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**INTERIM REPORT 3 – ANALYSIS OF FISH AND CLAM SAMPLES**

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**INTERIM REPORT #3 – Analysis of Fish and Clam Samples**

**STUDY TITLE**

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

**DATA REQUIREMENTS**

EPA TSCA Good Laboratory Practice Standards 40 CFR 792

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**INTERIM REPORT COMPLETION DATE**

June 17, 2008

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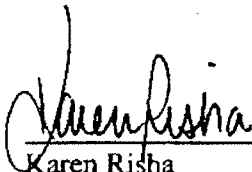
**PROJECT**

MPI Research Study Number: 0137.0219  
MPI Project Number: P0003267

Total Pages: 151

**GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT**

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



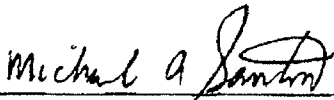
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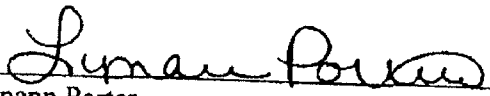
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**QUALITY ASSURANCE STATEMENT**

MPI Research's Quality Assurance Unit reviewed MPI Project Number P0003267, entitled, "Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, and Clams Using LC/MS/MS for the 3M Decatur Monitoring Program". All reviewed phases<sup>1</sup> were inspected for conduct according to MPI Research's Standard Operating Procedures, the Study Protocol, the Study Method, and all applicable Good Laboratory Practice Standards. All findings were reported to the MPI Principal Investigator, Management and to the Study Director.

<u>Phase</u>	<u>Date Inspected</u>	<u>Date Reported to Principal Investigator</u>	<u>Date Reported to MPI Management</u>	<u>Date Reported to Study Director</u>
Raw Data, Protocol Amendments 5-8, and Draft Report Review	05/27-29/08	05/29/08	05/29/08	05/29/08
Final Interim Report Review	06/04/08	06/04/08	06/04/08	06/04/08

  
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 Quality Assurance Research Group Leader, Quality Assurance Unit

6-17-08  
 Date

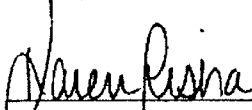
<sup>1</sup>Note: All in-lab inspections and the protocol review will be documented in the QA statement for the final analytical report at the conclusion of the study. This QA statement involves only the review of the interim report and associated raw data.

**CERTIFICATION OF AUTHENTICITY**

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

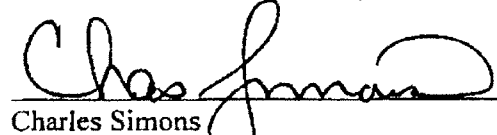
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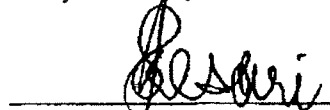
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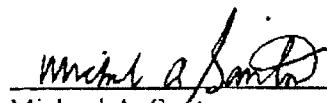
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**STUDY IDENTIFICATION**

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

MPI PROJECT NUMBER: P0003267

MPI STUDY NUMBER: 0137.0219

TYPE OF STUDY: Residue

SAMPLE MATRIX: Fish and Clam

TEST SUBSTANCES: Perfluorooctanoic Acid (PFOA)

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ANALYTICAL PHASE  
TIMETABLE: Study Initiation Date: 06/14/07  
Interim Analytical Start Date: 12/27/07  
Interim Analytical Termination Date: 05/12/08  
Interim Report Completion Date: 06/17/08

**PROJECT PERSONNEL**

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

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## 1.0 SUMMARY

MPI Research, Inc. successfully extracted and analyzed fish and clam samples for the determination of perfluorooctanoic acid (PFOA) according to 3M Environmental Laboratory Method ETS-8-049.2 (V0003970) detailed in MPI Protocol P0003267 Amendments #3 through #8 (Appendix A, pgs. 124-151).

