysis. All sample extracts from March 3, 2006 were then reanalyzed against the new curve. All results presented here are from the March 6, 2006 analysis against the new calibration curve.

2.3 Analysis

All sample and quality control extracts were analyzed for the target analytes using high performance liquid chromatography/ tandem mass spectrometry (HPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Table 5. Instrument Parameters

Instrument Name	ETSStan
Liquid Chromatograph	Agilent 1100
Guard column	Betasil C18 (2.1 mm.X100 mm), 5 µm
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 µm
Injection Volume	5 µL
Mass Spectrometer	Applied Biosystems API 4000
Ion Source	Turbo Spray
Electrode	Z-spray
Polarity	Negative
Software	Analyst 1.4.1

Table 6. Liquid Chromatography Gradient Program

Step Number	Total Time (min)	Flow Rate (µL/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0	300	80.0	20.0
1	1.0	300	80.0	20.0
2	14.5	300	10.0	90.0
3	15.5	300	10.0	90.0
4	16.5	300	80.0	20.0
5	20.0	300	80.0	20.0

Table 7. Mass Transitions

Analyte	Mass Transition Q1/Q3	Dwell Time (msec)
⁽¹⁾ PFBS	299/80	125
	299/99	125
⁽¹⁾ PFHS	399/80	125
	399/99	125
⁽¹⁾ PFOS	499/130	125
	499/99	125
	499/80	125
⁽¹⁾ PFOA	413/369	125
	413/219	125
	413/169	125

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

3 Data Analysis

3.1 Calibration

Calibration standards were prepared by spiking known amounts of methanolic stock solutions containing PFOA, PFOS, PFHS, and PFBS into 40 mL of ASTM type I water. Each spiked water standard was then extracted in the same manner as the collected samples. A total of twelve spiked standards ranging from 0.025 ng/mL to 25 ng/mL (nominal) were prepared. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data was not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each extracted calibration standard used to generate the final calibration curve met the method calibration accuracy requirement of $\pm 25\%$. The correlation coefficients (*r*) were greater than 0.999 for all analytes.

3.2 Limit of Quantitation (LOQ)

The LOQ for this analysis, as defined in ETS-8-154.1, is the lowest non-zero calibration standard in the curve in which the area counts are twice those of the method blank(s). The limit of quantitation for PFOA, PFOS, PFHS, and PFBS is 0.0250 ng/mL, 0.0249 ng/mL, 0.0252 ng/mL, and 0.0397 ng/mL respectively. Area count comparison for the method blanks and the lowest calibration standard will be provided in Section 3.5.1.

3.3 System Suitability

Five replicate injections of the 5 ng/mL extracted-calibration standard was analyzed at the beginning of the analytical sequence to demonstrate overall system suitability. The same standard was analyzed in triplicate at the end of the analytical sequence. All analytes met method acceptance criteria of less than 5% relative standard deviation (RSD) for peak area and less than 2% RSD for retention time for both opening and closing system suitability injections.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs produced recoveries within $100\% \pm 25\%$, which met method criteria.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below.

3.5.1 Method Blanks

Several method blanks were prepared by loading 40 mL of ASTM Type I water onto a C18 SPE cartridge and eluting with 5 mL of methanol using the same extraction procedure as the samples. Method blanks were prepared to evaluate the levels of background contamination in the overall extraction process (reagent water, glassware, SPE cartridges, etc.) Table 8 lists the area counts for the method blanks and the LOQ standard associated with the given data set.

Table 8. Method Blank Area Counts for Extractions performed on 3/6/2006.

Sample Name	Sample Comment	Sample ID	Area Counts			
			PFOA	PFOS	PFHS	PFBS
s060306a025	Method Blank-1	MB-060306-1	11741	⁽¹⁾ 2880	5939	13417
s060306a026	Method Blank-2	MB-060306-2	11689	1412	2265	4009
s060306a027	Method Blank-3	MB-060306-3	11131	1814	5101	18787
s060306a028	Method Blank-4	MB-060306-4	6189	993	1824	4178
s060306a029	Method Blank-5	MB-060306-5	8872	1510	1613	4717
s060306a030	Method Blank-6	MB-060306-6	5558	655	1218	1874
s060306a031	Method Blank-7	MB-060306-7	20205	1982	5292	11821
s060306a032	Method Blank-8	MB-060306-8	15076	1802	4387	14398
Area Counts of highest method blank multiplied by two			40410	3964	11878	37574
Area Counts LOQ standard			50284	5254	21147	54306
LOQ concentration, ng/mL			0.0250	0.0249	0.0252	0.0397

(1) Determined to be a statistical outlier at the 80% confidence level using Dixon's Q-test. Area value not used to establish the LOQ for PFOS.

3.5.2 Solvent Blanks

Several methanol solvent blanks were analyzed to assess system contamination and/or instrument carryover. Analyte peak area counts in all blank samples were less than half the area counts of the calibration standard used to establish the LOQ.

3.5.3 Field/Trip Blanks

Prior to sample collection, one sample container was filled with 450 mL of ASTM Type I water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank. The trip blank serves as an additional method blank that accounts for any storage conditions and/or holding time issues that the samples may experience. The target analytes were not detected above the stated LOQ in the trip blank.

3.6 Lab Control Spikes (LCSs)

For the extractions performed on March 3, 2006, low (0.25 ng/mL nominal concentration) and high (7.5 ng/mL nominal concentration) lab control spikes were prepared and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 40 mL of ASTM Type I water to produce the desired concentration. The spiked water samples were then extracted and analyzed in the same manner as the samples. Analysis of triplicate LCSs at the two specified levels cross-validates the

analytical method as used here for any modifications/deviations from ETS 8-154.1. Additionally, LCS results will be used to determine overall method uncertainty in Section 3.7. Table 9 summarizes the LCS recovery results from the March 6, 2006 extraction set. LCS recoveries from the March 3, 2006 extraction are not given here as no sample results were reported from this run.

Table 9. ⁽¹⁾Lab Control Spike Results.

Sample Description	Lab ID	PFOA			PFOS		
		Spiked Conc. (ng/mL)	Calculated Conc. (ng/mL)	% Recovery	Spiked Conc. (ng/mL)	Calculated Conc. (ng/mL)	% Recovery
1 ppb Lab Control Spike	LCS-060306-1	0.999	1.00	100	0.997	1.04	104
1 ppb Lab Control Spike	LCS-060306-2	0.999	1.01	101	0.997	1.03	103
1 ppb Lab Control Spike	LCS-060306-3	0.999	0.961	96.2	0.997	1.00	100
10 ppb Lab Control Spike	LCS-060306-4	9.99	9.19	92.1	9.97	9.91	99.4
10 ppb Lab Control Spike	LCS-060306-5	9.99	9.65	96.6	9.97	10.2	102
10 ppb Lab Control Spike	LCS-060306-6	9.99	9.85	98.6	9.97	10.1	101
Average %Recovery ± %RSD		97.4% ± 3.3%			102% ± 1.8%		

Sample Description	Lab ID	PFHS			PFBS		
		Spiked Conc. (ng/mL)	Calculated Conc. (ng/mL)	% Recovery	Spiked Conc. (ng/mL)	Calculated Conc. (ng/mL)	% Recovery
1 ppb Lab Control Spike	LCS-060306-1	1.01	1.08	107	0.992	1.04	105
1 ppb Lab Control Spike	LCS-060306-2	1.01	1.11	110	0.992	1.05	106
1 ppb Lab Control Spike	LCS-060306-3	1.01	1.08	107	0.992	1.03	104
10 ppb Lab Control Spike	LCS-060306-4	10.1	9.85	97.7	9.92	9.17	92.4
10 ppb Lab Control Spike	LCS-060306-5	10.1	9.93	98.5	9.92	9.32	94.0
10 ppb Lab Control Spike	LCS-060306-6	10.1	9.84	97.6	9.92	9.49	95.7
Average %Recovery ± %RSD		103% ± 5.5%			99.4% ± 6.1%		

- (1) All results and average values listed to three significant figures according to EPA rounding rules. %RSD values given to two significant figures. Due to rounding, values may vary slightly from those in the raw data.

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

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

3.7 Analytical Uncertainty

Both the accuracy (percent recovery) and precision (%RSD) of the lab control spikes were used to estimate the overall method's analytical uncertainty for a given analyte. For example, the overall accuracy and precision for PFOA based on LCS results was 97.4% ± 3.3%. The measured precision (%RSD) is then used to determine the range of the accuracy.

Example:

$$97.4 * (0.033) = 3.25$$

$$97.4 + 3.25 = 100.7; 97.4 - 3.25 = 94.20$$

Thus, LCS accuracy results range from 94.2% to 100.7%. The absolute difference of the low and high ends of this range when compared 100%, are then calculated.

$$100.7\% - 100\% = 0.70\%$$
$$100\% - 94.2\% = 5.8\%$$

The most conservative (largest) absolute difference is then used as the analytical uncertainty for the given analyte. Therefore, the analytical uncertainty for PFOA is given as $100 \pm 5.8\%$ for these results. For PFOS, PFHS, and PFBS, the analytical uncertainty, as defined here, is $100 \pm 3.7\%$, $100 \pm 8.6\%$, and $100 \pm 6.6\%$, respectively.

3.8 Field Matrix Spikes (FMS)

Low (nominal concentration of 1.0 ng/mL) and high (nominal concentration 10 ng/mL) field matrix spikes were collected at each sampling point to verify that the analytical method is applicable to the collected matrix. Field matrix spike recoveries within method acceptance criteria of $100 \pm 30\%$ confirm that "unknown" components in the sample matrix do not interfere with the extraction and analysis of the analytes of interest. Field matrix spike results will be presented in the next section with the sample data.

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

4 Data Summary

The tables (Tables 10-19) below summarize the sample results, field matrix spike recoveries, and lab matrix spike recoveries (if applicable) for the nine locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (RPD) or relative percent standard deviation (RSD) of the sample and sample duplicate (and laboratory duplicate – if applicable). All field matrix spike recoveries were within $100 \pm 30\%$ for all analytes except for the high level PFOS field matrix spike associated with PC45 (LIMS ID# E06-0151-101) which produced a recovery of 148%. The analytical accuracy for the PFOS results associated with this well have not been adjusted as the low field matrix spike, as well as the low and high level lab matrix spikes, produced recoveries within $100 \pm 30\%$.

Table 10. ⁽¹⁾Sample Results for OKMN GWS RW40.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-082	OKMN GWS RW40 0 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-083	OKMN GWS RW40 DB 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-084	OKMN GWS RW40 LS 060302	1.15	115	1.22	122	1.15	114	0.976	98.4
E06-0151-085	OKMN GWS RW40 HS 060302	7.26	72.7	9.93	99.6	9.05	90.0	8.4	84.6
Average Concentration (ng/mL) ± %RPD		<0.0250		<0.0249		<0.0252		<0.0397	

NA= Not Applicable.

(1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data. %RPD values not given with both the sample and sample duplicate concentrations are less than the reported LOQ.

Table 11. ⁽¹⁾Sample Results for OKMN GWS SP42.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-086	OKMN GWS SP42 0 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-087	OKMN GWS SP42 DB 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-088	OKMN GWS SP42 LS 060302	0.93	93.1	1.18	118	1.03	102	0.963	97.0
E06-0151-089	OKMN GWS SP42 HS 060302	7.25	72.6	10.1	101	8.54	84.9	8.21	82.7
Average Concentration (ng/mL) ± %RPD		<0.0250		<0.0249		<0.0252		<0.0397	

NA= Not Applicable.

(1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data. %RPD values not given with both the sample and sample duplicate concentrations are less than the reported LOQ.

Table 12. ⁽¹⁾Sample Results for OKMN GWS PL43.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-090	OKMN GWS PL43 O 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-091	OKMN GWS PL43 DB 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-092	OKMN GWS PL43 LS 060302	1.02	102	1.2	120	1.09	108	0.982	99.0
E06-0151-093	OKMN GWS PL43 HS 060302	8.29	83.0	11.7	117	10.1	100	9.31	93.8
Average Concentration (ng/mL) ±%RPD		<0.0250		<0.0249		<0.0252		<0.0397	

NA= Not Applicable.

(1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data. %RPD values not given with both the sample and sample duplicate concentrations are less than the reported LOQ.

Table 13. ⁽¹⁾Sample Results for OKMN GWS RW39.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-094	OKMN GWS RW39 O 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-095	OKMN GWS RW39 DB 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-096	OKMN GWS RW39 LS 060302	0.954	95.5	1.07	107	0.968	96.2	0.951	95.8
E06-0151-097	OKMN GWS RW39 HS 060302	7.43	74.4	10.5	105	9.52	94.6	9.25	93.2
Average Concentration (ng/mL) ±%RPD		<0.0250		<0.0249		<0.0252		<0.0397	

NA= Not Applicable.

(1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data. %RPD values not given with both the sample and sample duplicate concentrations are less than the reported LOQ.

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Table 14. ⁽¹⁾Sample Results for OKMN GWS PC45.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-098	OKMN GWS PC45 0 060303	0.522	NA	0.464	NA	0.0649	NA	<0.0397	NA
E06-0151-099	OKMN GWS PC45 DB 060303	0.514	NA	0.449	NA	0.0547	NA	<0.0397	NA
E06-0151-098 ⁽²⁾	OKMN GWS PC45 0 060303	0.592	NA	0.390	NA	0.0635	NA	<0.0397	NA
E06-0151-100	OKMN GWS PC45 LS 060303	1.45	90.6	1.64	121	1.11	105	1.05	106
E06-0151-101	OKMN GWS PC45 HS 060303	9.93	94.0	15.2	⁽³⁾ 148	10.3	102	9.74	98.2
⁽⁴⁾ E06-0151-098-LMS Low	Laboratory Matrix Spike at 1 ng/mL	1.59	105	1.41	97.5	1.07	100	0.944	95.1
⁽⁴⁾ E06-0151-098-LMS High	Laboratory Matrix Spike at 10 ng/mL	11.3	108	11.0	106	10.5	104	9.05	91.2
Average Concentration (ng/mL) ± %RSD		0.543 ± 7.9		0.434 ± 9.0		0.0610 ± 9.1		<0.0397	

NA= Not Applicable.

- (1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data.
- (2) Sample results are from a duplicate laboratory extraction of the sample. All three replicate values used to determine average and %RSD.
- (3) FMS recovery exceeded method acceptance criteria of 100±30%.
- (4) Laboratory matrix spikes were prepared by spiking the 40 mL sample aliquot prior to centrifugation.

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Table 15. ⁽¹⁾Sample Results for OKMN GWS SP44.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-102	OKMN GWS SP44 O 060303	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-103	OKMN GWS SP44 DB 060303	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-104	OKMN GWS SP44 LS 060303	0.943	94.4	1.08	108	0.991	98.5	0.942	94.9
E06-0151-105	OKMN GWS SP44 HS 060303	8.82	88.3	11.9	119	9.60	95.4	9.16	92.3
Average Concentration (ng/mL) $\pm$ %RPD		<0.0250		<0.0249		<0.0252		<0.0397	

NA= Not Applicable.

(1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data. %RPD values not given with both the sample and sample duplicate concentrations are less than the reported LOQ.

Table 16. ⁽¹⁾Sample Results for OKMN GWS RW38.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-106	OKMN GWS RW38 O 060303	0.0525	NA	0.187	NA	<0.0252	NA	<0.0397	NA
E06-0151-107	OKMN GWS RW38 DB 060303	0.0556	NA	0.195	NA	<0.0252	NA	<0.0397	NA
E06-0151-108	OKMN GWS RW38 LS 060303	1.02	96.7	1.42	123	1.06	105	0.969	97.6
E06-0151-109	OKMN GWS RW38 HS 060303	7.94	78.9	10.2	100	9.27	92.1	8.86	89.3
Average Concentration (ng/mL) $\pm$ %RPD		0.0540 $\pm$ 5.7		0.191 $\pm$ 4.2		<0.0252		<0.0397	

NA= Not Applicable.

(1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data. %RPD values not given with both the sample and sample duplicate concentrations are less than the reported LOQ.

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Table 17. ⁽¹⁾Sample Results for OKMN GWS RW37.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-110	OKMN GWS RW37 0 060303	0.282	NA	2.51	NA	<0.0252	NA	<0.0397	NA
E06-0151-111	OKMN GWS RW37 DB 060303	0.298	NA	2.56	NA	<0.0252	NA	<0.0397	NA
E06-0151-110 ⁽²⁾	OKMN GWS RW37 0 060303	0.312	NA	2.47	NA	<0.0252	NA	<0.0397	NA
E06-0151-112	OKMN GWS RW37 LS 060303	1.22	92.3	3.43	92.2	1.19	118	1.16	117
E06-0151-113	OKMN GWS RW37 HS 060303	7.73	74.4	11.7	92.1	10.2	101	9.83	99.1
⁽³⁾ E06-0151-110-LMS Low	Laboratory Matrix Spike at 1 ng/mL	1.47	117	3.71	120	0.983	97.7	0.941	94.8
⁽³⁾ E06-0151-110-LMS High	Laboratory Matrix Spike at 10 ng/mL	9.62	93.3	12.3	98.2	8.91	88.6	8.82	88.9
Average Concentration (ng/mL) ±%RSD		0.297 ± 5.0		2.51 ± 1.8		<0.0252		<0.0397	

NA= Not Applicable.

- (1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data.
- (2) Sample results are from a duplicate laboratory extraction of the sample. All three replicate values used to determine average and %RSD.
- (3) Laboratory matrix spikes were prepared by spiking a 40 mL sample aliquot prior to centrifugation.

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Table 18. ⁽¹⁾Sample Results for OKMN GWS RW39.

3M LIMS ID	Description	PFOA		PFOS		PFHS		PFBS	
		Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
E06-0151-114	OKMN GWS PL41 O 060303	0.0766	NA	0.0268	NA	<0.0252	NA	<0.0397	NA
E06-0151-115	OKMN GWS PL41 DB 060303	0.0796	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-116	OKMN GWS PL41 LS 060303	1.11	103	1.13	111	1.10	109	0.995	100
E06-0151-117	OKMN GWS PL41 HS 060303	9.93	98.6	12.1	121	11.7	116	10.3	104
Average Concentration (ng/mL) ± %RPD		0.0781 ± 3.8		⁽²⁾ 0.0268		<0.0252		<0.0397	

NA= Not Applicable.

- (1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data. %RPD values not given with both the sample and sample duplicate concentrations are less than the reported LOQ.
- (2) The average value reported reflects the single result that was above the LOQ. A true sample average and %RSD were not determined.

Table 19. ⁽¹⁾Sample Results for the Trip Blank.

		PFOA		PFOS		PFHS		PFBS	
3M LIMS ID	Description	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery	Conc. (ng/mL)	%Recovery
⁽²⁾ Extracted 3-3-06									
E06-0151-079	OKMN PW TRIP2 O 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-080	OKMN PW TRIP2 LS 060302	0.808	80.9	0.788	79.0	0.808	80.3	0.783	78.9
E06-0151-081	OKMN PW TRIP2 HS 060302	7.47	74.8	8.25	82.7	8.04	79.9	7.67	77.3
Extracted 3-6-06									
E06-0151-079	OKMN PW TRIP2 O 060302	<0.0250	NA	<0.0249	NA	<0.0252	NA	<0.0397	NA
E06-0151-080	OKMN PW TRIP2 LS 060302	1.04	104	0.928	93.0	1.04	103	0.982	99.0
E06-0151-081	OKMN PW TRIP2 HS 060302	9.04	90.5	10.2	102	10.0	99.4	9.58	96.5

NA= Not Applicable.

- (1) Table displays rounded values for all concentrations and percent recovery values (3 significant figures), %RPD values (2 significant figures). Recovery and RPD values may vary slightly from the values in the raw data. %RPD values not given with both the sample and sample duplicate concentrations are less than the reported LOQ.
- (2) Samples extracted on 3-3-06 but analyzed against the calibration curve prepared on 3-6-06.

5 Conclusion

PFOA, PFOS, PFHS, and PFBS results for the collected samples were presented in Table 1. Laboratory control spikes were used to determine the method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the analytical uncertainty for the results ($100 \pm 5.8\%$ for PFOA, $100 \pm 3.7\%$ for PFOS, $100 \pm 8.6\%$ for PFHS, and $100 \pm 6.6\%$ for PFBS). Field matrix spike recoveries within $100 \pm 30\%$ demonstrated that the analytical method was appropriate for the given sample matrix.