The limits of quantitation (LOQ) for the analyte in the fish fillet samples are listed in Table I. The target LOQ for the method for fish fillet samples was 0.20 ng/g. The limits of quantitation (LOQ) for the analyte in the whole body fish samples are listed in Table III. The target LOQ for the method for whole body fish samples was 0.20 ng/g. The limits of quantitation (LOQ) for the analyte in the clam samples are listed in Tables V. The target LOQ for the method for clam samples was 0.20 ng/g. After evaluation of the reagent blanks (method blanks) used for the analysis, the LOQ was determined. There were no instances where a raised LOQ was required. A discussion of the process used to evaluate the reagent blanks can be found in section 6.3 of the report. The LOQ for the analyte in the re-extracted whole body fish sample is listed in Table IV. The target LOQ for the method for the re-extracted whole body fish sample was 0.20 ng/g.

Analytical results and assessed accuracies for the analysis of PFOA found in the fish fillet samples are summarized in Table I. Fortification recoveries for PFOA in the fish fillet samples are detailed in Table VI. The average percent recovery  $\pm$  standard deviation for PFOA in the fish fillet samples was  $105 \pm 23\%$ . The results for one fish fillet sample were not reported (NR) due to low spike recovery outside of the acceptance criteria range. The sample was re-extracted and reanalyzed (Table II), but matrix spike recovery of the re-extracted sample (Table VII) was also outside of the acceptance criteria range. Analytical results and assessed accuracies for the analysis of PFOA found in the whole body fish samples are summarized in Table III. Fortification recoveries for PFOA in the whole body fish samples are detailed in Table VIII. The average percent recovery  $\pm$  standard deviation for PFOA in the whole body fish samples was  $99 \pm 20\%$ . The results for one fish fillet sample were not reported (NR) due to low spike recovery outside of the acceptance criteria range. The sample was re-extracted and reanalyzed, and the analytical results and assessed accuracies for the analysis of PFOA found in the re-extracted whole body fish sample are summarized in Table IV. The fortification recovery for PFOA in the re-extracted whole body fish sample is detailed in Table VIII. The percent recovery for PFOA in the re-extracted whole body fish sample was 89%. Analytical results and assessed accuracies for the analysis of PFOA found in the clam samples are summarized in Table V. Fortification recoveries for PFOA in the clam samples are detailed in Table X. The average percent recovery  $\pm$  standard deviation for PFOA in the clam samples was  $111 \pm 19\%$ .

The assessed accuracy for the majority of the samples reported is  $\pm 30\%$ . The accuracies were assessed for each sample by reviewing the matrix spike whose spiking level most closely matches the endogenous concentration found in the sample.

## **2.0 OBJECTIVE**

The objective of the analytical part of this study was to determine levels of perfluorooctanoic acid (PFOA) in fish and clam samples according to Protocol P0003267 and Amendments #3 through #8 (Appendix A).

## **3.0 INTRODUCTION**

This report details the results of the analysis for the determination of PFOA in fish and clams using the 3M Environmental Laboratory analytical method ETS-8-049.2 (V0003970) entitled, "Determination of Fluorochemicals via Solid-Phase Extraction of Fish Tissues (Fillet or Whole Body) by High Performance Liquid Chromatography with Tandem Mass Spectrometry" detailed in MPI Protocol P0003267 Amendments #3 through #8.

The study was initiated on June 14, 2007, when the study director signed protocol number P0003267. The analytical start date for this interim report was December 27, 2007, and the analytical termination date for this interim report was May 12, 2008.

## **4.0 ANALYTICAL TEST SAMPLES**

A total of one hundred and eighteen samples (ExyLIMS ID C0227544, C0227546 – C0227656, and C0227658 – C0227663 from login ID L00010436), one hundred and twelve fish and six clams, were received on dry ice on December 20, 2006 from Charles Young at Weston Solutions, Inc. The one hundred and twelve fish samples represented six sample sites: DL1, DL2, DL3, DOU, DMC, and DBC. Each site consisted of twenty samples, with the exception of site DL2 which consisted of twelve samples. The twenty samples represented ten catfish samples (five catfish fillets and five whole body catfish samples) and ten bass samples (five bass fillets and five whole body bass samples). For the DL2 site, the twelve samples represented ten catfish samples (five catfish fillets and five whole body catfish samples) and two bass samples (two whole body bass samples). All samples were logged in by MPI personnel and placed in frozen storage.

The sample IDs assigned by the client follow this formula:  
Dxx-x02-xxxxxx-x(x)-0612xx.

The first string begins with D for Decatur, Alabama and the xx defines the sampling area where:

DL3 = Up river Letter of Intent (LOI) sampling location LOC-3

DL2 = Cross river LOI sampling location LOC-2

DL1 = Down river Fox Creek mouth LOI sampling location LOC-1

DBC = Dicks Creek mouth  
 DOU = Outlet at 3M WWTP outfall  
 DMC = Downstream Mallard Creek mouth

The second string defines the general category of the biota sample and the sampling round:

F02 = Fish sample second round  
 I02 = Invertebrate sample second round

The third string defines the species (first two characters), the sample type (third character) and the sample number (last three characters) within that sampling area and type of sample:

IP = channel catfish (*Ictalurus punctatus*)  
 MS = largemouth bass (*Micropterus salmoides*)  
 CF = American clam (*Corbicula fluminea*)

F = Fillet tissue sample  
 W = Whole body sample

001 through 1005 = sample number within the sampling area and sample type

The fourth string additionally defines the sample type where:

0 = Primary field sample  
 1 = Field duplicate sample  
 RB = Equipment rinsewater blank sample

The fifth string defines the sample preparation date in YYMMDD format.

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

## 5.0 REFERENCE MATERIAL

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

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

The molecular structure of the standard is given below:

**PFOA**

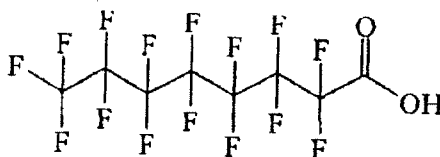
Chemical Name: Perfluorooctanoic acid

Molecular Weight: 414

Transitions Monitored: 413 → 369

413 → 219

Structure:



## **6.0 DESCRIPTION OF ANALYTICAL METHOD**

The 3M Environmental Laboratory analytical method ETS-8-049.2 (V0003970) entitled, "Determination of Fluorochemicals via Solid-Phase Extraction of Fish Tissues (Fillet or Whole Body) by High Performance Liquid Chromatography with Tandem Mass Spectrometry" and details in Amendments #3 through #8 (Appendix A, pgs. 124-151) were used for the sample analysis in this study.

### **6.1 Extraction Procedure for Fish and Clams**

#### *6.1.1 Sample Preparation*

Before the samples could be weighed for the extraction, they had to be processed. To process, the frozen samples were placed into a food processor and homogenized with dry ice. The samples were transferred to one-gallon Ziploc bags and placed in frozen storage with bag left open to allow the dry ice to sublime. After sublimation, the sample bags were sealed and remained in frozen storage until time of analysis. Sample processing records are located in the Sample Information section of the raw data package except for those that were processed and supplied by the study sponsor as outlined in the protocol deviation found in the raw data package.

#### *6.1.2 Sample Extraction*

A 3-gram portion of each sample was weighed into a graduated cylinder, and the samples were brought up to 30 mL with acetonitrile. Each sample



was then homogenized for ~2 minutes. 5-mL aliquots of each sample were transferred to 15-mL centrifuge tubes, fortified as appropriate, and vortex mixed. The samples were placed into a freezer for ~1 hour, and then centrifuged for ~20 minutes at ~3000 rpm at -5°C. The supernatant was decanted into 100 mL of 2% H<sub>3</sub>PO<sub>4</sub> (aq) and vortexed. Next, the samples (catfish fillets, bass fillets, catfish whole bodies and clams) were loaded onto preconditioned HLB SPE cartridges (conditioned with 2 mL of methanol followed by 2 mL of water) and drawn through under vacuum, discarding the eluent. The whole body bass samples were loaded onto preconditioned tC18 SPE cartridges (conditioned with 5 mL of methanol followed by 5 mL of water) and drawn through under vacuum, discarding the eluent. After the samples were drawn through the cartridges, the cartridges were washed with 2 mL of 5% methanol (aq) (for the fillet samples) followed by 2 mL of 2% formic acid (aq) and suctioned dry. The HLB SPE cartridges were then eluted with 2 mL of 5% NH<sub>4</sub>OH in methanol, and the tC18 SPE cartridges were eluted with 5 mL of 5% NH<sub>4</sub>OH in methanol. The samples were then transferred to HPLC autosampler vials and diluted as necessary in 5% NH<sub>4</sub>OH in methanol. Each sample was analyzed by LC/MS/MS electrospray.