6 Data / Sample Retention

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

7 Signatures

Susan T. Wolf 3/10/06
Susan T. Wolf, 3M Senior Chemist, Date

Michelle D. Malinsky 3/13/06
Michelle D. Malinsky, Ph.D., 3M Technical Lead, Date

William K. Reagen 3/13/06
William K. Reagen, Ph.D., Environmental Laboratory Management, Date

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

Debra K. Reed 3/10/06
Quality Assurance Representative, Date

INTERIM REPORT #1 Analysis of Oakdale Water Samples

STUDY TITLE

Analysis of Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Cottage Grove 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

May 19, 2005

PERFORMING LABORATORY

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STUDY SPONSOR

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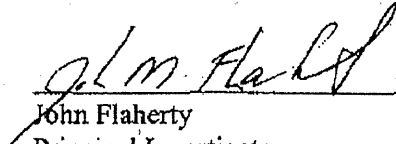
PROJECT

Protocol Number: P0001400
Exygen Study Number: P0001400

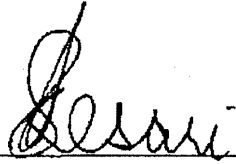
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GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

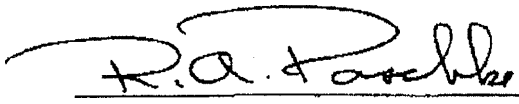
Exygen Study Number P0001400, entitled "Analysis of Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Cottage Grove Monitoring Program," conducted for 3M Company, is being performed in compliance with EPA TSCA Good Laboratory Practice Standards 40 CFR 792 by Exygen Research.


John Flaherty
Principal Investigator
Exygen Research

5/19/05
Date


Jaisimha Kesari P.E., DEE
Study Director
Weston Solutions, Inc.

5/27/05
Date

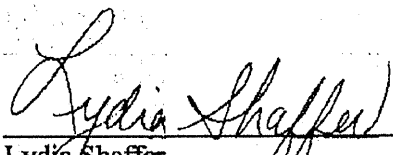

Robert A. Paschke
Sponsor Representative
3M Company

6/1/05
Date

QUALITY ASSURANCE STATEMENT

Exygen Research's Quality Assurance Unit reviewed Exygen Study Number P0001400, entitled, "Analysis of Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Cottage Grove Monitoring Program." All reviewed phases¹ were inspected for conduct according to Exygen Research's Standard Operating Procedures, the Study Protocol, and all applicable Good Laboratory Practice Standards. All findings were reported to the Exygen Principal Investigator and Management and to the Study Director.

<u>Phase</u>	<u>Date Inspected</u>	<u>Date Reported to Principal Investigator</u>	<u>Date Reported to Exygen Management</u>	<u>Date Reported to Study Director</u>
1. Protocol Review	03/24/05	03/25/05	03/29/05	05/19/05
2. Extraction Review	03/24/05	03/25/05	03/29/05	05/19/05
3. Raw Data Review and Interim Analytical Report Review	05/16-17/05	05/18/05	05/19/05	05/19/05


Lydia Shaffer
Technical Lead, Quality Assurance Unit

05/19/05
Date

¹Note: All inspections will be documented in the QA statement for the final analytical report at the conclusion of the study.

CERTIFICATION OF AUTHENTICITY

This interim report, for Exygen Study Number P0001400, is a true and complete representation of the raw data.

Submitted by: Exygen Research
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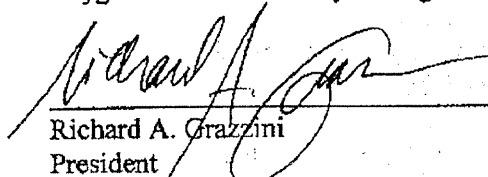
Principal Investigator, Exygen:



John Flaherty
Vice President
Exygen Research

5/19/05
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Exygen Research Facility Management:



Richard A. Grazzini
President
Exygen Research

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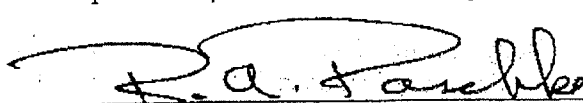
Study Director, Weston Solutions, Inc.



Jaisimha Resari P.E., DEE
Weston Solutions, Inc.

5/27/05
Date

Sponsor Representative, 3M Company:



Robert A. Paschke
Manager, 3M Corporate Environmental Programs

6/01/05
Date

STUDY IDENTIFICATION

Analysis of Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Cottage Grove Monitoring Program

PROTOCOL NUMBER: P0001400

EXYGEN STUDY NUMBER: P0001400

TYPE OF STUDY: Residue

SAMPLE MATRIX: Water

TEST SUBSTANCE: Perfluorooctanoic acid (PFOA),
Perfluorobutanesulfonate (PFBS),
Perfluorohexanesulfonate (PFHS), and
Perfluorooctanesulfonate (PFOS)

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TIMETABLE: Interim Analytical Start Date: 03/21/05
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PROJECT PERSONNEL

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

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1.0 SUMMARY

Exygen Research extracted and analyzed water samples for the determination of perfluorooctanoic acid (PFOA), perfluorobutanesulfonate (PFBS), perfluorohexanesulfonate (PFHS), and perfluorooctanesulfonate (PFOS) according to Exygen Method V0001780 (Appendix A).

The limit of quantitation for PFOA, PFBS, PFHS and PFOS in water was 50 ng/L and the limit of detection for PFOA, PFBS, PFHS and PFOS in water was 25 ng/L.

Analytical results for the analysis of PFOA, PFBS, PFHS and PFOS in water samples are summarized in Table 1. The overall average percent recoveries $\pm$ standard deviations for all fortifications of PFOA, PFBS, PFHS and PFOS in water samples were $93\% \pm 19$, $141\% \pm 21$, $96\% \pm 15$ and $109\% \pm 32$, respectively. The overall average percent recovery $\pm$ standard deviation for all fortifications of the surrogate field spike compound, ^{13}C -PFOA, in water samples was $83\% \pm 21$.

2.0 OBJECTIVE

The objective of the analytical part of this study was to determine levels of perfluorooctanoic acid (PFOA), perfluorobutanesulfonate (PFBS), perfluorohexanesulfonate (PFHS), and perfluorooctanesulfonate (PFOS) in water according to Protocol P0001400 (Appendix A).

3.0 INTRODUCTION

This report details the results of the analysis for the determination of PFOA, PFBS, PFHS and PFOS in water using the analytical method entitled, "V0001780: Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS."

The study was initiated on March 03, 2005, when the study director signed protocol number P0001400. The analytical start date for this interim report was March 21, 2005, and the analytical termination date for this interim report was April 21, 2005.

4.0 ANALYTICAL TEST SAMPLES

One hundred and twenty-one water samples, which correspond to twenty-nine sample sites, (Exygen ID C0064404-C0064438, C0064442-C0064461, C0064467-C0064532) were received at ambient temperature on March 15, 2005 from Tim Frinak at Weston Solutions, Inc. The samples were logged in by Exygen personnel and placed in refrigerated storage.

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 Exygen Research.

5.0 REFERENCE MATERIAL

The analytical standard, PFOA, was purchased from Sigma Aldrich and was received at Exygen on December 08, 2003. The surrogate spiking standard, ^{13}C labeled perfluorooctanoic acid (^{13}C PFOA), was received at Exygen on April 15, 2004 from the 3M Company. The analytical standards, PFBS and PFHS, were supplied by 3M. PFBS was received from 3M at Exygen on July, 06, 2000. PFHS was received from 3M at Exygen on January 20, 2003. PFOS was purchased from Fluka Corporation and was received at Exygen on April 23, 2003.

The available information for the reference materials is listed below. PFOA was stored ambient. PFBS, PFHS and ^{13}C PFOA were stored frozen and PFOS was stored refrigerated.

<u>Compound</u>	<u>Exygen Inventory No.</u>	<u>Lot #</u>	<u>Purity (%)</u>	<u>Expiration Date</u>
PFOA	SP0003800	23116HB	97.64	12/08/05
^{13}C PFOA	SP0004184	3507-195	97	03/29/09
PFBS	SP0000252	101	96.7	12/04/06
PFHS	SP0002401	SE036	98.6	10/18/06
PFOS	SP0002694	430180-1	101.2	04/23/06

The molecular structures of PFOA, ^{13}C PFOA, PFBS, PFHS and PFOS are given on the following pages:

PFOA

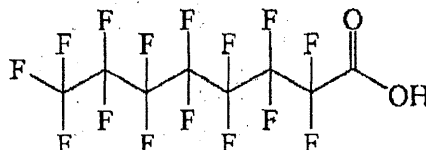
Chemical Name: Perfluorooctanoic acid

Molecular Weight: 414

Transitions Monitored: 413 → 369 (for quantification) and

413 → 219 (for confirmation)

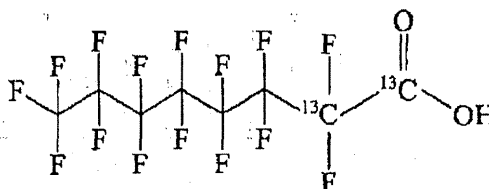
Structure:

**¹³C PFOA**Chemical Name: 1,2-¹³C perfluorooctanoic acid

Molecular Weight: 416

Transition Monitored: 415 → 370

Structure:

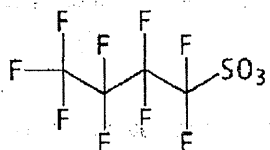
**PFBS**

Chemical Name: Perfluorobutanesulfonate

Molecular Weight: 338 supplied as the potassium salt ($C_4F_9SO_3K^+$)

Transitions Monitored: 299 → 99

Structure:



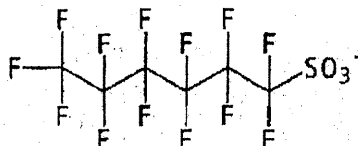
PFHS

Chemical Name: Perfluorohexanesulfonate

Molecular Weight: 438 supplied as the potassium salt ($C_6F_{13}SO_3K^+$)

Transitions Monitored: 399 → 80

Structure:

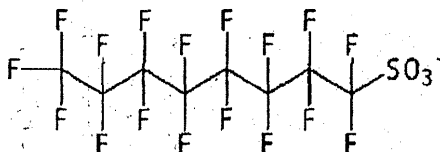
**PFOS**

Chemical Name: Perfluorooctanesulfonate

Molecular Weight: 538 supplied as the potassium salt ($C_8F_{17}SO_3K^+$)

Transitions Monitored: 499 → 80

Structure:

**6.0 DESCRIPTION OF ANALYTICAL METHOD**

The analytical method "V0001780: Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS" was used for this study.

6.1. Extraction Procedure

A 40 mL aliquot of the water sample was used for the extraction procedure. After fortification of appropriate samples, the samples were loaded onto a C_{18} SPE cartridge conditioned with 10 mL of methanol and 5 mL of water. The eluate was discarded. Approximately five milliliters of methanol was added to the cartridge. Five milliliters of eluate was collected into a graduated 15 mL polypropylene centrifuge tube. Each sample was analyzed by LC/MS/MS electrospray.

6.2 Preparation of Standards and Fortification Solutions

Individual stock standard solutions of PFOA, ^{13}C PFOA, PFBS, PFHS and PFOS were prepared as specified in Exygen method V0001780. The stock standard solutions were prepared at a concentration of 100 $\mu\text{g/mL}$ by dissolving 10 mg of each of the standard (corrected for purity and salt content, if necessary) in methanol. From these solutions, 1.0 $\mu\text{g/mL}$ fortification standard solutions were prepared by taking 1 mL of the appropriate stock and bringing the volume up to 100 mL with methanol. By taking 10 mL of the appropriate 1.0 $\mu\text{g/mL}$ fortification standard and bringing the volume up to 100 mL with methanol, 0.1 $\mu\text{g/mL}$ fortification standards were prepared. By taking 10 mL of the appropriate 0.1 $\mu\text{g/mL}$ fortification standard and bringing the volume up to 100 mL with methanol, 0.01 $\mu\text{g/mL}$ fortification standards were prepared.

Several sets of standards containing PFOA, ^{13}C PFOA, PFBS, PFHS and PFOS were prepared in water and processed through the extraction procedure, identical to samples. The following concentrations were prepared:

Conc. of Fort. Solution (ng/mL) ¹	Fort Volume (μL)	Volume of Fortified Sample (mL)	Final Conc. of Calibration Std. (ng/L)
0	0	40	0
10	100	40	25
10	200	40	50
10	400	40	100
100	100	40	250
100	200	40	500
100	400	40	1000

¹ of PFOA, ^{13}C PFOA, PFBS, PFHS and PFOS

An additional mixed stock solution of PFOA, ^{13}C PFOA, PFBS, PFHS, and PFOS was prepared at 1000 $\mu\text{g/mL}$ and diluted to 100 and 10 $\mu\text{g/mL}$ for bottle spiking purposes. Complete details can be found in the raw data package associated with this study.

The stock standard solution and all fortification and calibration standard solutions were stored in a refrigerator ($4^\circ \pm 2^\circ\text{C}$) when not in use. Documentation of standard preparation is located in the raw data package associated with this interim report.

6.3 Chromatography

Quantification of PFOA, PFBS, PFHS and PFOS was accomplished by LC/MS/MS electrospray. The retention time of PFOA, PFBS, PFHS and PFOS was ~12 min, ~5 mins, ~11 mins, and ~13 mins, respectively. Peaks above the LOD were not detected in any of the reagent blank samples corresponding to the analyte retention time.

6.4 Instrument Sensitivity

The smallest standard amount injected during the chromatographic run had a concentration of 25 ng/L of PFOA, PFBS, PFHS and PFOS.

6.5 Description of LC/MS/MS Instrument and Operating Conditions

Instrument: API 4000 Biomolecular Mass Analyzer
Interface: Turbo Ion Spray Liquid Introduction Interface
Computer: DELL OptiPlex GX400
Software: Windows NT, Analyst 1.4.1
HPLC: Hewlett Packard (HP) Series 1100
HP Quat Pump
HP Vacuum Degasser
HP Autosampler
HP Column Oven

HPLC Column: Thermo Fluophase RP, 50 mm x 2.1 mm
Column Temp.: 30° C
Injection Vol.: 15 µL
Mobile Phase (A): 2 mM Ammonium Acetate in water
Mobile Phase (B): Methanol

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>
0.0	65	35
1.0	65	35
8.0	25	75
10.0	25	75
11.0	65	35
18.0	65	35

Total run time: ~18 min
Flow Rate: 0.3 mL/min

Ions monitored:

Analyte	Mode	Transition Monitored	Approximate Retention Time (min)
PFOA	negative	413 → 369	~12 min.
PFOA Confirm Ion	negative	413 → 219	~12 min.
¹³ C PFOA	negative	415 → 370	~12 min.
PFBS	negative	299 → 99	~5 min.
PFHS	negative	399 → 80	~11 min.
PFOS	negative	499 → 80	~13 min.

6.6 Quantitation and Example Calculation

Fifteen microliters of sample or calibration standard were 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 six concentrations of standards. The concentration was determined from the equations below.

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

Equation 1:

$$\text{Analyte found (ng/L)} = \frac{(\text{Peak area} - \text{intercept}) \times \text{DF}}{\text{slope}}$$

Where: DF = Dilution Factor, factor by which the final volume was diluted, if necessary.

For samples fortified with known amounts of PFOA, PFBS, PFHS, PFOS and ¹³C PFOA prior to extraction, Equation 2 was used to calculate the percent recovery.

Equation 2:

Recovery (%) =

$$\frac{(\text{analyte found (ng/L)} - \text{analyte in control (ng/L)})}{\text{amount added (ng/L)}} \times 100\%$$

An example of a calculation using an actual sample and results from the PFOA analysis follows:

Water sample Exygen ID C0064432 Spk F (Set: 032105BR), fortified at 50000 ng/L with PFOA where:

peak area	=	1170161
intercept	=	26200
slope	=	1330
dilution factor	=	100
ng/L PFOA added (fort level)	=	50000
amt in corresponding sample (ng/L)*	=	37600

(*The primary sample result was used for all calculations)

From equation 1:

$$\text{Analyte found (ng/L)} = \frac{[1170161 - 26200] \times 100}{1330}$$

$$= 86012 \text{ ng/L}^*$$

From equation 2:

$$\% \text{ Recovery} = \frac{(86012 \text{ ng/L} - 37600 \text{ ng/L}) \times 100\%}{50000 \text{ ng/L}}$$

$$= 97 \%$$

(*values are slightly different from raw data due to rounding)

7.0 EXPERIMENTAL DESIGN

¹³C PFOA was used as a surrogate for all the samples. ¹³C PFOA was added to the sample collection bottles in the laboratory before being shipped to the field for sampling. For samples designated as field matrix spikes, PFBS, PFHS, PFOS and PFOA were also 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.

The samples were extracted in five sets. Each set consisted of one reagent blank and two reagent blanks fortified at known concentrations. The first set contained six sample sites, the second set contained six samples sites, along with a trip blank and two trip blank spikes. The next two sets contained seven sample sites each. The fifth set contained three sample sites, along with a second trip blank and two trip blank spikes. For each site, a sample, a field duplicate and two-matrix field spikes were collected. For each site, a laboratory duplicate of the primary sample was extracted and two laboratory matrix spikes were also extracted. For the two laboratory matrix spikes, two 40 mL portions of the primary sample collected for the site was poured from the bottle and fortified. Not only were PFBS, PFHS, PFOS and PFOA added in the laboratory prior to extraction, but also ¹³C PFOA was added. The additional ¹³C PFOA was added because the levels of PFBS, PFHS, PFOS and PFOA spiked into the samples were known to exceed the calibration ranges and were not analyzed without dilution; therefore, ¹³C PFOA levels were adjusted to require the same dilution as the other analytes.

For each primary sample collected, between the field and lab, seven individual QC results were reported for ¹³C PFOA. Some samples gave recoveries for ¹³C PFOA outside of the normal acceptance range of 70-130%. Because laboratory control spikes for ¹³C PFOA were acceptable in every set, instrument washes were free of ¹³C PFOA, at least one ¹³C PFOA recovery per the seven done for the sample was acceptable and no evident errors in spiking or extracting could be found, no re-extractions were performed. ¹³C PFOA recoveries outside of 70-130% are deemed indicative of matrix effects.

8.0 RESULTS

Analytical results for the analysis of PFOA, PFBS, PFHS, and PFOS in water samples are summarized in **Table I**.

Fortification recoveries for PFOA, PFBS, PFHS and PFOS in the water samples are detailed in **Tables II and III**. The overall average percent recoveries $\pm$ standard deviations for all fortifications of PFOA, PFBS, PFHS and PFOS in water samples were $93\% \pm 19$, $141\% \pm 21$, $96\% \pm 15$ and $109\% \pm 32$, respectively. Fortification recoveries for ^{13}C PFOA in the water samples are detailed in **Table IV**. The overall average percent recovery $\pm$ standard deviation for all fortifications of ^{13}C -PFOA in water samples was $83\% \pm 21$.

9.0 CONCLUSIONS

The water samples were successfully extracted and analyzed for PFBS, PFHS, PFOS and PFOA according to analytical method V0001780. There were no circumstances that may have affected the data quality or integrity.

10.0 RETENTION OF DATA AND SAMPLES

All original paper data generated by Exygen Research that pertains to this interim report will be shipped to the sponsor. 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 interim analytical report and all original facility-specific raw data, will be retained in the Exygen Research archives for the period of time specified in EPA TSCA Good Laboratory Practice Standards 40 CFR 792.

TABLES

Table I. Summary of PFBS, PFHS, PFOS and PFOA in Water Samples

Exygen ID	Client Sample ID	Analyte Found (ppt, ng/L)			
		C4 Sulfonate PFBS	C6 Sulfonate PFHS	C8 Sulfonate PFOS	C8 Acid PFOA
C0064404	OKLMN-GW-W3-0-050310	312	162	4750	6330
C0064404 Rep	OKLMN-GW-W3-0-050310*	296	160	3350	6220
C0064405	OKLMN-GW-W3-0-050310 Dup	303	159	3190	5990
C0064408	OKLMN-GW-W8-0-050310	57.0	ND	56.0	290
C0064408 Rep	OKLMN-GW-W8-0-050310*	63.8	ND	55.7	275
C0064409	OKLMN-GW-W8-0-050310 Dup	60.1	ND	56.3	284
C0064412	OKLMN-GW-W26-0-050310	73400	222000	13500000	79300000
C0064412 Rep	OKLMN-GW-W26-0-050310*	75700	244000	14400000	70200000
C0064413	OKLMN-GW-W26-0-050310 Dup	71400	214000	12200000	71800000
C0064416	OKLMN-GW-W33-0-050310	26800	92300	4810000	1750000
C0064416 Rep	OKLMN-GW-W33-0-050310*	26800	84800	4870000	2020000
C0064417	OKLMN-GW-W33-0-050310 Dup	28500	81600	4540000	2790000
C0064420	OKLMN-GW-W205-0-050310	8260	6740	146000	147000
C0064420 Rep	OKLMN-GW-W205-0-050310*	8800	6610	119000	150000
C0064421	OKLMN-GW-W205-0-050310 Dup	7850	7070	116000	158000
C0064424	OKLMN-GW-W215-0-050310	801	789	37100	4400
C0064424 Rep	OKLMN-GW-W215-0-050310*	81.2	256	27000	3510
C0064425	OKLMN-GW-W215-0-050310 Dup	75.8	270	36900	4070
C0064428	OKLMN-GW-W2010-0-050310	ND	ND	ND	NQ
C0064428 Rep	OKLMN-GW-W2010-0-050310*	ND	ND	ND	ND
C0064429	OKLMN-GW-W2010-0-050310 Dup	ND	ND	ND	ND
C0064432	OKLMN-GW-PW7-0-050310	4460	4800	71600	37600
C0064432 Rep	OKLMN-GW-PW7-0-050310*	4340	5080	67600	38400
C0064433	OKLMN-GW-PW7-0-050310 Dup	4630	5080	72400	38200
C0064436	OKLMN-GW-TRIP1-0-050310	ND	ND	ND	ND
C0064442	OKLMN-GW-PW8-0-050310	13300	23800	34300	61400
C0064442 Rep	OKLMN-GW-PW8-0-050310*	14100	23900	35600	60600
C0064443	OKLMN-GW-PW8-0-050310 Dup	14100	23000	33400	58500
C0064446	OKLMN-GW-PW9-0-050310	2110	5180	84400	49000
C0064446 Rep	OKLMN-GW-PW9-0-050310*	2140	5000	84900	46700
C0064447	OKLMN-GW-PW9-0-050310 Dup	1930	5140	88600	49600
C0064450	OKLMN-GW-W6102-0-050310	NQ	ND	NQ	214
C0064450 Rep	OKLMN-GW-W6102-0-050310*	53.5	ND	NQ	228
C0064451	OKLMN-GW-W6102-0-050310 Dup	NQ	ND	ND	227
C0064454	OKLMN-GW-W6104-0-050310	NQ	ND	109	207
C0064454 Rep	OKLMN-GW-W6104-0-050310*	NQ	ND	125	189
C0064455	OKLMN-GW-W6104-0-050310 Dup	51.2	ND	118	164
C0064458	OKLMN-GW-W6201-0-050310	74.0	ND	NQ	95.5
C0064458 Rep	OKLMN-GW-W6201-0-050310*	74.1	ND	NO	88.3
C0064459	OKLMN-GW-W6201-0-050310 Dup	78.0	ND	NQ	132
C0064467	OKLMN-GW-W20-0-050309	13900	12900	46600	285000
C0064467 Rep	OKLMN-GW-W20-0-050309*	10700	9040	38300	243000
C0064468	OKLMN-GW-W20-0-050309 Dup	10000	7860	30100	190000
C0064471	OKLMN-GW-W22-0-050309	36600	31400	45800	317000
C0064471 Rep	OKLMN-GW-W22-0-050309*	32200	29500	34000	301000
C0064472	OKLMN-GW-W22-0-050309 Dup	37500	25700	26100	244000

*Laboratory Duplicate

ND = Not detected at or above 25 ng/L

NQ = Not quantifiable = Measured concentration between 25 ng/L and the Limit of Quantitation (LOQ) which is 50 ng/L

Table I. Summary of PFBS, PFHS, PFOS and PFOA in Water Samples Continued

Exygen ID	Client Sample ID	Analyte Found (ppt, ng/L)			
		C4 Sulfonate PFBS	C6 Sulfonate PFHS	C8 Sulfonate PFOS	C8 Acid PFOA
C0064475	OKLMN-GW-W31-0-050309	1300	5130	132000	23100
C0064475 Rep	OKLMN-GW-W31-0-050309*	1360	4780	132000	22300
C0064476	OKLMN-GW-W31-0-050309 Dup	1360	5190	156000	24900
C0064478	OKLMN-GW-W32-0-050309	2180	3000	47100	117000
C0064478 Rep	OKLMN-GW-W32-0-050309*	2290	3120	44400	93300
C0064479	OKLMN-GW-W32-0-050309 Dup	2960	2940	63000	125000
C0064482	OKLMN-GW-W217-0-050309	ND	ND	NQ	NQ
C0064482 Rep	OKLMN-GW-W217-0-050309*	ND	ND	NQ	NQ
C0064483	OKLMN-GW-W217-0-050309 Dup	ND	ND	128	NQ
C0064486	OKLMN-GW-W2008-0-050309	6780	10100	15500	59300
C0064486 Rep	OKLMN-GW-W2008-0-050309*	7170	10800	17800	58700
C0064487	OKLMN-GW-W2008-0-050309 Dup	6930	10300	18800	60300
C0064490	OKLMN-GW-W2012-0-050309	2210	4700	117000	48900
C0064490 Rep	OKLMN-GW-W2012-0-050309*	1960	4470	118000	46400
C0064491	OKLMN-GW-W2012-0-050309 Dup	1920	4420	139000	51400
C0064494	OKLMN-GW-W80-0-050309	ND	ND	ND	302
C0064494 Rep	OKLMN-GW-W80-0-050309*	ND	ND	ND	280
C0064495	OKLMN-GW-W80-0-050309 Dup	ND	ND	ND	328
C0064498	OKLMN-GW-PW1-0-050310	6620	4180	66900	37300
C0064498 Rep	OKLMN-GW-PW1-0-050310*	5650	4150	71600	38300
C0064499	OKLMN-GW-PW1-0-050310 Dup	5580	4170	70900	39100
C0064502	OKLMN-GW-PW2-0-050310	641	501	351	3630
C0064502 Rep	OKLMN-GW-PW2-0-050310*	624	491	383	3890
C0064503	OKLMN-GW-PW2-0-050310 Dup	894	700	303	5160
C0064506	OKLMN-GW-PW3-0-050310	6970	10000	32700	177000
C0064506 Rep	OKLMN-GW-PW3-0-050310*	6740	9740	25500	160000
C0064507	OKLMN-GW-PW3-0-050310 Dup	6570	9990	32300	176000
C0064510	OKLMN-GW-PW4-0-050310	8130	7890	33800	72100
C0064510 Rep	OKLMN-GW-PW4-0-050310*	7700	7480	30300	67800
C0064511	OKLMN-GW-PW4-0-050310 Dup	7810	7550	27800	65900
C0064514	OKLMN-GW-PW6-0-050310	7930	14500	90000	78400
C0064514 Rep	OKLMN-GW-PW6-0-050310*	8000	14600	86000	78700
C0064515	OKLMN-GW-PW6-0-050310 Dup	8130	14600	86400	78500
C0064518	OKLMN-GW-PW10-0-050310	13100	29200	1400000	719000
C0064518 Rep	OKLMN-GW-PW10-0-050310*	12700	27400	1780000	781000
C0064519	OKLMN-GW-PW10-0-050310 Dup	11900	26800	1740000	814000
C0064522	OKLMN-GW-PW11-0-050310	6520	8820	40800	50300
C0064522 Rep	OKLMN-GW-PW11-0-050310*	6490	10090	50800	71600
C0064523	OKLMN-GW-PW11-0-050310 Dup	7150	9050	43400	60600
C0064526	OKLMN-SW-0-050310	NQ	NQ	1190	491
C0064526 Rep	OKLMN-SW-0-050310*	NQ	NQ	1200	529
C0064527	OKLMN-SW-0-050310 Dup	NQ	NQ	1270	537
C0064530	OKLMN-GW-TRIP2-0-050310	ND	ND	ND	ND

*Laboratory Duplicate

ND = Not detected at or above 25 ng/L.

NQ = Not quantifiable = Measured concentration between 25 ng/L and the Limit of Quantitation (LOQ) which is 50 ng/L.

Table II. Matrix Spike Recovery of PFBS and PFHS in Water Samples

Sample Description	C4 Sulfonate PFBS				C8 Sulfonate PFHS			
	Amount Spiked (ng/L)	Amount Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amount Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-W3-0-050310 (C0084404 Spk C, 5000 ng/L Lab Spike)	5000	312	6980	133	5000	182	5130	59
OKLMN-GW-W3-0-050310 (C0084404 Spk D, 50000 ng/L Lab Spike)	50000	312	87400	134	50000	162	47500	95
OKLMN-GW-W3-0-050310 Low Spike 5 ppb (C0084406, 5000 ng/L Field Spike)	5000	312	7450	143	5000	162	5140	100
OKLMN-GW-W3-0-050310 High Spike 50 ppb (C0084407, 50000 ng/L Field Spike)	50000	312	74700	148	50000	162	54400	108
OKLMN-GW-W8-0-050310 (C0084408 Spk E, 5000 ng/L Lab Spike)	5000	57.0	7020	138	5000	ND	5120	102
OKLMN-GW-W8-0-050310 (C0084408 Spk F, 50000 ng/L Lab Spike)	50000	57.0	70000	140	50000	ND	51400	103
OKLMN-GW-W8-0-050310 Low Spike 5 ppb (C0084410, 5000 ng/L Field Spike)	5000	57.0	7410	147	5000	ND	5510	110
OKLMN-GW-W8-0-050310 High Spike 50 ppb (C0084411, 50000 ng/L Field Spike)	50000	57.0	70500	141	50000	ND	53300	107
OKLMN-GW-W28-0-050310 (C0084412 Spk G, 5000 ng/L Lab Spike)	5000	73400	73100	*	5000	222000	207000	*
OKLMN-GW-W28-0-050310 (C0084412 Spk H, 50000 ng/L Lab Spike)	50000	73400	125000	103	50000	222000	252000	*
OKLMN-GW-W28-0-050310 Low Spike 5 ppb (C0084414, 5000 ng/L Field Spike)	5000	73400	78200	*	5000	222000	210000	*
OKLMN-GW-W28-0-050310 High Spike 50 ppb (C0084415, 50000 ng/L Field Spike)	50000	73400	147000	147	50000	222000	288000	*
OKLMN-GW-W33-0-050310 (C0084418 Spk I, 5000 ng/L Lab Spike)	5000	26800	33600	*	5000	82300	78000	*
OKLMN-GW-W33-0-050310 (C0084418 Spk J, 50000 ng/L Lab Spike)	50000	26800	133000	212	50000	82300	122000	59
OKLMN-GW-W33-0-050310 Low Spike 5 ppb (C0084418, 5000 ng/L Field Spike)	5000	26800	38700	*	5000	82300	85200	*
OKLMN-GW-W33-0-050310 High Spike 50 ppb (C0084419, 50000 ng/L Field Spike)	50000	26800	108000	102	50000	82300	150000	115
OKLMN-GW-W205-0-050310 (C0084420 Spk K, 5000 ng/L Lab Spike)	5000	8280	15400	143	5000	6740	12500	115
OKLMN-GW-W205-0-050310 (C0084420 Spk L, 50000 ng/L Lab Spike)	50000	8280	72500	128	50000	6740	53100	93
OKLMN-GW-W205-0-050310 Low Spike 5 ppb (C0084422, 5000 ng/L Field Spike)	5000	8280	15200	130	5000	6740	11800	101
OKLMN-GW-W205-0-050310 High Spike 50 ppb (C0084423, 50000 ng/L Field Spike)	50000	8280	82800	149	50000	6740	60600	108
OKLMN-GW-W215-0-050310 (C0084424 Spk M, 5000 ng/L Lab Spike)	5000	801.0	7200	128	5000	769	5280	90
OKLMN-GW-W215-0-050310 (C0084424 Spk N, 50000 ng/L Lab Spike)	50000	801.0	87200	133	50000	769	50300	99
OKLMN-GW-W215-0-050310 Low Spike 5 ppb (C0084426, 5000 ng/L Field Spike)	5000	801.0	8720	118	5000	769	5240	88
OKLMN-GW-W215-0-050310 High Spike 50 ppb (C0084427, 50000 ng/L Field Spike)	50000	801.0	86900	132	50000	769	48500	85

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 25 ng/L (ppt)

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of Quantitation (LOQ) which is 50 ng/L (ppt)

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

Table II. Matrix Spike Recovery of PFBS and PFHS in Water Samples Continued

Sample Description	C4 Sulfonate PFBS				C6 Sulfonate PFHS			
	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-W2010-0-050310 (C0064428 Spk C, 5000 ng/L Lab Spike)	5000	ND	6590	132	5000	ND	4800	96
OKLMN-GW-W2010-0-050310 (C0064432 Spk D, 50000 ng/L Lab Spike)	50000	ND	69500	139	50000	ND	48700	97
OKLMN-GW-W2010-0-050310 Low Spike 5 ppb (C0064430, 5000 ng/L Field Spike)	5000	ND	6700	134	5000	ND	6700	94
OKLMN-GW-W2010-0-050310 High Spike 50 ppb (C0064431, 50000 ng/L Field Spike)	50000	ND	83800	128	50000	ND	43700	87
OKLMN-GW-PW7-0-050310 (C0064432 Spk E, 5000 ng/L Lab Spike)	5000	4460	12200	155	5000	4900	9310	90
OKLMN-GW-PW7-0-050310 (C0064432 Spk F, 50000 ng/L Lab Spike)	50000	4480	72900	137	50000	4800	50900	92
OKLMN-GW-PW7-0-050310 Low Spike 5 ppb (C0064434, 5000 ng/L Field Spike)	5000	4480	13200	175	5000	4800	9370	91
OKLMN-GW-PW7-0-050310 High Spike 50 ppb (C0064435, 50000 ng/L Field Spike)	50000	4460	80000	151	50000	4800	59000	108
OKLMN-GW-TRIP1-0-050310 Low Spike 5 ppb (C0064437, 5000 ng/L Field Spike)	5000	ND	6540	131	5000	ND	4590	92
OKLMN-GW-TRIP1-0-050310 High Spike 50 ppb (C0064438, 50000 ng/L Field Spike)	50000	ND	44400	89	50000	ND	46100	92
OKLMN-GW-PW8-0-050310 (C0064442 Spk G, 5000 ng/L Lab Spike)	5000	13300	21400	162	5000	23800	29700	118
OKLMN-GW-PW8-0-050310 (C0064442 Spk H, 50000 ng/L Lab Spike)	50000	13300	79700	133	50000	23800	70300	90
OKLMN-GW-PW8-0-050310 Low Spike 5 ppb (C0064444, 5000 ng/L Field Spike)	5000	13300	21000	154	5000	23800	30000	124
OKLMN-GW-PW8-0-050310 High Spike 50 ppb (C0064445, 50000 ng/L Field Spike)	50000	13300	80700	135	50000	23800	78100	105
OKLMN-GW-PW9-0-050310 (C0064446 Spk I, 5000 ng/L Lab Spike)	5000	2110	8290	144	5000	5180	9310	83
OKLMN-GW-PW9-0-050310 (C0064446 Spk J, 50000 ng/L Lab Spike)	50000	2110	68900	134	50000	5180	54400	88
OKLMN-GW-PW9-0-050310 Low Spike 5 ppb (C0064448, 5000 ng/L Field Spike)	5000	2110	8290	144	5000	5180	9350	84
OKLMN-GW-PW9-0-050310 High Spike 50 ppb (C0064448, 50000 ng/L Field Spike)	50000	2110	74800	146	50000	5180	54800	98
OKLMN-GW-W8102-0-050310 (C0064450 Spk K, 5000 ng/L Lab Spike)	5000	NQ	7350	147	5000	ND	4890	98
OKLMN-GW-W8102-0-050310 (C0064450 Spk L, 50000 ng/L Lab Spike)	50000	NQ	87500	135	50000	ND	47400	95
OKLMN-GW-W8102-0-050310 Low Spike 5 ppb (C0064452, 5000 ng/L Field Spike)	5000	NQ	7120	142	3000	ND	5040	101
OKLMN-GW-W8102-0-050310 High Spike 50 ppb (C0064453, 50000 ng/L Field Spike)	50000	NQ	70700	141	50000	ND	50800	101

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 25 ng/L (ppt).

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of Quantitation (LOQ) which is 50 ng/L (ppt).

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

Table II. Matrix Spike Recovery of PFBS and PFHS in Water Samples Continued

Sample Description	C4 Sulfonate PFBS				C6 Sulfonate PFHS			
	Amount Spiked (ng/L)	Amount Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amount Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-W8104-0-050310 (C0084454 Spk M, 5000 ng/L Lab Spike)	5000	NQ	8680	134	5000	ND	5050	101
OKLMN-GW-W8104-0-050310 (C0084454 Spk N, 50000 ng/L Lab Spike)	50000	NQ	73000	146	50000	ND	48000	96
OKLMN-GW-W8104-0-050310 Low Spike 5 ppb (C0084454, 5000 ng/L Field Spike)	5000	NQ	8730	135	5000	ND	4720	94
OKLMN-GW-W8104-0-050310 High Spike 50 ppb (C0084457, 50000 ng/L Field Spike)	50000	NQ	69700	139	50000	ND	51200	102
OKLMN-GW-W8201-0-050310 (C0084454 Spk C, 5000 ng/L Lab Spike)	5000	74.0	6580	130	5000	ND	4610	92
OKLMN-GW-W8201-0-050310 (C0084454 Spk D, 50000 ng/L Lab Spike)	50000	74.0	65500	131	50000	ND	44400	89
OKLMN-GW-W8201-0-050310 Low Spike 0.5 ppb (C0084460, 500 ng/L Field Spike)	500	74.0	677	121	500	ND	543	108
OKLMN-GW-W8201-0-050310 High Spike 5 ppb (C0084461, 5000 ng/L Field Spike)	5000	74.0	7560	150	5000	ND	5380	108
OKLMN-GW-W20-0-050309 (C0084467 Spk E, 5000 ng/L Lab Spike)	5000	13900	19400	110	5000	12900	16200	86
OKLMN-GW-W20-0-050309 (C0084467 Spk F, 50000 ng/L Lab Spike)	50000	13900	77100	126	50000	12900	56200	85
OKLMN-GW-W20-0-050309 Low Spike 5 ppb (C0084466, 5000 ng/L Field Spike)	5000	13900	20900	140	5000	12900	16200	86
OKLMN-GW-W20-0-050309 High Spike 50 ppb (C0084470, 50000 ng/L Field Spike)	50000	13900	85000	142	50000	12900	48400	71
OKLMN-GW-W22-0-050309 (C0084471 Spk G, 5000 ng/L Lab Spike)	5000	36800	44100	*	5000	31400	37000	*
OKLMN-GW-W22-0-050309 (C0084471 Spk H, 50000 ng/L Lab Spike)	50000	36800	109000	145	50000	31400	73000	83
OKLMN-GW-W22-0-050309 Low Spike 5 ppb (C0084473, 5000 ng/L Field Spike)	5000	36800	44000	*	5000	31400	35900	*
OKLMN-GW-W22-0-050309 High Spike 50 ppb (C0084474, 50000 ng/L Field Spike)	50000	36800	111000	149	50000	31400	62200	62
OKLMN-GW-W31-0-050309 (C0084475 Spk I, 5000 ng/L Lab Spike)	5000	1300	6450	143	5000	5130	8090	79
OKLMN-GW-W31-0-050309 (C0084475 Spk J, 50000 ng/L Lab Spike)	50000	1300	70500	138	50000	5130	51200	92
OKLMN-GW-W31-0-050309 High Spike 50 ppb (C0084477, 50000 ng/L Field Spike)	50000	1300	90900	179	50000	5130	63200	116
OKLMN-GW-W32-0-050309 (C0084479 Spk K, 5000 ng/L Lab Spike)	5000	2180	8910	135	5000	3000	7400	88
OKLMN-GW-W32-0-050309 (C0084479 Spk L, 50000 ng/L Lab Spike)	50000	2180	87300	130	50000	3000	50900	94
OKLMN-GW-W32-0-050309 Low Spike 5 ppb (C0084480, 5000 ng/L Field Spike)	5000	2180	10800	172	5000	3000	7910	98
OKLMN-GW-W32-0-050309 High Spike 50 ppb (C0084481, 50000 ng/L Field Spike)	50000	2180	65500	127	50000	3000	52800	100

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above 25 ng/L (ppt).