## 6.2 Preparation of Standards and Fortification Solutions

A stock standard solution was prepared as specified in the method. The stock standard solution was prepared at a concentration of 1000 µg/mL by dissolving 0.1 g of the standard (corrected for purity and salt content, if necessary) in acetonitrile. From that solution, a 100 µg/mL fortification standard solution was prepared by taking 10 mL of the stock and bringing the volume up to 100 mL with acetonitrile. By taking 10 mL of the 100 µg/mL fortification standard and bringing the volume up to 100 mL with acetonitrile, a 10 µg/mL fortification standard was prepared. By taking 10 mL of the 10 µg/mL fortification standard and bringing the volume up to 100 mL with acetonitrile, a 1.0 µg/mL fortification standard was prepared. By taking 10 mL of the 1.0 µg/mL fortification standard and bringing the volume up to 100 mL with acetonitrile, a 0.1 µg/mL fortification standard was prepared. By taking 10 mL of the 0.1 µg/mL fortification standard and bringing the volume up to 100 mL with acetonitrile, a 0.01 µg/mL fortification standard was prepared. By taking 10 mL of the 0.01 µg/mL fortification standard and bringing the volume up to 100 mL with acetonitrile, a 0.001 µg/mL fortification standard was prepared.

A set of extracted calibration standards was prepared for each type of matrix (catfish fillet, whole body catfish, bass fillet, whole body bass, and clams). The extracted calibration standards were processed through the same extraction procedure as outlined above.

The following concentrations were prepared for the catfish fillet, bass fillet, whole body catfish, and clam samples:

Conc. of Fort. Solution (ng/mL)	Aliquot Volume (mL)	Final Volume of Standard (mL)	Final Conc. of Calibration Std. (ng/mL)	Final Conc. of Calibration Std. (ng/g)
1.0	50	2.0	0.025	0.10
1.0	100	2.0	0.050	0.20
10	25	2.0	0.125	0.50
10	50	2.0	0.25	1.0
10	100	2.0	0.50	2.0
10	125	2.0	0.625	2.5
100	25	2.0	1.25	5.0
100	50	2.0	2.5	10
100	100	2.0	5.0	20

The following concentrations were prepared for the whole body bass samples:

Conc. of Fort. Solution (ng/mL)	Aliquot Volume (mL)	Final Volume of Standard (mL)	Final Conc. of Calibration Std. (ng/mL)	Final Conc. of Calibration Std. (ng/g)
1.0	50	5.0	0.01	0.10
1.0	100	5.0	0.02	0.20
10	25	5.0	0.05	0.50
10	50	5.0	0.10	1.0
10	100	5.0	0.20	2.0
100	25	5.0	0.50	5.0
100	50	5.0	1.0	10
100	100	5.0	2.0	20
1000	25	5.0	5.0	50

The stock standard solution was stored in a freezer ( $-20^{\circ} \pm 5^{\circ}\text{C}$ ) when not in use. All other 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 the analyte was accomplished by LC/MS/MS electrospray. The retention time of PFOA ranged from 9.3 to 9.8 minutes. A method blank prepared for each data set was used to determine the LOQ. In instances where there were no peaks in the method blank, the LOQ was determined by the concentration of the lowest standard injected in the analytical run that met the 70–130% recovery range of its known value. In instances where there was a peak detected in the method blank, the blank was evaluated. If the response of the method blank was less than 50 % of the response of the lowest standard meeting the recovery criteria, then the LOQ was determined by the lowest standard. If the response of the method blank was greater than 50 % of the response of

the lowest standard meeting the recovery criteria, then the LOQ was raised to the standard that met the less than 50 % criteria.