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of Quantitation (LOQ) which is 50 ng/L (ppt).

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

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Table II. Matrix Spike Recovery of PFBS and PFHS in Water Samples Continued

Sample Description	C4 Sulfonate PFBS				C8 Sulfonate PFHS			
	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-W217-0-050309 (C0064482 Spk M, 5000 ng/L Lab Spike)	5000	ND	8680	134	5000	ND	4890	98
OKLMN-GW-W217-0-050309 (C0064482 Spk M, 50000 ng/L Lab Spike)	50000	ND	86800	134	50000	ND	48500	93
OKLMN-GW-W217-0-050309 Low Spike 3 ppb (C0064484, 3000 ng/L Field Spike)	5000	ND	7570	151	5000	ND	4590	92
OKLMN-GW-W217-0-050309 High Spike 80 ppb (C0064485, 80000 ng/L Field Spike)	50000	ND	71200	142	50000	ND	51300	103
OKLMN-GW-W2008-0-050309 (C0064486 Spk O, 5000 ng/L Lab Spike)	5000	6780	14300	150	5000	10100	16000	118
OKLMN-GW-W2008-0-050309 (C0064486 Spk P, 50000 ng/L Lab Spike)	50000	6780	73800	134	50000	10100	56500	93
OKLMN-GW-W2008-0-050309 Low Spike 5 ppb (C0064488, 5000 ng/L Field Spike)	5000	6780	18500	194	5000	10100	19300	184
OKLMN-GW-W2008-0-050309 High Spike 50 ppb (C0064488, 50000 ng/L Field Spike)	50000	6780	87900	182	50000	10100	84800	109
OKLMN-GW-W2012-0-050309 (C0064489 Spk C, 5000 ng/L Lab Spike)	5000	2210	8800	132	5000	4700	8140	89
OKLMN-GW-W2012-0-050309 (C0064489 Spk D, 50000 ng/L Lab Spike)	50000	2210	86400	128	50000	4700	46900	84
OKLMN-GW-W2012-0-050309 Low Spike 5 ppb (C0064492, 5000 ng/L Field Spike)	5000	2210	8210	120	5000	4700	8950	85
OKLMN-GW-W2012-0-050309 High Spike 50 ppb (C0064493, 50000 ng/L Field Spike)	50000	2210	71500	139	50000	4700	53800	98
OKLMN-GW-W80-0-050309 (C0064494 Spk E, 5000 ng/L Lab Spike)	5000	ND	8850	137	5000	ND	4990	100
OKLMN-GW-W80-0-050309 (C0064494 Spk F, 50000 ng/L Lab Spike)	50000	ND	64700	129	50000	ND	46200	92
OKLMN-GW-W80-0-050309 Low Spike 5 ppb (C0064496, 5000 ng/L Field Spike)	5000	ND	7390	148	5000	ND	5200	104
OKLMN-GW-W80-0-050309 High Spike 50 ppb (C0064497, 50000 ng/L Field Spike)	50000	ND	69700	139	50000	ND	48200	98
OKLMN-GW-PW1-0-050310 (C0064499 Spk G, 5000 ng/L Lab Spike)	5000	5620	13600	160	5000	4180	8880	94
OKLMN-GW-PW1-0-050310 (C0064499 Spk H, 50000 ng/L Lab Spike)	50000	5620	72000	133	50000	4180	52000	96
OKLMN-GW-PW1-0-050309 Low Spike 5 ppb (C0064500, 5000 ng/L Field Spike)	5000	5620	13100	150	5000	4150	7690	71
OKLMN-GW-PW1-0-050309 High Spike 50 ppb (C0064501, 50000 ng/L Field Spike)	50000	5620	69700	128	50000	4180	47700	87
OKLMN-GW-PW2-0-050310 (C0064502 Spk I, 5000 ng/L Lab Spike)	5000	641	7720	142	5000	501	5280	95
OKLMN-GW-PW2-0-050310 (C0064502 Spk J, 50000 ng/L Lab Spike)	50000	641	65900	131	50000	501	47700	94
OKLMN-GW-PW2-0-050310 Low Spike 5 ppb (C0064504, 5000 ng/L Field Spike)	5000	641	7030	128	5000	501	5190	94
OKLMN-GW-PW2-0-050310 High Spike 50 ppb (C0064505, 50000 ng/L Field Spike)	50000	641	64300	127	50000	501	44300	89

* Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 25 ng/L (ppt)

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of Quantitation (LOQ) which is 50 ng/L (ppt)

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

Table II. Matrix Spike Recovery of PFBS and PFHS in Water Samples Continued

Sample Description	C4 Sulfonate PFBS				C8 Sulfonate PFHS			
	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-PW3-0-050310 (C0064506 Spk K, 5000 ng/L Lab Spike)	5000	6970	13800	137	5000	10000	13800	72
OKLMN-GW-PW3-0-050310 (C0064506 Spk L, 50000 ng/L Lab Spike)	50000	6970	98900	180	50000	10000	54400	86
OKLMN-GW-PW3-0-050310 Low Spike 5 ppb (C0064506, 5000 ng/L Field Spike)	5000	6970	15200	165	5000	10000	16300	128
OKLMN-GW-PW3-0-050310 High Spike 50 ppb (C0064506, 50000 ng/L Field Spike)	50000	6970	92300	171	50000	10000	54900	90
OKLMN-GW-PW4-0-050310 (C0064518 Spk M, 5000 ng/L Lab Spike)	5000	8130	17400	185	5000	7890	13900	120
OKLMN-GW-PW4-0-050310 (C0064518 Spk N, 50000 ng/L Lab Spike)	50000	8130	95000	174	50000	7890	53600	91
OKLMN-GW-PW4-0-050310 Low Spike 5 ppb (C0064518, 5000 ng/L Field Spike)	5000	8130	16300	183	5000	7890	13500	112
OKLMN-GW-PW4-0-050310 High Spike 50 ppb (C0064518, 50000 ng/L Field Spike)	50000	8130	88900	162	50000	7890	58200	101
OKLMN-GW-PW6-0-050310 (C0064514 Spk O, 5000 ng/L Lab Spike)	5000	7930	17200	185	5000	14500	20800	122
OKLMN-GW-PW6-0-050310 (C0064514 Spk P, 50000 ng/L Lab Spike)	50000	7930	90500	165	50000	14500	58800	89
OKLMN-GW-PW6-0-050310 Low Spike 5 ppb (C0064514, 5000 ng/L Field Spike)	5000	7930	17100	183	5000	14500	20800	128
OKLMN-GW-PW6-0-050310 High Spike 50 ppb (C0064517, 50000 ng/L Field Spike)	50000	7930	94800	173	50000	14500	69400	90
OKLMN-GW-PW10-0-050310 (C0064519 Spk C, 5000 ng/L Lab Spike)	5000	13100	19300	124	5000	29200	34300	102
OKLMN-GW-PW10-0-050310 (C0064519 Spk D, 50000 ng/L Lab Spike)	50000	13100	78700	133	50000	29200	71000	84
OKLMN-GW-PW10-0-050310 Low Spike 5 ppb (C0064520, 5000 ng/L Field Spike)	5000	13100	19000	118	5000	29200	33500	86
OKLMN-GW-PW10-0-050310 High Spike 50 ppb (C0064521, 50000 ng/L Field Spike)	50000	13100	74000	122	50000	29200	71300	84
OKLMN-GW-PW11-0-050310 (C0064522 Spk E, 5000 ng/L Lab Spike)	5000	6520	12700	124	5000	8820	14200	100
OKLMN-GW-PW11-0-050310 (C0064522 Spk F, 50000 ng/L Lab Spike)	50000	6520	88300	124	50000	8820	65100	89
OKLMN-GW-PW11-0-050310 Low Spike 5 ppb (C0064524, 5000 ng/L Field Spike)	5000	6520	13200	134	5000	8820	13300	90
OKLMN-GW-PW11-0-050310 High Spike 50 ppb (C0064525, 50000 ng/L Field Spike)	50000	6520	63800	114	50000	8820	54800	92

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 25 ng/L (ppt).

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of Quantitation (LOQ) which is 50 ng/L (ppt).

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

Table II. Matrix Spike Recovery of PFBS and PFHS in Water Samples Continued

	C4 Sulfonate PFBS				C6 Sulfonate PFHS				
Sample Description	Amount Spiked (ng/L)	Amt Found In Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found In Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	
OKLMN-SW-0-050310 (C0084828 Spk G, 5000 ng/L Lab Spike)	5000	NQ	5890	120	5000	NQ	4790	96	
OKLMN-SW-0-050310 (C0084828 Spk H, 50000 ng/L Lab Spike)	50000	NQ	63900	128	50000	NQ	45100	90	
OKLMN-SW-0-050310 Low Spike 5 ppb (C0084828, 5000 ng/L Field Spike)	5000	NQ	7280	145	5000	NQ	5250	105	
OKLMN-SW-0-050310 High Spike 50 ppb (C0084828, 50000 ng/L Field Spike)	50000	NQ	65000	130	50000	NQ	49400	99	
OKLMN-GW-TRIP2-0-050310 Low Spike 5 ppb (C0084631, 5000 ng/L Spike)	5000	ND	4490	90	5000	ND	4970	99	
OKLMN-GW-TRIP2-0-050310 High Spike 50 ppb (C0084632, 50000 ng/L Spike)	50000	ND	28100	58	50000	ND	49000	98	
Average:				141	Average:				98
Standard Deviation:				21	Standard Deviation:				15

Table III. Matrix Spike Recovery of PFOS and PFOA in Water Samples

Sample Description	C8 Sulfonate PFOS				C8 Acid PFOA			
	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-W3-0-050310 (C0064404 Spk C, 5000 ng/L Lab Spike)	5000	4750	12400	163	5000	6330	11900	111
OKLMN-GW-W3-0-050310 (C0064404 Spk D, 50000 ng/L Lab Spike)	50000	4750	73500	138	50000	8330	59200	106
OKLMN-GW-W3-0-050310 Low Spike 5 ppb (C0064404, 5000 ng/L Field Spike)	5000	4750	10800	123	5000	6330	9470	83
OKLMN-GW-W3-0-050310 High Spike 50 ppb (C0064407, 50000 ng/L Field Spike)	50000	4750	84500	160	50000	6330	40100	80
OKLMN-GW-W8-0-050310 (C0064408 Spk E, 5000 ng/L Lab Spike)	5000	59.0	4940	98	5000	290	5350	101
OKLMN-GW-W8-0-050310 (C0064408 Spk F, 50000 ng/L Lab Spike)	50000	59.0	55000	110	50000	290	51900	103
OKLMN-GW-W8-0-050310 Low Spike 5 ppb (C0064410, 5000 ng/L Field Spike)	5000	59.0	5380	106	5000	290	8050	116
OKLMN-GW-W8-0-050310 High Spike 50 ppb (C0064411, 50000 ng/L Field Spike)	50000	59.0	50500	101	50000	290	38500	77
OKLMN-GW-W26-0-050310 (C0064412 Spk G, 5000 ng/L Lab Spike)	5000	13500000	9450000	*	5000	79300000	66300800	*
OKLMN-GW-W26-0-050310 (C0064412 Spk H, 50000 ng/L Lab Spike)	50000	13500000	7390000	*	50000	79300000	68300000	*
OKLMN-GW-W26-0-050310 Low Spike 5 ppb (C0064414, 5000 ng/L Field Spike)	5000	13500000	8050000	*	5000	79300000	73100000	*
OKLMN-GW-W26-0-050310 High Spike 50 ppb (C0064415, 50000 ng/L Field Spike)	50000	13500000	7210000	*	50000	79300000	57900000	*
OKLMN-GW-W33-0-050310 (C0064416 Spk I, 5000 ng/L Lab Spike)	5000	4610000	4720000	*	5000	1750000	1520000	*
OKLMN-GW-W33-0-050310 (C0064416 Spk J, 50000 ng/L Lab Spike)	50000	4610000	4730000	*	50000	1750000	1650000	*
OKLMN-GW-W33-0-050310 Low Spike 5 ppb (C0064418, 5000 ng/L Field Spike)	5000	4610000	4820000	*	5000	1750000	2350000	*
OKLMN-GW-W33-0-050310 High Spike 50 ppb (C0064418, 50000 ng/L Field Spike)	50000	4610000	4520000	*	50000	1750000	1630000	*
OKLMN-GW-W205-0-050310 (C0064420 Spk K, 5000 ng/L Lab Spike)	5000	148000	136000	*	5000	147000	160000	*
OKLMN-GW-W205-0-050310 (C0064420 Spk L, 50000 ng/L Lab Spike)	50000	148000	156000	20	50000	147000	190000	88
OKLMN-GW-W205-0-050310 Low Spike 5 ppb (C0064422, 5000 ng/L Field Spike)	5000	148000	129000	*	5000	147000	143000	*
OKLMN-GW-W205-0-050310 High Spike 50 ppb (C0064423, 50000 ng/L Field Spike)	50000	148000	221000	150	50000	147000	184000	74
OKLMN-GW-W215-0-050310 (C0064424 Spk M, 5000 ng/L Lab Spike)	5000	37100	52400	*	5000	4400	8850	89
OKLMN-GW-W215-0-050310 (C0064424 Spk N, 50000 ng/L Lab Spike)	50000	37100	96800	120	50000	4400	56700	105
OKLMN-GW-W215-0-050310 Low Spike 5 ppb (C0064426, 5000 ng/L Field Spike)	5000	37100	58600	*	5000	4400	8690	85
OKLMN-GW-W215-0-050310 High Spike 50 ppb (C0064427, 50000 ng/L Field Spike)	50000	37100	101000	128	50000	4400	38200	70

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 25 ng/L (ppt)

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of Quantitation (LOQ) which is 50 ng/L (ppt)

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

Table III. Matrix Spike Recovery of PFOS and PFOA in Water Samples Continued

Sample Description	C8 Sulfonate PFOS				C8 Acid PFOA			
	Amount Spiked (ng/L)	Amt Found In Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found In Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-W2010-0-050310 (C0064428 Spk C, 5000 ng/L Lab Spike)	5000	ND	5170	103	5000	NQ	5290	106
OKLMN-GW-W2010-0-050310 (C0064428 Spk D, 50000 ng/L Lab Spike)	50000	ND	54600	109	50000	NQ	54300	109
OKLMN-GW-W2010-0-050310 Low Spike 5 ppb (C0064430, 5000 ng/L Field Spike)	5000	ND	5270	105	5000	NQ	4710	94
OKLMN-GW-W2010-0-050310 High Spike 50 ppb (C0064431, 50000 ng/L Field Spike)	50000	ND	54800	110	50000	NQ	32300	65
OKLMN-GW-PW7-0-050310 (C0064432 Spk E, 5000 ng/L Lab Spike)	5000	71600	78500	*	5000	37800	41900	*
OKLMN-GW-PW7-0-050310 (C0064432 Spk F, 50000 ng/L Lab Spike)	50000	71600	119000	95	50000	37600	86100	87
OKLMN-GW-PW7-0-050310 Low Spike 5 ppb (C0064434, 5000 ng/L Field Spike)	5000	71600	72800	*	5000	37800	42800	*
OKLMN-GW-PW7-0-050310 High Spike 50 ppb (C0064435, 50000 ng/L Field Spike)	50000	71600	118000	88	50000	37600	67100	59
OKLMN-GW-TRIP1-0-050310 Low Spike 5 ppb (C0064437, 5000 ng/L Field Spike)	5000	ND	5780	116	5000	ND	5360	107
OKLMN-GW-TRIP1-0-050310 High Spike 50 ppb (C0064438, 50000 ng/L Field Spike)	50000	ND	53500	107	50000	ND	50500	101
OKLMN-GW-PW8-0-050310 (C0064442 Spk G, 5000 ng/L Lab Spike)	5000	34300	38500	*	5000	61400	61000	*
OKLMN-GW-PW8-0-050310 (C0064442 Spk H, 50000 ng/L Lab Spike)	50000	34300	89800	111	50000	61400	113000	103
OKLMN-GW-PW8-0-050310 Low Spike 5 ppb (C0064444, 5000 ng/L Field Spike)	5000	34300	38400	*	5000	61400	58300	*
OKLMN-GW-PW8-0-050310 High Spike 50 ppb (C0064445, 50000 ng/L Field Spike)	50000	34300	83100	166	50000	61400	82200	42
OKLMN-GW-PW8-0-050310 (C0064446 Spk I, 5000 ng/L Lab Spike)	5000	84400	83200	*	5000	49000	50400	*
OKLMN-GW-PW8-0-050310 (C0064446 Spk J, 50000 ng/L Lab Spike)	50000	84400	138000	107	50000	49000	87900	98
OKLMN-GW-PW8-0-050310 Low Spike 5 ppb (C0064448, 5000 ng/L Field Spike)	5000	84400	86400	*	5000	49000	55200	*
OKLMN-GW-PW8-0-050310 High Spike 50 ppb (C0064449, 50000 ng/L Field Spike)	50000	84400	178000	183	50000	49000	83200	88
OKLMN-GW-W8102-0-050310 (C0064450 Spk K, 5000 ng/L Lab Spike)	5000	NQ	5300	106	5000	214	5880	113
OKLMN-GW-W8102-0-050310 (C0064450 Spk L, 50000 ng/L Lab Spike)	50000	NQ	55800	112	50000	214	52700	105
OKLMN-GW-W8102-0-050310 Low Spike 5 ppb (C0064453, 5000 ng/L Field Spike)	5000	NQ	5480	110	5000	214	5610	108
OKLMN-GW-W8102-0-050310 High Spike 50 ppb (C0064455, 50000 ng/L Field Spike)	50000	NQ	53200	106	50000	214	35300	70

*Sample residue exceeds the spiking level significantly, therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 26 ng/L (ppt)

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of

Quantitation (LOQ) which is 50 ng/L (ppt)