#### 6.4 Instrument Sensitivity

The smallest standard amount injected during the chromatographic run had a concentration of 0.025 ng/mL for the catfish fillet, bass fillet, whole body catfish and clam samples, and a concentration of 0.010 ng/mL for the whole body bass samples.

#### 6.5 Description of LC/MS/MS Instruments and Operating Conditions

Instruments: API 5000 Biomolecular Mass Analyzer  
 Interface: SCIEX Turbo Ion Spray Liquid Introduction Interface  
 Computer: DELL Precision 360  
 DELL OptiPlex GX400  
 Software: PE SCIEX Analyst 1.4.1  
 HPLC: Hewlett Packard (HP) Series 1200  
 Hewlett Packard (HP) Series 1100  
     HP Quat Pump                      HP Vacuum Degasser  
     HP Autosampler                    HP Column Oven  
 HPLC Column: Thermo Electron Betasil C18, 100 mm x 2.1 mm, 5µm  
 Column Temp.: ~30° C  
 Injection Vol.: 10 µL  
 Mobile Phase (A): 2 mM Ammonium Acetate in water  
 Mobile Phase (B): Acetonitrile  
 Gradient:

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>	<u>Flow Rate</u> <u>(µL/mL)</u>
0.0	90	10	400
1.0	90	10	400
8.5	25	75	300
13.5	25	75	300
14.0	90	10	300
17.0	90	10	400
25.0	90	10	400

Total run time: ~25 min

Ions monitored:

<u>Analyte</u>	<u>Mode</u>	<u>Transition</u> <u>Monitored</u>	<u>Retention Time</u> <u>(min)</u>
PFOA	negative	413 → 369	~9.55 min.
PFOA Confirmation Ion	negative	413 → 219	~9.55 min.

## 6.6 Quantitation and Example Calculation

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

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

### Equation 1:

$$\text{Analyte found (ng/mL)} = \frac{((\sqrt{B^2 + 4AY} - B))}{2A} \times \text{Dilution Factor}$$

Where: A = X variable 1

B = X variable 2

C = intercept

Y = peak area

Equation 2 was used to convert the amount of analyte found in ng/mL to ng/g (ppb).

### Equation 2:

$$\text{Analyte found (ppb)} = \frac{[\text{analyte found (ng/mL)} \times \text{final volume}^{\wedge} \times \text{aliquot factor}(6)^*]}{\text{sample weight (3 g)}}$$

^ Final volume of 2mL for catfish fillet, bass fillet, whole body catfish, and clams.

Final volume of 5mL for whole body bass.

\*Aliquot factor calculated by the following equation:

$$\frac{[\text{initial volume of sample (30mL)}]}{[\text{extraction aliquot volume (5mL)}]} = 6$$

For samples fortified with known amounts of analyte prior to extraction, Equation 3 was used to calculate the percent recovery.

### Equation 3:

Recovery (%) =

$$\frac{(\text{total analyte found (ng/g)} - \text{average analyte in sample (ng/g)})}{\text{analyte added (ng/g)}} \times 100\%$$

An example of a calculation using an actual sample follows:

Bass fillet sample ExyLIMS ID: C0227547 Spike H (Set: 122707B), fortified at 2.0 ng/g with PFOA where:

Y (peak area)	=	73943
A (X variable 1)	=	-10600
B (X variable 2)	=	147000
C (intercept)	=	1250
dilution factor	=	1
ng/g PFOA added (fort level)	=	2.0 ng/g
average amt in corresponding sample	=	0.370

From equation 1:

Analyte found (ng/mL) =

$$\frac{((\sqrt{(147000^2 + 4(-10600)(73943)} - 4(-10600)(1250))) - 147000)}{2(-10600)} \times 1$$

$$= 0.514 \text{ ng/mL}$$

From equation 2:

$$\text{Analyte found, wet weight (ng/g)} = \frac{(0.514 \text{ ng/mL} \times 2 \text{ mL} \times 6)}{3 \text{ g}}$$

$$= 2.06 \text{ ng/g}$$

From equation 3:

$$\% \text{ Recovery} = \frac{(2.06 \text{ ng/g} - 0.370 \text{ ng/g})}{2.0 \text{ ng/g}} \times 100\%$$

$$= 85\%$$

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

## 7.0 EXPERIMENTAL DESIGN

For all fish and clam samples designated as laboratory matrix spikes, PFOA was added at a known concentration to the samples in the laboratory after the samples were weighed, homogenized and aliquoted for the extraction.

The fish samples were extracted in twenty seven sets. Each set included one reagent blank (method blank), two matrix controls, three matrix controls fortified at a lower level, three matrix controls fortified at a mid level, and three matrix controls fortified at a higher level of known concentrations. The catfish fillet samples were extracted in six sets. All six sets contained five catfish fillet samples, each from the same sample site. The bass fillet fish samples were extracted in five sets. All five sets contained five bass fillet fish

samples, each from the same sample site. The whole body catfish samples were extracted in six sets. All six sets contained five whole body catfish samples, each from the same sample site. The whole body bass samples were initially partially extracted in four sets. The initial analysis was not reported due to quality control failures. The samples were then extracted using a modified version of the method (outlined in protocol amendment #6). The whole body bass samples were then extracted in six sets. Five of the sets contained five whole body bass samples, each from the same sample site and one of the sets contained two samples, each from the same sample site.

The clam samples were extracted in one set. The set included one reagent blank (method blank), two matrix controls, three matrix controls fortified at a lower level, three matrix controls fortified at a mid level, and three matrix controls fortified at a higher level of known concentrations. The set contained six clam samples, one sample from each of the six sample sites.

One catfish fillet sample was re-extracted in one set. The set included one reagent blank (method blank), two matrix controls, three matrix controls fortified at a lower level, three matrix controls fortified at a mid level, and three matrix controls fortified at a higher level of known concentrations and the one catfish fillet sample.

One whole body bass sample was re-extracted in one set. The set included one reagent blank (method blank), two matrix controls, three matrix controls fortified at a lower level, three matrix controls fortified at a mid level, and three matrix controls fortified at a higher level of known concentrations and the one whole body bass sample.

Accuracies were assessed for each sample by reviewing the individual QC results obtained for each sample.

## 8.0 RESULTS

The limits of quantitation (LOQ) for the analyte in the fish fillet samples are listed in Table I. The target LOQ for the method for fish fillet samples was 0.20 ng/g. The limits of quantitation (LOQ) for the analyte in the whole body fish samples are listed in Table III. The target LOQ for the method for whole body fish samples was 0.20 ng/g. The limits of quantitation (LOQ) for the analyte in the clam samples are listed in Tables V. The target LOQ for the method for clam samples was 0.20 ng/g. After evaluation of the reagent blanks (method blanks) used for the analysis, the LOQ was determined. There were no instances where a raised LOQ was required. A discussion of the process used to evaluate the reagent blanks can be found in section 6.3 of the report. The LOQ for the analyte in the re-extracted whole body fish sample is listed in Table IV. The target LOQ for the method for the re-extracted whole body fish sample was 0.20 ng/g.

Analytical results and assessed accuracies for the analysis of PFOA found in the fish fillet samples are summarized in Table I. Fortification recoveries for PFOA in the fish fillet

samples are detailed in **Table VI**. The average percent recovery  $\pm$  standard deviation for PFOA in the fish fillet samples was  $105 \pm 23\%$ . The results for one fish fillet sample were not reported (NR) due to low spike recovery outside of the acceptance criteria range. The sample was re-extracted and reanalyzed (**Table II**), but matrix spike recovery of the re-extracted sample (**Table VII**) was also outside of the acceptance criteria range. Analytical results and assessed accuracies for the analysis of PFOA found in the whole body fish samples are summarized in **Table III**. Fortification recoveries for PFOA in the whole body fish samples are detailed in **Table VIII**. The average percent recovery  $\pm$  standard deviation for PFOA in the whole body fish samples was  $99 \pm 20\%$ . The results for one fish fillet sample were not reported (NR) due to low spike recovery outside of the acceptance criteria range. The sample was re-extracted and reanalyzed, and the analytical results and assessed accuracies for the analysis of PFOA found in the re-extracted whole body fish sample are summarized in **Table IV**. The fortification recovery for PFOA in the re-extracted whole body fish sample is detailed in **Table VIII**. The percent recovery for PFOA in the re-extracted whole body fish sample was 89%. Analytical results and assessed accuracies for the analysis of PFOA found in the clam samples are summarized in **Table V**. Fortification recoveries for PFOA in the clam samples are detailed in **Table X**. The average percent recovery  $\pm$  standard deviation for PFOA in the clam samples was  $111 \pm 19\%$ .