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

Table III. Matrix Spike Recovery of PFOS and PFOA in Water Samples Continued

Sample Description	C8 Sulfonate PFOS				C8 Acid PFOA			
	Amount Spiked (ng/L)	Amount Found In Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amount Found In Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-W8104-0-050310 (C0064454 Spk M, 5000 ng/L Lab Spike)	5000	109	5540	108	5000	207	5290	102
OKLMN-GW-W8104-0-050310 (C0064454 Spk N, 50000 ng/L Lab Spike)	50000	109	58100	116	50000	207	57300	114
OKLMN-GW-W8104-0-050310 Low Spike 5 ppb (C0064454, 5000 ng/L Field Spike)	5000	109	5700	112	5000	207	5470	105
OKLMN-GW-W8104-0-050310 High Spike 50 ppb (C0064457, 50000 ng/L Field Spike)	50000	109	56700	113	50000	207	35800	71
OKLMN-GW-W8201-0-050310 (C0064458 Spk C, 5000 ng/L Lab Spike)	5000	NQ	4980	100	5000	95.5	4950	97
OKLMN-GW-W8201-0-050310 (C0064458 Spk D, 50000 ng/L Lab Spike)	50000	NQ	50000	100	50000	95.5	50800	101
OKLMN-GW-W8201-0-050310 Low Spike 0.5 ppb (C0064454, 500 ng/L Field Spike)	500	NQ	670	134	500	95.5	608	102
OKLMN-GW-W8201-0-050310 High Spike 5 ppb (C0064451, 5000 ng/L Field Spike)	5000	NQ	6800	132	5000	95.5	6130	121
OKLMN-GW-W20-0-050309 (C0064467 Spk E, 5000 ng/L Lab Spike)	5000	46600	48900	*	5000	285000	289000	*
OKLMN-GW-W20-0-050309 (C0064467 Spk F, 50000 ng/L Lab Spike)	50000	46600	92800	92	50000	285000	295000	*
OKLMN-GW-W20-0-050309 Low Spike 5 ppb (C0064460, 5000 ng/L Field Spike)	5000	46600	41900	*	5000	285000	278000	*
OKLMN-GW-W20-0-050309 High Spike 50 ppb (C0064470, 50000 ng/L Field Spike)	50000	46600	69600	26	50000	285000	264000	*
OKLMN-GW-W22-0-050309 (C0064471 Spk G, 5000 ng/L Lab Spike)	5000	45800	44600	*	5000	317000	330000	*
OKLMN-GW-W22-0-050309 (C0064471 Spk H, 50000 ng/L Lab Spike)	50000	45800	98500	105	50000	317000	412000	*
OKLMN-GW-W22-0-050309 Low Spike 5 ppb (C0064473, 5000 ng/L Field Spike)	5000	45800	41200	*	5000	317000	324000	*
OKLMN-GW-W22-0-050309 High Spike 50 ppb (C0064474, 50000 ng/L Field Spike)	50000	45800	65800	40	50000	317000	298000	*
OKLMN-GW-W31-0-050309 (C0064475 Spk I, 5000 ng/L Lab Spike)	5000	132000	116000	*	5000	23100	26400	*
OKLMN-GW-W31-0-050309 (C0064475 Spk J, 50000 ng/L Lab Spike)	50000	132000	158000	72	50000	23100	71300	96
OKLMN-GW-W31-0-050309 High Spike 50 ppb (C0064477, 50000 ng/L Field Spike)	50000	132000	216000	168	50000	23100	65700	65
OKLMN-GW-W32-0-050309 (C0064478 Spk K, 5000 ng/L Lab Spike)	5000	47100	62200	*	5000	117000	109000	*
OKLMN-GW-W32-0-050309 (C0064478 Spk L, 50000 ng/L Lab Spike)	50000	47100	115000	138	50000	117000	151000	*
OKLMN-GW-W32-0-050309 Low Spike 5 ppb (C0064480, 5000 ng/L Field Spike)	5000	47100	74000	*	5000	117000	131000	*
OKLMN-GW-W32-0-050309 High Spike 50 ppb (C0064481, 50000 ng/L Field Spike)	50000	47100	120000	148	50000	117000	157000	*

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

NQ = Not detected at or above 25 ng/L (ppt)

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of Quantitation (LOQ) which is 50 ng/L (ppt)

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

Table III. Matrix Spike Recovery of PFOS and PFOA in Water Samples Continued

Sample Description	C8 Sulfonate PFOS				C8 Acid PFOA			
	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-W217-0-050309 (C0064432 Spk M, 5000 ng/L Lab Spike)	5000	NQ	4790	96	5000	NQ	4860	97
OKLMN-GW-W217-0-050309 (C0064432 Spk N, 50000 ng/L Lab Spike)	50000	NQ	49700	99	50000	NQ	52200	104
OKLMN-GW-W217-0-050309 Low Spike 5 ppb (C0064434, 5000 ng/L Field Spike)	5000	NQ	5800	116	5000	NQ	4880	98
OKLMN-GW-W217-0-050309 High Spike 50 ppb (C0064433, 50000 ng/L Field Spike)	50000	NQ	65300	131	50000	NQ	42800	85
OKLMN-GW-W2008-0-050309 (C0064436 Spk O, 5000 ng/L Lab Spike)	5000	15500	20800	106	5000	59300	64800	*
OKLMN-GW-W2008-0-050309 (C0064436 Spk P, 50000 ng/L Lab Spike)	50000	15500	69300	106	50000	59300	114000	109
OKLMN-GW-W2008-0-050309 Low Spike 5 ppb (C0064436, 5000 ng/L Field Spike)	5000	15500	27000	230	5000	59300	73600	*
OKLMN-GW-W2008-0-050309 High Spike 50 ppb (C0064436, 50000 ng/L Field Spike)	50000	15500	71300	112	50000	59300	94800	71
OKLMN-GW-W2012-0-050309 (C0064430 Spk C, 5000 ng/L Lab Spike)	5000	117000	143000	*	5000	46900	57700	*
OKLMN-GW-W2012-0-050309 (C0064430 Spk D, 50000 ng/L Lab Spike)	50000	117000	185000	138	50000	46900	94700	96
OKLMN-GW-W2012-0-050309 Low Spike 5 ppb (C0064430, 5000 ng/L Field Spike)	5000	117000	115000	*	5000	46900	48500	*
OKLMN-GW-W2012-0-050309 High Spike 80 ppb (C0064430, 50000 ng/L Field Spike)	50000	117000	203000	172	50000	46900	89500	85
OKLMN-GW-W80-0-050309 (C0064434 Spk E, 5000 ng/L Lab Spike)	5000	ND	5280	106	5000	302	5810	110
OKLMN-GW-W80-0-050309 (C0064434 Spk F, 50000 ng/L Lab Spike)	50000	ND	52900	106	50000	302	53100	108
OKLMN-GW-W80-0-050309 Low Spike 5 ppb (C0064434, 5000 ng/L Field Spike)	5000	ND	5720	114	5000	302	5860	111
OKLMN-GW-W80-0-050309 High Spike 50 ppb (C0064437, 50000 ng/L Field Spike)	50000	ND	57600	115	50000	302	42200	84
OKLMN-GW-PW1-0-050310 (C0064438 Spk G, 5000 ng/L Lab Spike)	5000	66900	72000	*	5000	37300	47700	*
OKLMN-GW-PW1-0-050310 (C0064438 Spk H, 50000 ng/L Lab Spike)	50000	66900	116000	102	50000	37300	90200	106
OKLMN-GW-PW1-0-050310 Low Spike 5 ppb (C0064380, 5000 ng/L Field Spike)	5000	66900	86900	*	5000	37300	40400	*
OKLMN-GW-PW1-0-050310 High Spike 50 ppb (C0064381, 50000 ng/L Field Spike)	50000	66900	85200	57	50000	37300	80500	86
OKLMN-GW-PW2-0-050310 (C0064382 Spk I, 5000 ng/L Lab Spike)	5000	351	5480	103	5000	3630	8720	102
OKLMN-GW-PW2-0-050310 (C0064382 Spk J, 50000 ng/L Lab Spike)	50000	351	59800	119	50000	3630	58800	110
OKLMN-GW-PW2-0-050310 Low Spike 5 ppb (C0064384, 5000 ng/L Field Spike)	5000	351	4280	79	5000	3630	7860	85
OKLMN-GW-PW2-0-050310 High Spike 50 ppb (C0064385, 50000 ng/L Field Spike)	50000	351	40500	80	50000	3630	39500	72

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 25 ng/L (ppt).

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of

Quantitation (LOQ) which is 50 ng/L (ppt).

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

Table III. Matrix Spike Recovery of PFOS and PFOA in Water Samples Continued

Sample Description	C8 Sulfonate PFOS				C8 Acid PFOA			
	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)
OKLMN-GW-PW3-0-050310 (C0064506 Spk K, 5000 ng/L Lab Spike)	5000	32700	43300	*	5000	177000	138000	*
OKLMN-GW-PW3-0-050310 (C0064506 Spk L, 50000 ng/L Lab Spike)	50000	32700	66100	67	50000	177000	180000	26
OKLMN-GW-PW3-0-050310 Low Spike 5 ppb (C0064506, 5000 ng/L Field Spike)	5000	32700	33800	*	5000	177000	186000	*
OKLMN-GW-PW3-0-050310 High Spike 50 ppb (C0064506, 50000 ng/L Field Spike)	50000	32700	90000	55	50000	177000	204000	54
OKLMN-GW-PW4-0-050310 (C0064510 Spk M, 5000 ng/L Lab Spike)	5000	33800	42500	*	5000	72100	77500	*
OKLMN-GW-PW4-0-050310 (C0064510 Spk N, 50000 ng/L Lab Spike)	50000	33800	75700	64	50000	72100	121000	96
OKLMN-GW-PW4-0-050310 Low Spike 5 ppb (C0064510, 5000 ng/L Field Spike)	5000	33800	42200	*	5000	72100	80700	*
OKLMN-GW-PW4-0-050310 High Spike 50 ppb (C0064510, 50000 ng/L Field Spike)	50000	33800	81200	65	50000	72100	118000	88
OKLMN-GW-PW6-0-050310 (C0064514 Spk O, 5000 ng/L Lab Spike)	5000	90000	97600	*	5000	78400	82900	*
OKLMN-GW-PW6-0-050310 (C0064514 Spk P, 50000 ng/L Lab Spike)	50000	90000	139000	98	50000	78400	133000	106
OKLMN-GW-PW6-0-050310 Low Spike 5 ppb (C0064514, 5000 ng/L Field Spike)	5000	90000	94100	*	5000	78400	87000	*
OKLMN-GW-PW6-0-050310 High Spike 50 ppb (C0064517, 50000 ng/L Field Spike)	50000	90000	124000	68	50000	78400	116000	75
OKLMN-GW-PW10-0-050310 (C0064518 Spk C, 5000 ng/L Lab Spike)	5000	1400000	1410000	*	5000	719000	868000	*
OKLMN-GW-PW10-0-050310 (C0064518 Spk D, 50000 ng/L Lab Spike)	50000	1400000	1390000	*	50000	719000	840000	*
OKLMN-GW-PW10-0-050310 Low Spike 5 ppb (C0064520, 5000 ng/L Field Spike)	5000	1400000	855000	*	5000	719000	692000	*
OKLMN-GW-PW10-0-050310 High Spike 50 ppb (C0064521, 50000 ng/L Field Spike)	50000	1400000	1910000	*	50000	719000	747000	*
OKLMN-GW-PW11-0-050310 (C0064522 Spk E, 5000 ng/L Lab Spike)	5000	40800	46900	*	5000	60300	64700	*
OKLMN-GW-PW11-0-050310 (C0064522 Spk F, 50000 ng/L Lab Spike)	50000	40800	83100	85	50000	60300	105000	89
OKLMN-GW-PW11-0-050310 Low Spike 5 ppb (C0064524, 5000 ng/L Field Spike)	5000	40800	45400	*	5000	60300	83300	*
OKLMN-GW-PW11-0-050310 High Spike 50 ppb (C0064525, 50000 ng/L Field Spike)	50000	40800	88500	95	50000	60300	98100	76

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 25 ng/L (ppt)

NQ = Not quantifiable = Measured concentration between 25 ng/L (ppt) and the Limit of Quantitation (LOQ) which is 50 ng/L (ppt)

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

**Table III. Matrix Spike Recovery of PFOS and PFOA in Water
Samples Continued**

Sample Description	Cs Sulfonate PFOS				Cs Acid PFOA				
	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	Amount Spiked (ng/L)	Amt Found in Sample (ng/L)	Amount Recovered (ng/L)	Recovery (%)	
OKLMN-SW-0-050310 (C0064626 Spk G, 5000 ng/L Lab Spike)	5000	1190	6270	102	5000	491	5520	101	
OKLMN-SW-0-050310 (C0064626 Spk H, 50000 ng/L Lab Spike)	50000	1190	51600	101	50000	491	53200	105	
OKLMN-SW-0-050310 Low Spike 5 ppb (C0064626, 5000 ng/L Field Spike)	5000	1190	7220	121	5000	491	8020	111	
OKLMN-SW-0-050310 High Spike 50 ppb (C0064626, 50000 ng/L Field Spike)	50000	1190	58000	114	50000	491	44200	87	
OKLMN-GW-TRIP2-0-050310 Low Spike 5 ppb (C0064631, 5000 ng/L Spike)	5000	ND	6170	123	5000	ND	6040	121	
OKLMN-GW-TRIP2-0-050310 High Spike 50 ppb (C0064632, 50000 ng/L Spike)	50000	ND	57400	115	50000	ND	57300	115	
Average:				110	Average:				93
Standard Deviation:				32	Standard Deviation:				19

Table IV. Surrogate Spike Recovery of ^{13}C PFOA in Water Samples

Exygen ID	Sample Description	^{13}C -PFOA		
		Amount Spiked (ng/L)	Amount Recovered (ng/L)	Recovery (%)
C0064404 Spk C	OKLMN-GW-W3-0-050310	5500	5580	101
C0064404 Spk D	OKLMN-GW-W3-0-050310	50500	53900	107
C0064404	OKLMN-GW-W3-0-050310	500	431	86
C0064404 Rep	OKLMN-GW-W3-0-050310	500	422	84
C0064405	OKLMN-GW-W3-0-050310 Dup	500	416	83
C0064406	OKLMN-GW-W3-0-050310 Low Spike 5 ppb	5000	4730	95
C0064407	OKLMN-GW-W3-0-050310 High Spike 50 ppb	50000	34500	69
C0064408 Spk E	OKLMN-GW-W8-0-050310	5500	5840	106
C0064408 Spk F	OKLMN-GW-W8-0-050310	50500	55900	111
C0064408	OKLMN-GW-W8-0-050310	500	466	93
C0064408 Rep	OKLMN-GW-W8-0-050310	500	452	90
C0064409	OKLMN-GW-W8-0-050310 Dup	500	483	97
C0064410	OKLMN-GW-W8-0-050310 Low Spike 5 ppb	5000	5550	111
C0064411	OKLMN-GW-W8-0-050310 High Spike 50 ppb	50000	39400	79
C0064412 Spk G	OKLMN-GW-W26-0-050310	5500	5430	99
C0064412 Spk H	OKLMN-GW-W26-0-050310	50500	32900	65
C0064412	OKLMN-GW-W26-0-050310	500	7720	*
C0064412 Rep	OKLMN-GW-W26-0-050310	500	7660	*
C0064413	OKLMN-GW-W26-0-050310 Dup	500	7540	*
C0064414	OKLMN-GW-W26-0-050310 Low Spike 5 ppb	5000	5210	104
C0064415	OKLMN-GW-W26-0-050310 High Spike 50 ppb	50000	28000	56
C0064416 Spk I	OKLMN-GW-W33-0-050310	5500	3160	57
C0064416 Spk J	OKLMN-GW-W33-0-050310	50500	48000	95
C0064416	OKLMN-GW-W33-0-050310	500	262	52
C0064416 Rep	OKLMN-GW-W33-0-050310	500	256	51
C0064417	OKLMN-GW-W33-0-050310 Dup	500	261	52
C0064418	OKLMN-GW-W33-0-050310 Low Spike 5 ppb	5000	2750	55
C0064419	OKLMN-GW-W33-0-050310 High Spike 50 ppb	50000	42500	85
C0064420 Spk K	OKLMN-GW-W205-0-050310	5500	4940	90
C0064420 Spk L	OKLMN-GW-W205-0-050310	50500	53000	105
C0064420	OKLMN-GW-W205-0-050310	500	279	56
C0064420 Rep	OKLMN-GW-W205-0-050310	500	280	56
C0064421	OKLMN-GW-W205-0-050310 Dup	500	307	61
C0064422	OKLMN-GW-W205-0-050310 Low Spike 5 ppb	5000	4240	85
C0064423	OKLMN-GW-W205-0-050310 High Spike 50 ppb	50000	38000	76
C0064424 Spk M	OKLMN-GW-W215-0-050310	5500	5850	106
C0064424 Spk N	OKLMN-GW-W215-0-050310	50500	56700	112
C0064424	OKLMN-GW-W215-0-050310	500	1010	202
C0064424 Rep	OKLMN-GW-W215-0-050310	500	434	87
C0064425	OKLMN-GW-W215-0-050310 Dup	500	464	93
C0064426	OKLMN-GW-W215-0-050310 Low Spike 5 ppb	5000	4860	99
C0064427	OKLMN-GW-W215-0-050310 High Spike 50 ppb	50000	35600	71

*The very high PFOA level in the undiluted sample interfered with ^{13}C PFOA recovery.

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

**Table IV. Surrogate Spike Recovery of ^{13}C PFOA in Water Samples
Continued**

Exygen ID	Sample Description	^{13}C -PFOA		
		Amount Spiked (ng/L)	Amount Recovered (ng/L)	Recovery (%)
C0064428 Spk C	OKLMN-GW-W2010-0-050310	5500	5390	98
C0064428 Spk D	OKLMN-GW-W2010-0-050310	50500	53000	105
C0064428	OKLMN-GW-W2010-0-050310	500	432	86
C0064428 Rep	OKLMN-GW-W2010-0-050310	500	417	83
C0064429	OKLMN-GW-W2010-0-050310 Dup	500	510	102
C0064430	OKLMN-GW-W2010-0-050310 Low Spike 5 ppb	5000	4500	90
C0064431	OKLMN-GW-W2010-0-050310 High Spike 50 ppb	50000	30200	60
C0064432 Spk E	OKLMN-GW-PW7-0-050310	5500	5260	96
C0064432 Spk F	OKLMN-GW-PW7-0-050310	50500	51000	101
C0064432	OKLMN-GW-PW7-0-050310	500	341	68
C0064432 Rep	OKLMN-GW-PW7-0-050310	500	335	67
C0064433	OKLMN-GW-PW7-0-050310 Dup	500	325	65
C0064434	OKLMN-GW-PW7-0-050310 Low Spike 5 ppb	5000	4820	96
C0064436	OKLMN-GW-PW7-0-050310 High Spike 50 ppb	50000	33200	66
C0064436	OKLMN-GW-TRIP1-0-050310	500	510	102
C0064437	OKLMN-GW-TRIP1-0-050310 Low Spike 5 ppb	5000	5100	102
C0064438	OKLMN-GW-TRIP1-0-050310 High Spike 50 ppb	50000	61300	103
C0064442 Spk G	OKLMN-GW-PW8-0-050310	5500	5270	96
C0064442 Spk H	OKLMN-GW-PW8-0-050310	50500	52900	105
C0064442	OKLMN-GW-PW8-0-050310	500	321	64
C0064442 Rep	OKLMN-GW-PW8-0-050310	500	322	64
C0064443	OKLMN-GW-PW8-0-050310 Dup	500	300	60
C0064444	OKLMN-GW-PW8-0-050310 Low Spike 5 ppb	5000	3910	78
C0064445	OKLMN-GW-PW8-0-050310 High Spike 50 ppb	50000	25800	52
C0064446 Spk I	OKLMN-GW-PW9-0-050310	5500	5250	95
C0064446 Spk J	OKLMN-GW-PW9-0-050310	50500	53900	107
C0064446	OKLMN-GW-PW9-0-050310	500	315	63
C0064446 Rep	OKLMN-GW-PW9-0-050310	500	317	63
C0064447	OKLMN-GW-PW9-0-050310 Dup	500	338	68
C0064448	OKLMN-GW-PW9-0-050310 Low Spike 5 ppb	5000	4230	85
C0064449	OKLMN-GW-PW9-0-050310 High Spike 50 ppb	50000	24800	50
C0064450 Spk K	OKLMN-GW-W6102-0-050310	5500	5640	103
C0064450 Spk L	OKLMN-GW-W6102-0-050310	50500	53200	105
C0064450	OKLMN-GW-W6102-0-050310	500	424	85
C0064450 Rep	OKLMN-GW-W6102-0-050310	500	439	88
C0064451	OKLMN-GW-W6102-0-050310 Dup	500	480	96
C0064452	OKLMN-GW-W6102-0-050310 Low Spike 5 ppb	5000	5050	101
C0064453	OKLMN-GW-W6102-0-050310 High Spike 50 ppb	50000	32500	65