The assessed accuracy for the majority of the samples reported is  $\pm 30\%$ . The accuracies were assessed for each sample by reviewing the matrix spike whose spiking level most closely matches the endogenous concentration found in the sample.

## 9.0 CONCLUSION

Except as noted above, the fish and clam samples were successfully extracted and analyzed for PFOA according 3M Environmental Laboratory analytical method ETS-8-049.2 (V0003970).

## 10.0 RETENTION OF DATA AND SAMPLES

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

# TABLES



Table I. Summary of PFOA in Fish Fillet Samples

ExyLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227544	DL3-F02-MSF001-0-061207	0.434	0.20	30
C0227544 Rep	DL3-F02-MSF001-0-061207*	0.503	0.20	30
C0227546	DL3-F02-MSF002-0-061207	ND	0.20	30
C0227546 Rep	DL3-F02-MSF002-0-061207*	ND	0.20	30
C0227547	DL3-F02-MSF003-0-061207	0.359	0.20	30
C0227547 Rep	DL3-F02-MSF003-0-061207*	0.381	0.20	30
C0227548	DL3-F02-MSF004-0-061207	0.319	0.20	30
C0227548 Rep	DL3-F02-MSF004-0-061207*	0.270	0.20	30
C0227549	DL3-F02-MSF005-0-061207	ND	0.20	30
C0227549 Rep	DL3-F02-MSF005-0-061207*	ND	0.20	30
C0227550	DL3-F02-IPF001-0-061211	ND	0.20	30
C0227550 Rep	DL3-F02-IPF001-0-061211*	ND	0.20	30
C0227551	DL3-F02-IPF002-0-061212	ND	0.20	30
C0227551 Rep	DL3-F02-IPF002-0-061212*	ND	0.20	30
C0227552	DL3-F02-IPF003-0-061212	ND	0.20	30
C0227552 Rep	DL3-F02-IPF003-0-061212*	ND	0.20	30
C0227553	DL3-F02-IPF004-0-061212	ND	0.20	40
C0227553 Rep	DL3-F02-IPF004-0-061212*	ND	0.20	40
C0227554	DL3-F02-IPF005-0-061212	ND	0.20	30
C0227554 Rep	DL3-F02-IPF005-0-061212*	ND	0.20	30
C0227555	DL2-F02-IPF001-0-061209	ND	0.20	30
C0227555 Rep	DL2-F02-IPF001-0-061209*	ND	0.20	30
C0227556	DL2-F02-IPF002-0-061209	ND	0.20	30
C0227556 Rep	DL2-F02-IPF002-0-061209*	ND	0.20	30
C0227557	DL2-F02-IPF003-0-061209	ND	0.20	30
C0227557 Rep	DL2-F02-IPF003-0-061209*	ND	0.20	30
C0227558	DL2-F02-IPF004-0-061209	ND	0.20	50
C0227558 Rep	DL2-F02-IPF004-0-061209*	ND	0.20	50
C0227559	DL2-F02-IPF005-0-061209	ND	0.20	50
C0227559 Rep	DL2-F02-IPF005-0-061209*	ND	0.20	50
C0227560	DBC-F02-MSF001-0-061207	1.05 <sup>A</sup>	0.20	30
C0227560 Rep	DBC-F02-MSF001-0-061207*	0.787 <sup>A</sup>	0.20	30
C0227561	DBC-F02-MSF002-0-061207	0.327	0.20	30
C0227561 Rep	DBC-F02-MSF002-0-061207*	0.381	0.20	30
C0227562	DBC-F02-MSF003-0-061207	ND <sup>1</sup>	0.20	30
C0227562 Rep	DBC-F02-MSF003-0-061207*	0.207 <sup>1</sup>	0.20	30
C0227563	DBC-F02-MSF004-0-061207	ND	0.20	30
C0227563 Rep	DBC-F02-MSF004-0-061207*	ND	0.20	30
C0227564	DBC-F02-MSF005-0-061207	0.347 <sup>A</sup>	0.20	30
C0227564 Rep	DBC-F02-MSF005-0-061207*	0.288 <sup>A</sup>	0.20	30
C0227565	DBC-F02-IPF001-0-061211	1.42	0.20	30
C0227565 Rep	DBC-F02-IPF001-0-061211*	1.42	0.20	30
C0227566	DBC-F02-IPF002-0-061211	0.429	0.20	30
C0227566 Rep	DBC-F02-IPF002-0-061211*	0.511	0.20	30
C0227567	DBC-F02-IPF003-0-061211	0.367	0.20	30
C0227567 Rep	DBC-F02-IPF003-0-061211*	0.436	0.20	30