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

Table IV. Surrogate Spike Recovery of ^{13}C PFOA in Water Samples
Continued

Exygen ID	Sample Description	^{13}C -PFOA		
		Amount Spiked (ng/L)	Amount Recovered (ng/L)	Recovery (%)
C0064454 Spk M	OKLMN-GW-W6104-0-050310	5500	5510	100
C0064454 Spk N	OKLMN-GW-W6104-0-050310	50500	54500	108
C0064454	OKLMN-GW-W6104-0-050310	500	398	80
C0064454 Rep	OKLMN-GW-W6104-0-050310	500	417	83
C0064455	OKLMN-GW-W6104-0-050310 Dup	500	487	97
C0064456	OKLMN-GW-W6104-0-050310 Low Spike 5 ppb	5000	4920	98
C0064457	OKLMN-GW-W6104-0-050310 High Spike 50 ppb	50000	33800	68
C0064458 Spk C	OKLMN-GW-W6201-0-050310	5500	5460	99
C0064458 Spk D	OKLMN-GW-W6201-0-050310	50500	50300	100
C0064458	OKLMN-GW-W6201-0-050310	500	388	78
C0064458 Rep	OKLMN-GW-W6201-0-050310	500	391	78
C0064459	OKLMN-GW-W6201-0-050310 Dup	500	486	99
C0064460	OKLMN-GW-W6201-0-050310 Low Spike 0.5 ppb	500	491	98
C0064461	OKLMN-GW-W6201-0-050310 High Spike 5 ppb	5000	5440	109
C0064467 Spk E	OKLMN-GW-W20-0-050309	5500	4090	74
C0064467 Spk F	OKLMN-GW-W20-0-050309	50500	46500	92
C0064467	OKLMN-GW-W20-0-050309	500	272	54
C0064467 Rep	OKLMN-GW-W20-0-050309	500	270	54
C0064468	OKLMN-GW-W20-0-050309 Dup	500	270	54
C0064469	OKLMN-GW-W20-0-050309 Low Spike 5 ppb	5000	3790	76
C0064470	OKLMN-GW-W20-0-050309 High Spike 50 ppb	50000	26800	53
C0064471 Spk G	OKLMN-GW-W22-0-050309	5500	4380	80
C0064471 Spk H	OKLMN-GW-W22-0-050309	50500	50700	100
C0064471	OKLMN-GW-W22-0-050309	500	267	53
C0064471 Rep	OKLMN-GW-W22-0-050309	500	280	56
C0064472	OKLMN-GW-W22-0-050309 Dup	500	256	51
C0064473	OKLMN-GW-W22-0-050309 Low Spike 5 ppb	5000	3830	77
C0064474	OKLMN-GW-W22-0-050309 High Spike 50 ppb	50000	28800	58
C0064475 Spk I	OKLMN-GW-W31-0-050309	5500	5410	98
C0064475 Spk J	OKLMN-GW-W31-0-050309	50500	54200	107
C0064475	OKLMN-GW-W31-0-050309	500	391	78
C0064475 Rep	OKLMN-GW-W31-0-050309	500	391	78
C0064476	OKLMN-GW-W31-0-050309 Dup	500	371	74
C0064477	OKLMN-GW-W31-0-050309 High Spike 50 ppb	50000	44100	88
C0064478 Spk K	OKLMN-GW-W32-0-050309	5500	4840	88
C0064478 Spk L	OKLMN-GW-W32-0-050309	50500	51000	101
C0064478	OKLMN-GW-W32-0-050309	500	287	57
C0064478 Rep	OKLMN-GW-W32-0-050309	500	289	58
C0064479	OKLMN-GW-W32-0-050309 Dup	500	328	66
C0064480	OKLMN-GW-W32-0-050309 Low Spike 5 ppb	5000	4750	95
C0064481	OKLMN-GW-W32-0-050309 High Spike 50 ppb	50000	49000	98

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

**Table IV. Surrogate Spike Recovery of ^{13}C PFOA in Water Samples
Continued**

Exygen ID	Sample Description	^{13}C -PFOA		
		Amount Spiked (ng/L)	Amount Recovered (ng/L)	Recovery (%)
C0064482 Spk M	OKLMN-GW-W217-0-050309	5500	5410	98
C0064482 Spk N	OKLMN-GW-W217-0-050309	50500	52500	104
C0064482	OKLMN-GW-W217-0-050309	500	428	86
C0064482 Rep	OKLMN-GW-W217-0-050309	500	453	91
C0064483	OKLMN-GW-W217-0-050309 Dup	500	468	94
C0064484	OKLMN-GW-W217-0-050309 Low Spike 5 ppb	5000	4850	97
C0064485	OKLMN-GW-W217-0-050309 High Spike 50 ppb	50000	44700	89
C0064486 Spk O	OKLMN-GW-W2008-0-050309	5500	5140	93
C0064486 Spk P	OKLMN-GW-W2008-0-050309	50500	52100	103
C0064486	OKLMN-GW-W2008-0-050309	500	318	64
C0064486 Rep	OKLMN-GW-W2008-0-050309	500	328	65
C0064487	OKLMN-GW-W2008-0-050309 Dup	500	357	71
C0064488	OKLMN-GW-W2008-0-050309 Low Spike 5 ppb	5000	6040	121
C0064489	OKLMN-GW-W2008-0-050309 High Spike 50 ppb	50000	37800	76
C0064490 Spk C	OKLMN-GW-W2012-0-050309	5500	5050	92
C0064490 Spk D	OKLMN-GW-W2012-0-050309	50500	50700	100
C0064490	OKLMN-GW-W2012-0-050309	500	344	69
C0064490 Rep	OKLMN-GW-W2012-0-050309	500	342	68
C0064491	OKLMN-GW-W2012-0-050309 Dup	500	351	70
C0064492	OKLMN-GW-W2012-0-050309 Low Spike 5 ppb	5000	5010	100
C0064493	OKLMN-GW-W2012-0-050309 High Spike 50 ppb	50000	37900	76
C0064494 Spk E	OKLMN-GW-W80-0-050309	5500	5650	103
C0064494 Spk F	OKLMN-GW-W80-0-050309	50500	52400	104
C0064494	OKLMN-GW-W80-0-050309	500	486	97
C0064494 Rep	OKLMN-GW-W80-0-050309	500	438	88
C0064495	OKLMN-GW-W80-0-050309 Dup	500	502	100
C0064496	OKLMN-GW-W80-0-050309 Low Spike 5 ppb	5000	5410	108
C0064497	OKLMN-GW-W80-0-050309 High Spike 50 ppb	50000	41700	83
C0064498 Spk G	OKLMN-GW-PW1-0-050310	5500	5150	94
C0064498 Spk H	OKLMN-GW-PW1-0-050310	50500	53000	105
C0064498	OKLMN-GW-PW1-0-050310	500	331	66
C0064498 Rep	OKLMN-GW-PW1-0-050310	500	350	70
C0064499	OKLMN-GW-PW1-0-050310 Dup	500	352	70
C0064500	OKLMN-GW-PW1-0-050310 Low Spike 5 ppb	5000	4420	88
C0064501	OKLMN-GW-PW1-0-050310 High Spike 50 ppb	50000	41600	83
C0064502 Spk I	OKLMN-GW-PW2-0-050310	5500	5420	99
C0064502 Spk J	OKLMN-GW-PW2-0-050310	50500	52600	104
C0064502	OKLMN-GW-PW2-0-050310	500	440	88
C0064502 Rep	OKLMN-GW-PW2-0-050310	500	440	88
C0064503	OKLMN-GW-PW2-0-050310 Dup	500	431	86
C0064504	OKLMN-GW-PW2-0-050310 Low Spike 5 ppb	5000	4680	94
C0064505	OKLMN-GW-PW2-0-050310 High Spike 50 ppb	50000	34000	68

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

**Table IV. Surrogate Spike Recovery of ^{13}C PFOA in Water Samples
Continued**

Exygen ID	Sample Description	^{13}C -PFOA		
		Amount Spiked (ng/L)	Amount Recovered (ng/L)	Recovery (%)
C0064506 Spk K	OKLMN-GW-PW3-0-050310	5500	4640	84
C0064506 Spk L	OKLMN-GW-PW3-0-050310	50500	53100	105
C0064506	OKLMN-GW-PW3-0-050310	500	284	57
C0064506 Rep	OKLMN-GW-PW3-0-050310	500	275	55
C0064507	OKLMN-GW-PW3-0-050310 Dup	500	294	59
C0064508	OKLMN-GW-PW3-0-050310 Low Spike 5 ppb	5000	4080	82
C0064509	OKLMN-GW-PW3-0-050310 High Spike 50 ppb	50000	38300	77
C0064510 Spk M	OKLMN-GW-PW4-0-050310	5500	5180	94
C0064510 Spk N	OKLMN-GW-PW4-0-050310	50500	52400	104
C0064510	OKLMN-GW-PW4-0-050310	500	320	64
C0064510 Rep	OKLMN-GW-PW4-0-050310	500	324	65
C0064511	OKLMN-GW-PW4-0-050310 Dup	500	313	63
C0064512	OKLMN-GW-PW4-0-050310 Low Spike 5 ppb	5000	4870	97
C0064513	OKLMN-GW-PW4-0-050310 High Spike 50 ppb	50000	38000	76
C0064514 Spk O	OKLMN-GW-PW6-0-050310	5500	5050	92
C0064514 Spk P	OKLMN-GW-PW6-0-050310	50500	52300	104
C0064514	OKLMN-GW-PW6-0-050310	500	304	61
C0064514 Rep	OKLMN-GW-PW6-0-050310	500	311	62
C0064515	OKLMN-GW-PW6-0-050310 Dup	500	313	63
C0064516	OKLMN-GW-PW6-0-050310 Low Spike 5 ppb	5000	4740	95
C0064517	OKLMN-GW-PW6-0-050310 High Spike 50 ppb	50000	35100	70
C0064518 Spk C	OKLMN-GW-PW10-0-050310	5500	3870	67
C0064518 Spk D	OKLMN-GW-PW10-0-050310	50500	48500	96
C0064518	OKLMN-GW-PW10-0-050310	500	255	51
C0064518 Rep	OKLMN-GW-PW10-0-050310	500	249	50
C0064519	OKLMN-GW-PW10-0-050310 Dup	500	258	52
C0064520	OKLMN-GW-PW10-0-050310 Low Spike 5 ppb	5000	2980	60
C0064521	OKLMN-GW-PW10-0-050310 High Spike 50 ppb	50000	34100	68
C0064522 Spk E	OKLMN-GW-PW11-0-050310	5500	5180	94
C0064522 Spk F	OKLMN-GW-PW11-0-050310	50500	51600	102
C0064522	OKLMN-GW-PW11-0-050310	500	319	64
C0064522 Rep	OKLMN-GW-PW11-0-050310	500	347	69
C0064523	OKLMN-GW-PW11-0-050310 Dup	500	312	62
C0064524	OKLMN-GW-PW11-0-050310 Low Spike 5 ppb	5000	4400	88
C0064525	OKLMN-GW-PW11-0-050310 High Spike 50 ppb	50000	36000	72
C0064526 Spk G	OKLMN-SW-0-050310	5500	5270	96
C0064526 Spk H	OKLMN-SW-0-050310	50500	52000	103
C0064526	OKLMN-SW-0-050310	500	124	25
C0064526 Rep	OKLMN-SW-0-050310	500	132	26
C0064527	OKLMN-SW-0-050310 Dup	500	505	101
C0064528	OKLMN-SW-0-050310 Low Spike 5 ppb	5000	5320	106
C0064529	OKLMN-SW-0-050310 High Spike 50 ppb	50000	41200	82

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

**Table IV. Surrogate Spike Recovery of ^{13}C PFOA in Water Samples
Continued**

Exygen ID	Sample Description	^{13}C -PFOA		
		Amount Spiked (ng/L)	Amount Recovered (ng/L)	Recovery (%)
C0064530	OKLMN-GW-TRIP2-0-050310	500	494	99
C0064531	OKLMN-GW-TRIP2-0-050310 Low Spike 5 ppb	5000	5520	110
C0064532	OKLMN-GW-TRIP2-0-050310 High Spike 50 ppb	50000	52900	106
Average:				83
Standard Deviation:				21

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

FIGURES

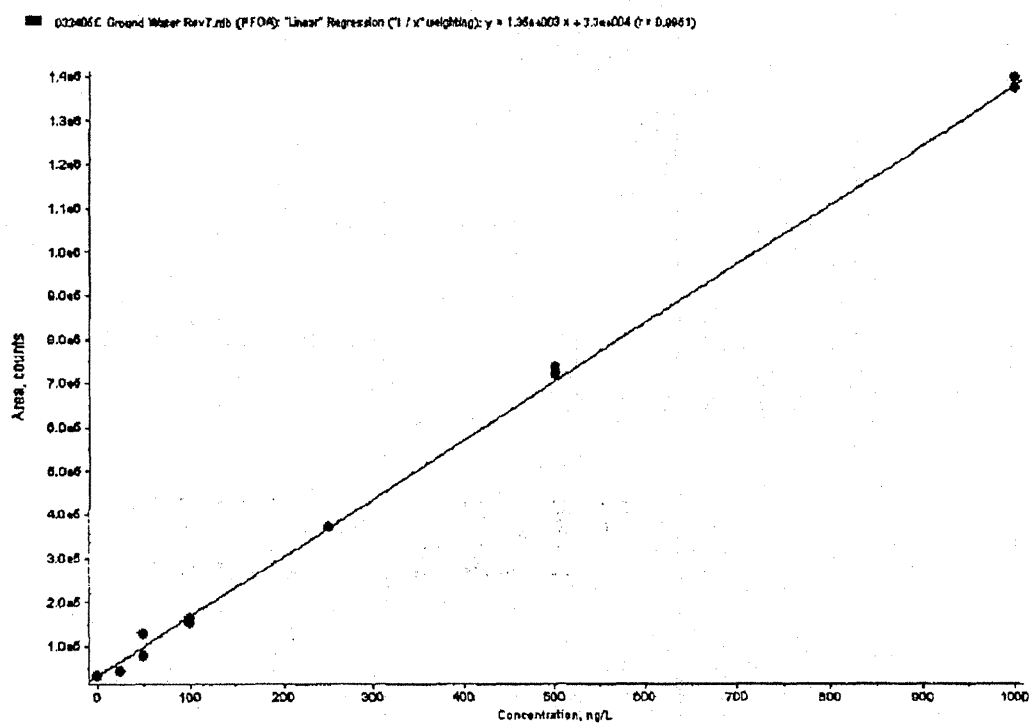
Figure 1. Typical Calibration Curve for PFOA in Reagent Water

Figure 2. Extracted Standards of PFOA in Reagent Water, 25 ng/L and 50 ng/L, Respectively

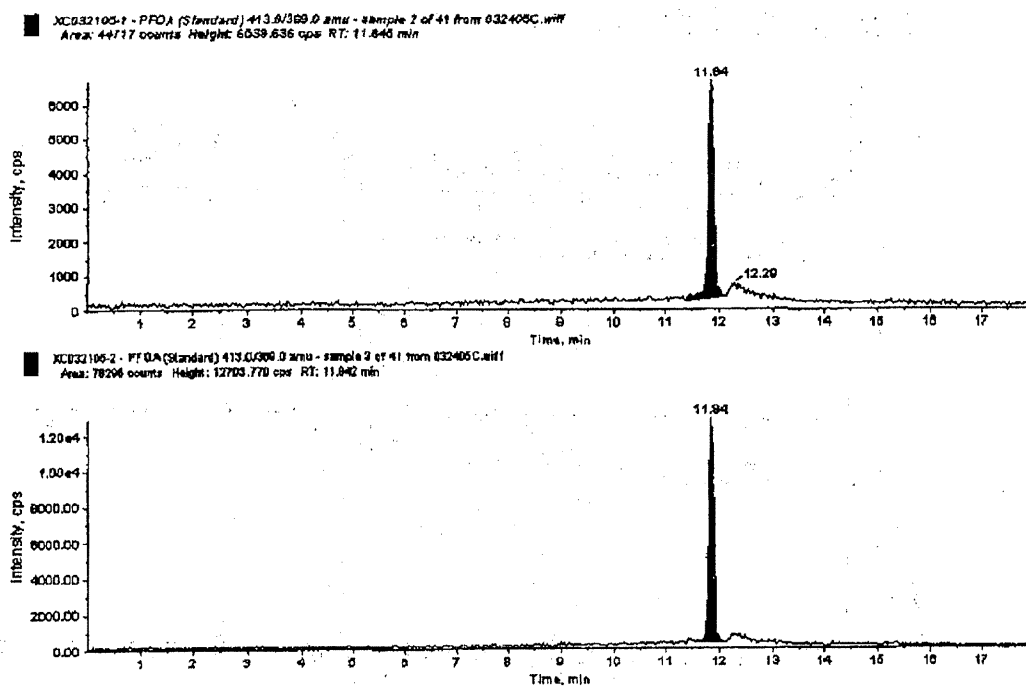
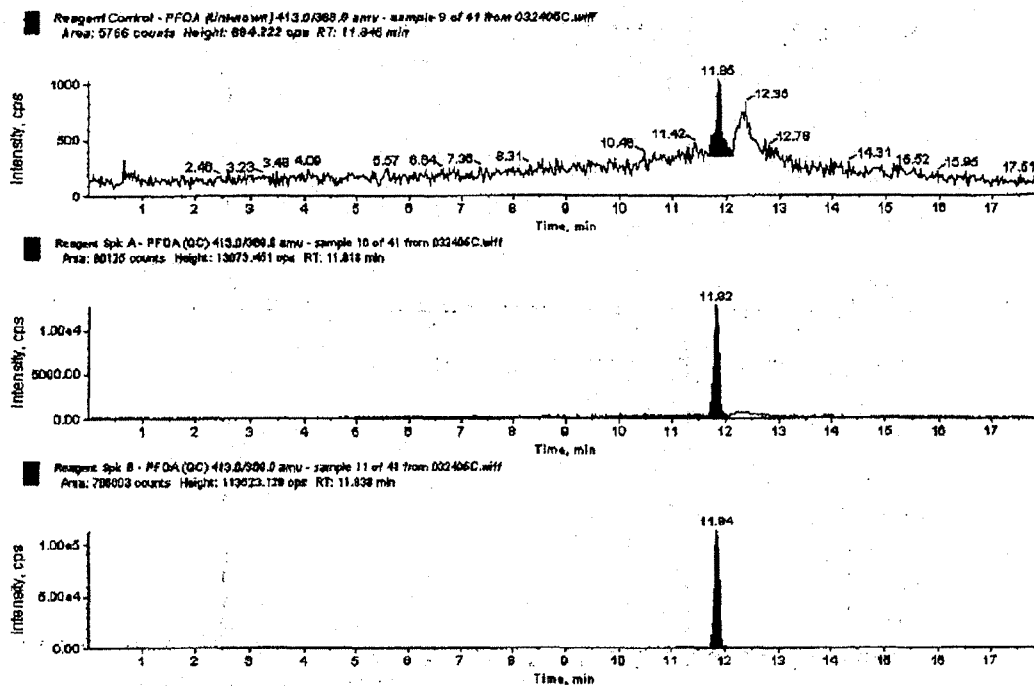


Figure 3. PFOA in Reagent Water, 50 ng/L Fortified Reagent Water, and 500 ng/L Fortified Reagent Water, Respectively



Note: A linear lot of PFOA was used to calibrate the instrument. The two peaks shown in Figure 4 are due to different isomers present in environmental samples. The area counts for all isomers present are included to report one total PFOA value.

Figure 4. Chromatogram Representing an Oakdale Water Sample Analyzed for PFOA, DF=100 (Exygen ID: C0064522, Data Set: 032405CR)

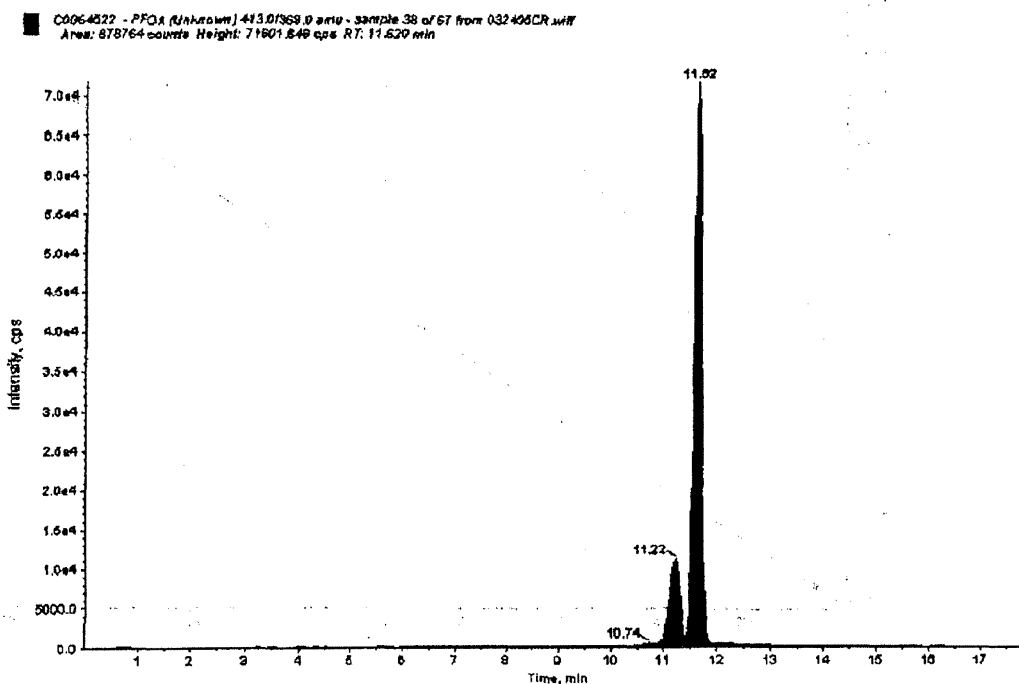


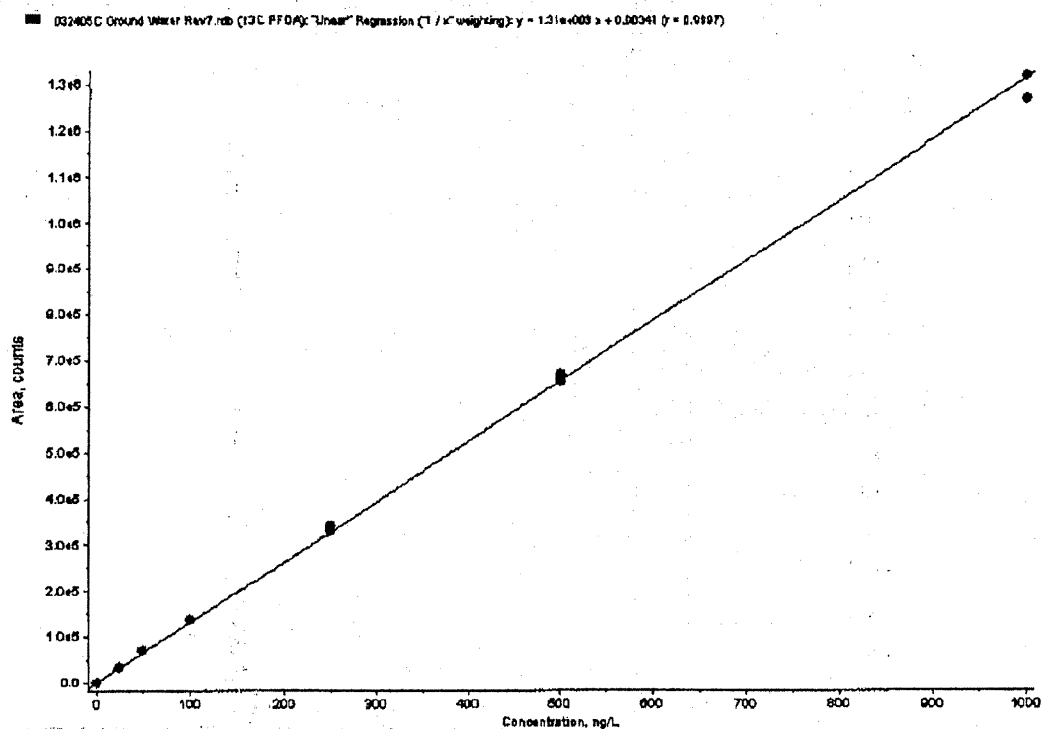
Figure 5. Typical Calibration Curve for ^{13}C PFOA in Reagent Water

Figure 6. Extracted Standards of ^{13}C PFOA in Reagent Water, 25 ng/L and 50 ng/L, Respectively

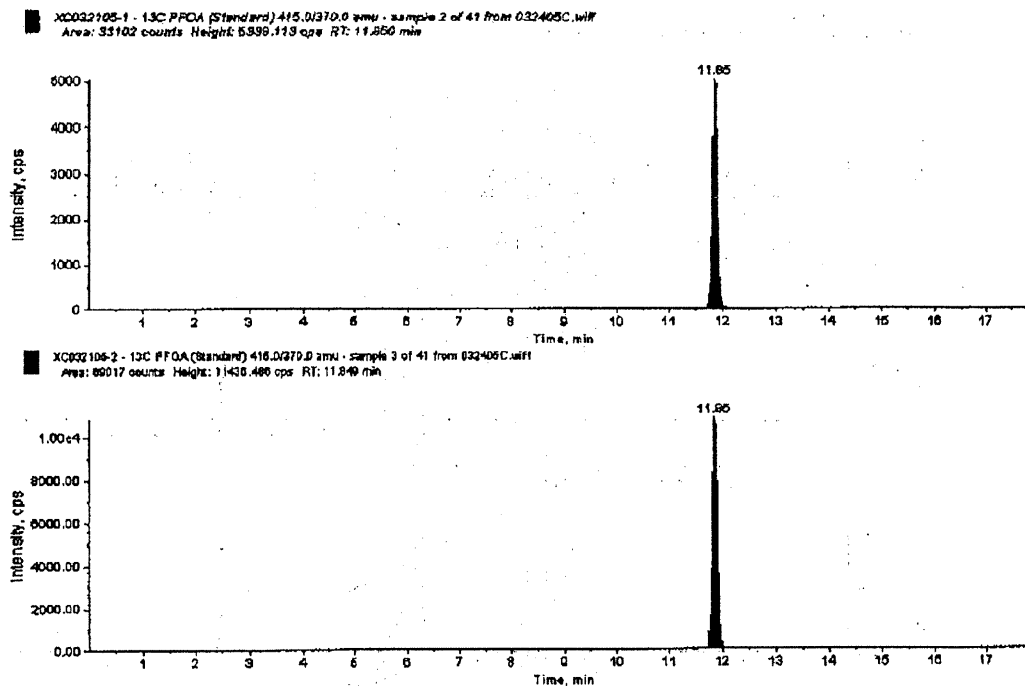


Figure 7. ^{13}C PFOA in Reagent Water, 50 ng/L Fortified Reagent Water, and 500 ng/L Fortified Reagent Water, Respectively

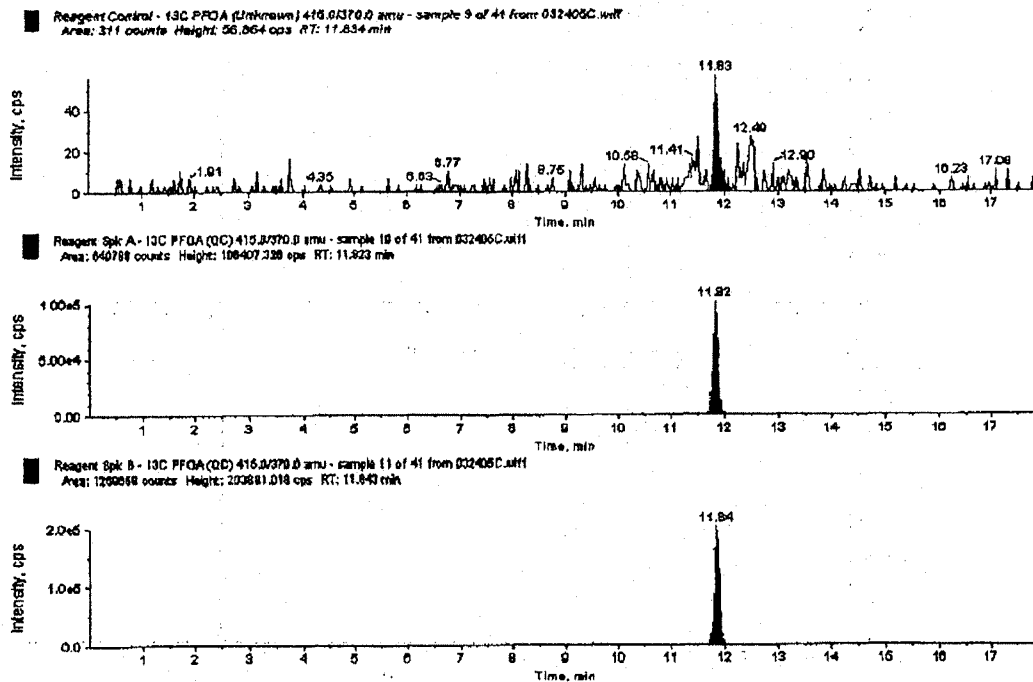


Figure 8. Chromatogram Representing an Oakdale Water Sample Analyzed for ^{13}C PFOA (Exygen ID: C0064522, Data Set: 032405C)

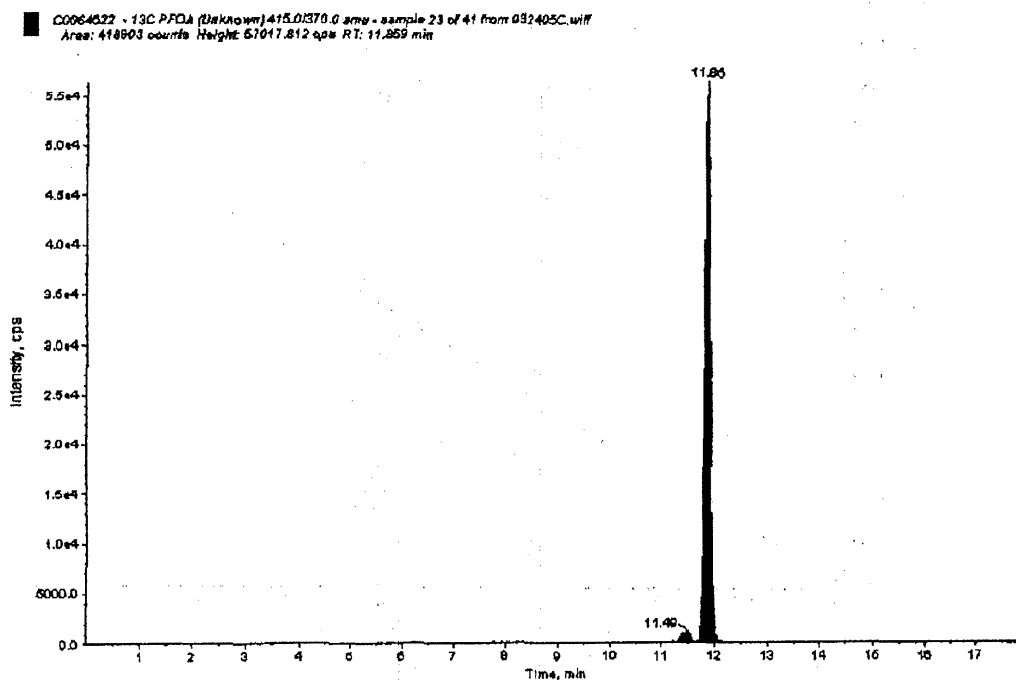


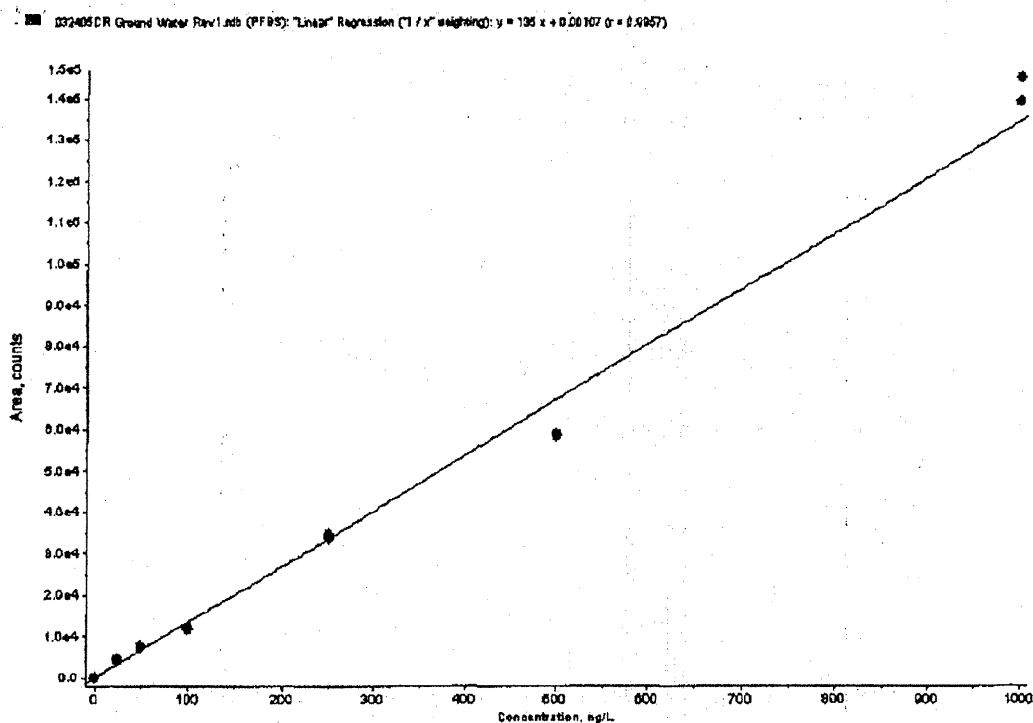
Figure 9. Typical Calibration Curve for PFBS in Reagent Water

Figure 10. Extracted Standards of PFBS in Reagent Water, 25 ng/L and 50 ng/L, Respectively

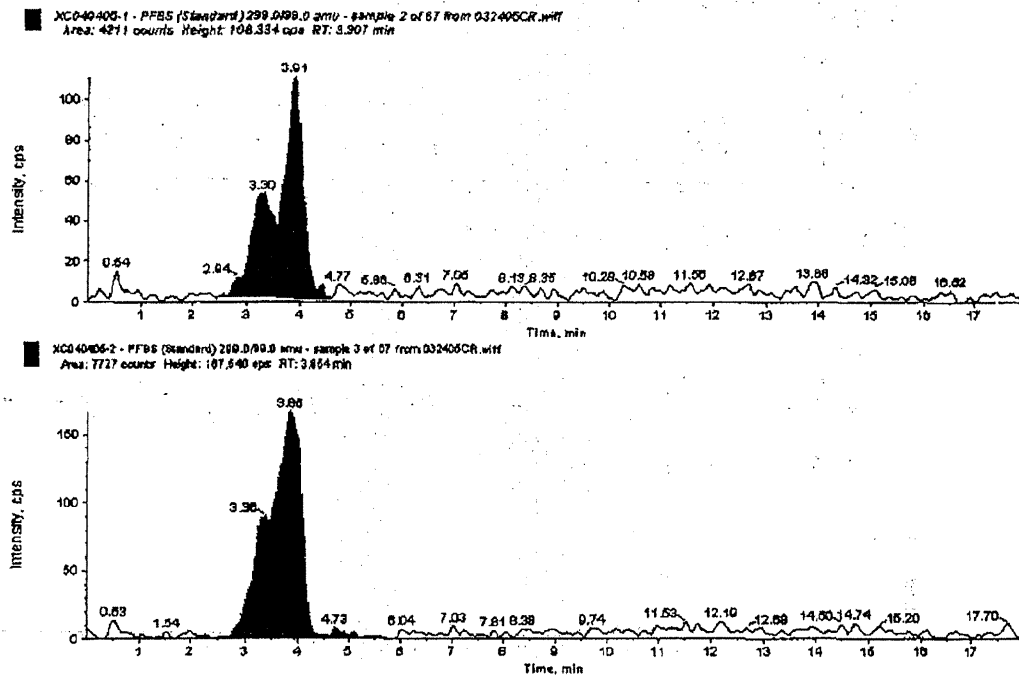


Figure 11. PFBS in Reagent Water, 50 ng/L Fortified Reagent Water, and 500 ng/L Fortified Reagent Water, Respectively

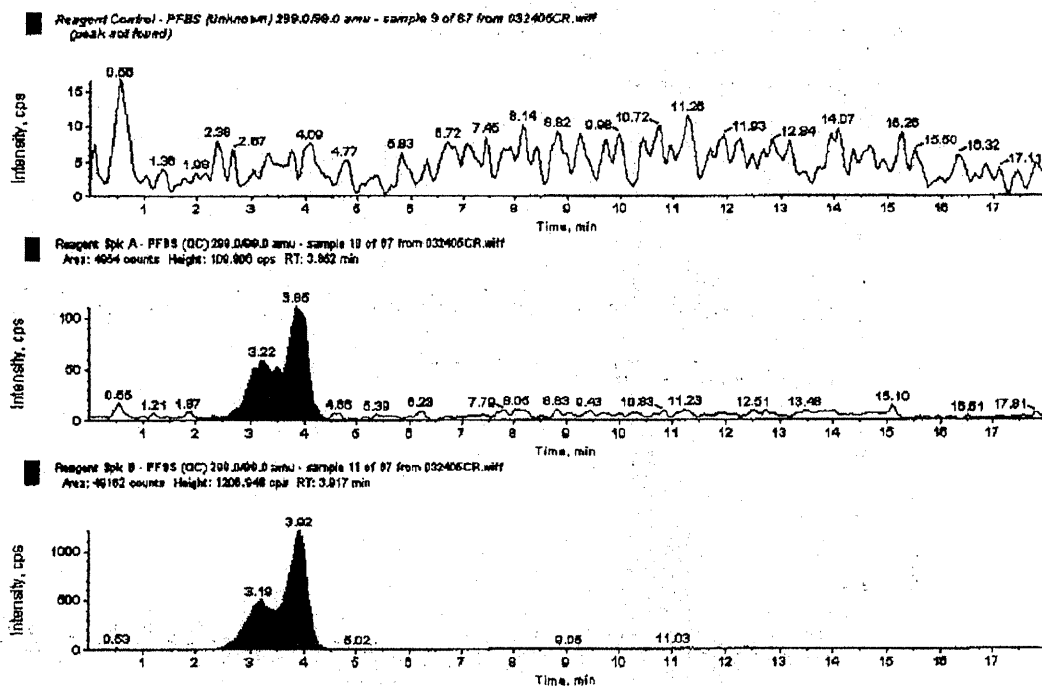


Figure 12. Chromatogram Representing an Oakdale Water Sample Analyzed for PFBS, DF=100 (Exygen ID: C0064522, Data Set: 032405CR)

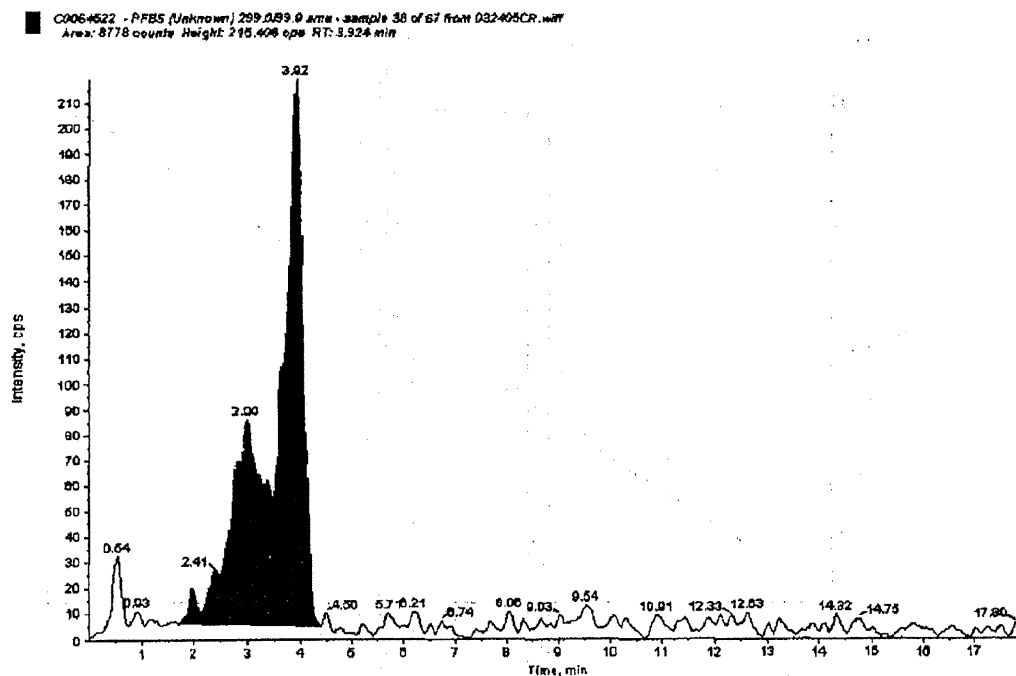


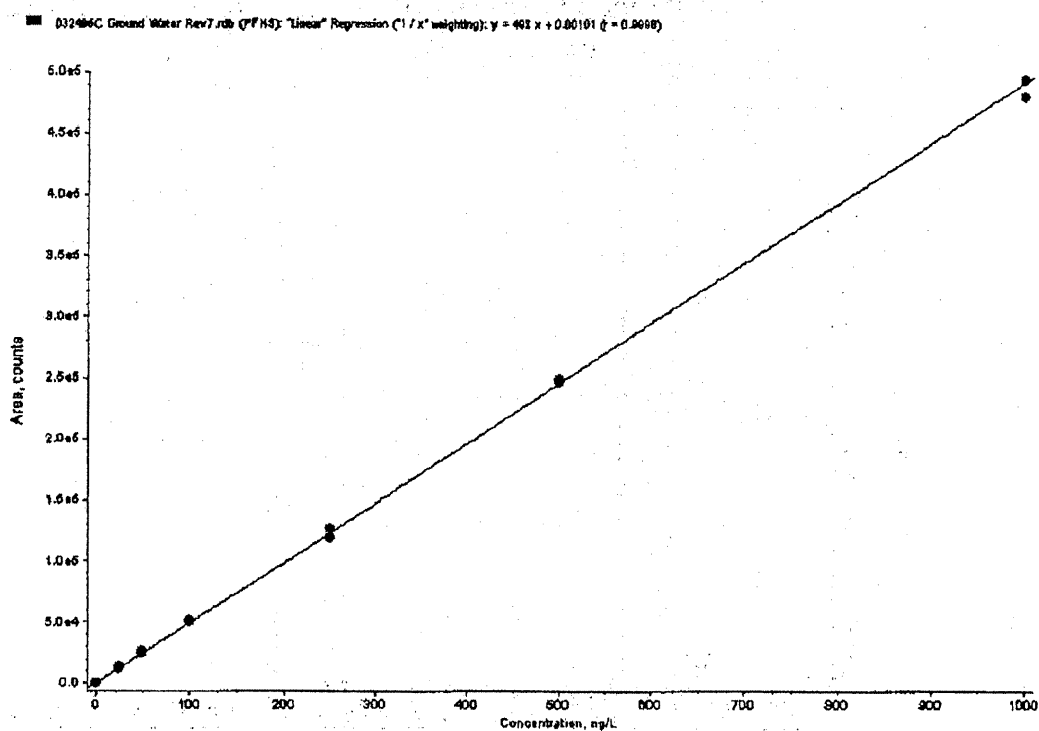
Figure 13. Typical Calibration Curve for PFHS in Reagent Water

Figure 14. Extracted Standards of PFHS in Reagent Water, 25 ng/L and 50 ng/L, Respectively

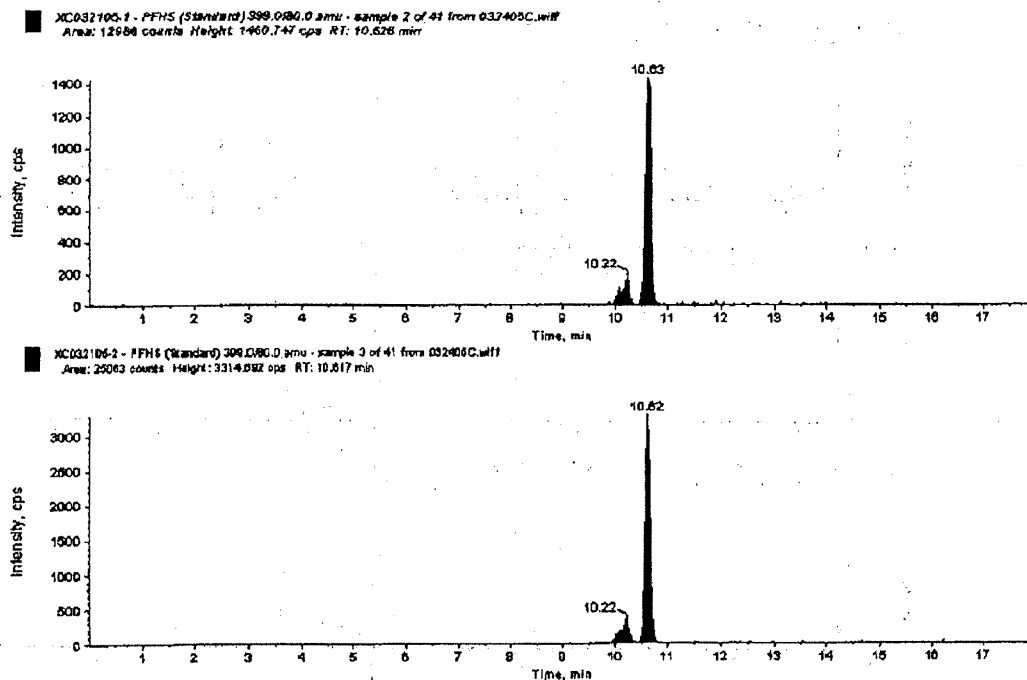


Figure 15. PFHS in Reagent Water, 50 ng/L Fortified Reagent Water, and 500 ng/L Fortified Reagent Water, Respectively

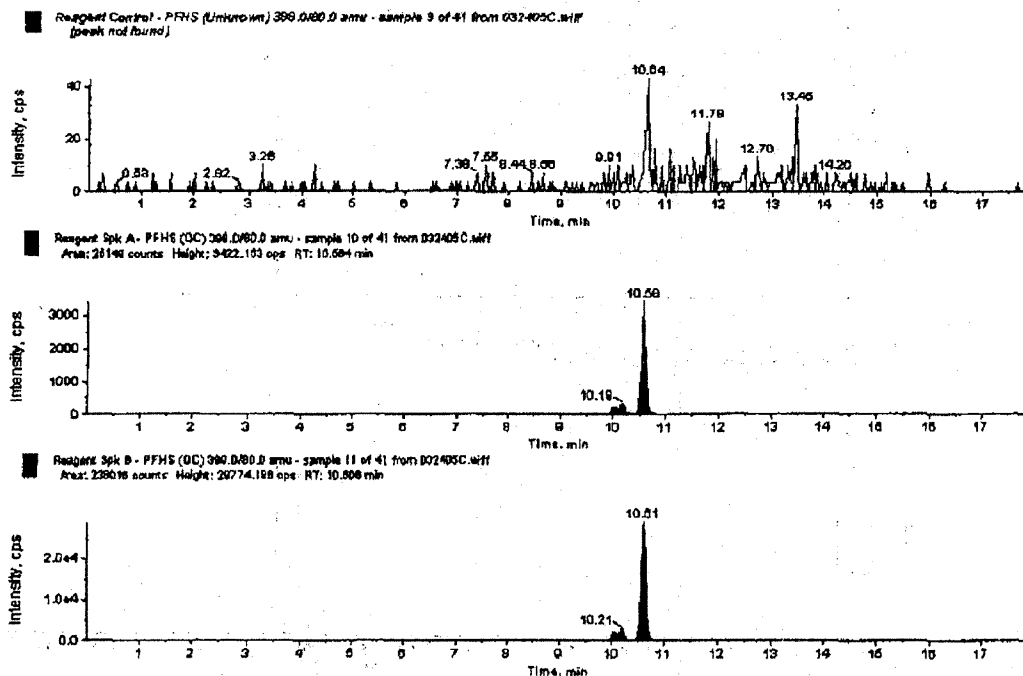


Figure 16. Chromatogram Representing an Oakdale Water Sample Analyzed for PFHS, DF=100 (Exygen ID: C0064522, Data Set: 032405CR)

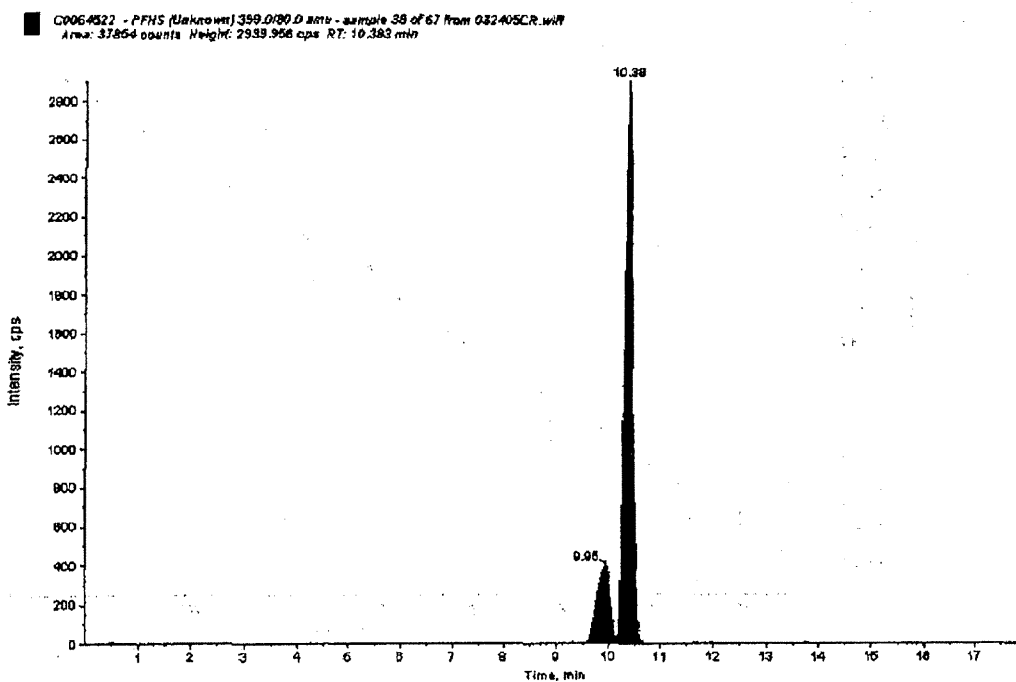


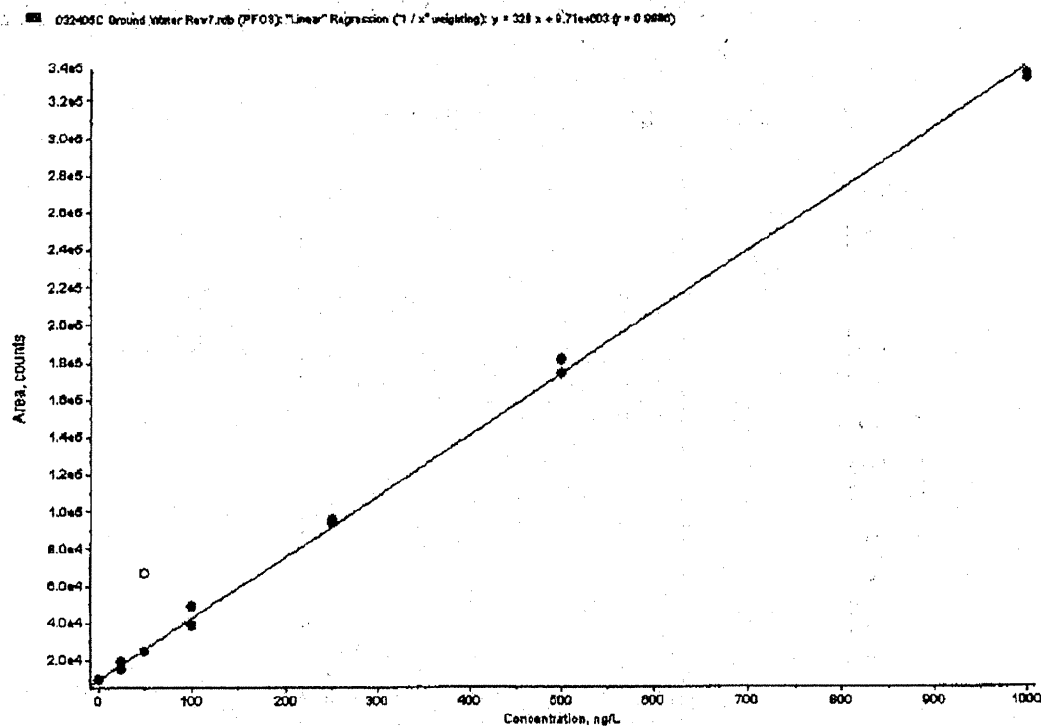
Figure 17. Typical Calibration Curve for PFOS in Reagent Water

Figure 18. Extracted Standards of PFOS in Reagent Water, 25 ng/L and 50 ng/L, Respectively

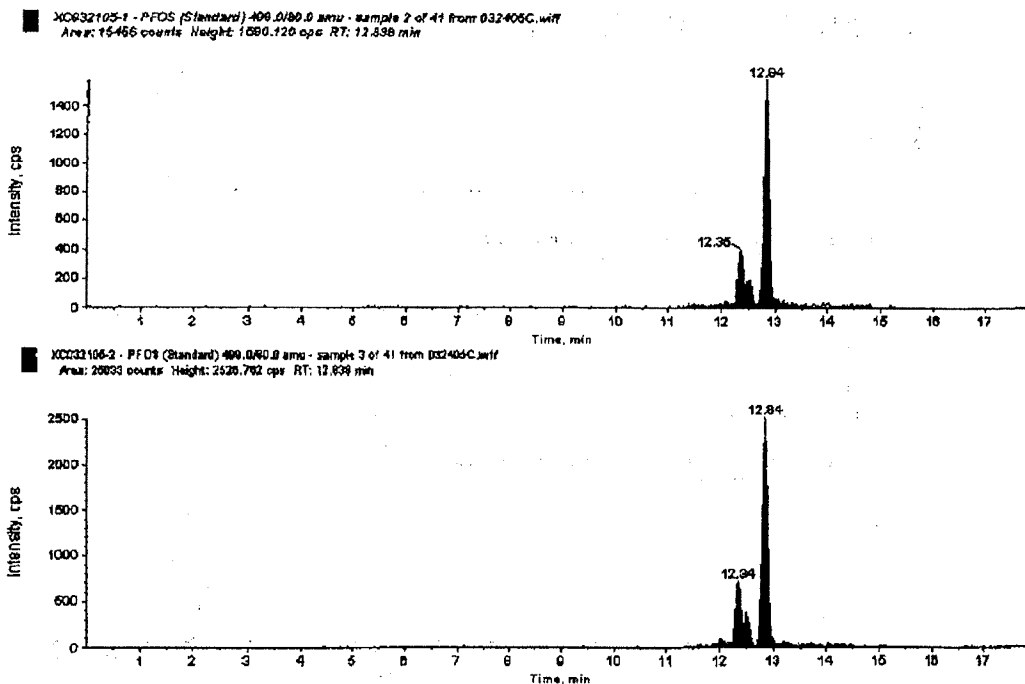


Figure 19. PFOS in Reagent Water, 50 ng/L Fortified Reagent Water, and 500 ng/L Fortified Reagent Water, Respectively

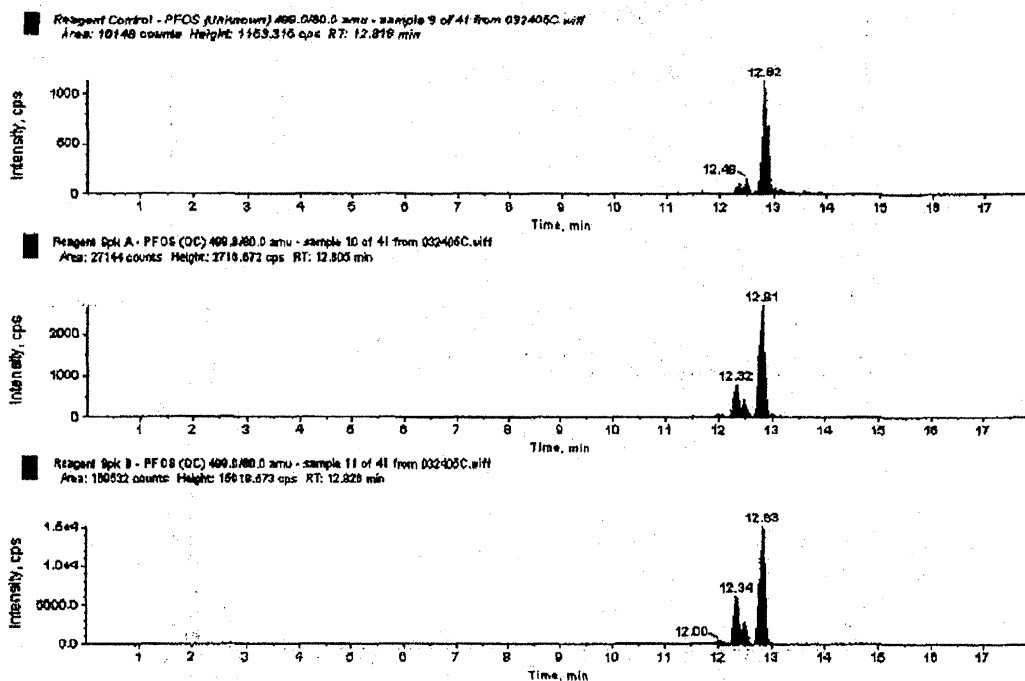
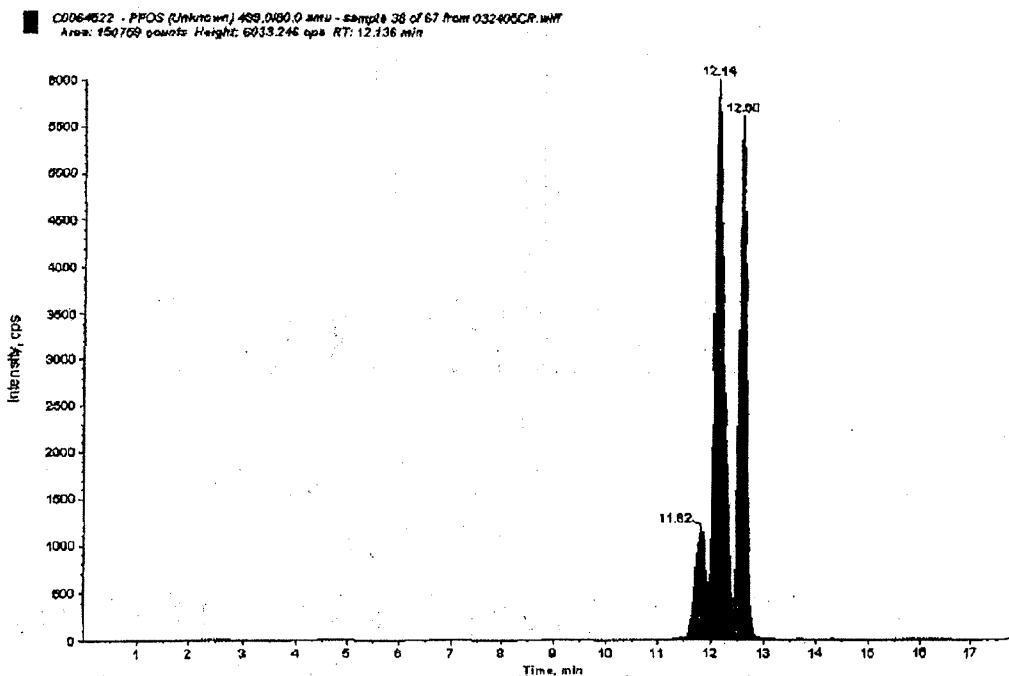


Figure 20. Chromatogram Representing an Oakdale Water Sample Analyzed for PFOS, DF=100 (Exygen ID: C0064522, Data Set: 032405CR)



APPENDIX A

Study Protocol

P0001400

**(Exygen Study No. P0001400) with
Analytical Method: V0001780: "Method of
Analysis for the Determination of
Perfluorooctanoic Acid (PFOA) in Water
by LC/MS/MS"**