\*Laboratory Duplicate

<sup>A</sup>Relative Percent Difference > 20%<sup>1</sup>Relative Percent Difference was not calculated due to the presence of a nondetect and resulting uncertainty.

ND = Not detected at or above the acceptable LOQ.

Table I. Summary of PFOA in Fish Fillet Samples (continued)

ExyLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227568	DBC-F02-IPF004-0-061211	0.441	0.20	30
C0227568 Rep	DBC-F02-IPF004-0-061211*	0.423	0.20	30
C0227569	DBC-F02-IPF005-0-061211	0.728	0.20	50
C0227569 Rep	DBC-F02-IPF005-0-061211*	0.760	0.20	50
C0227570	DOU-F02-MSF001-0-061209	0.898 <sup>A</sup>	0.20	30
C0227570 Rep	DOU-F02-MSF001-0-061209*	1.12 <sup>A</sup>	0.20	30
C0227571	DOU-F02-MSF002-0-061212	0.748 <sup>A</sup>	0.20	30
C0227571 Rep	DOU-F02-MSF002-0-061212*	0.329 <sup>A</sup>	0.20	30
C0227572	DOU-F02-MSF003-0-061212	0.617 <sup>A</sup>	0.20	30
C0227572 Rep	DOU-F02-MSF003-0-061212*	0.502 <sup>A</sup>	0.20	30
C0227573	DOU-F02-MSF004-0-061212	0.501	0.20	30
C0227573 Rep	DOU-F02-MSF004-0-061212*	0.584	0.20	30
C0227574	DOU-F02-MSF005-0-061212	0.422 <sup>A</sup>	0.20	30
C0227574 Rep	DOU-F02-MSF005-0-061212*	0.343 <sup>A</sup>	0.20	30
C0227575	DOU-F02-IPF001-0-061212	0.319	0.20	30
C0227575 Rep	DOU-F02-IPF001-0-061212*	0.295	0.20	30
C0227576	DOU-F02-IPF002-0-061212	0.540	0.20	30
C0227576 Rep	DOU-F02-IPF002-0-061212*	0.526	0.20	30
C0227577	DOU-F02-IPF003-0-061212	0.452	0.20	30
C0227577 Rep	DOU-F02-IPF003-0-061212*	0.530	0.20	30
C0227578	DOU-F02-IPF004-0-061212	0.696 <sup>A</sup>	0.20	50
C022778 Rep	DOU-F02-IPF004-0-061212*	1.05 <sup>A</sup>	0.20	50
C0227579	DOU-F02-IPF005-0-061211	NR <sup>*</sup>	-	-
C0227579 Rep	DOU-F02-IPF005-0-061211*	NR <sup>*</sup>	-	-
C0227580	DLI-F02-MSF001-0-061209	ND	0.20	30
C0227580 Rep	DLI-F02-MSF001-0-061209*	ND	0.20	30
C0227581	DLI-F02-MSF002-0-061209	ND	0.20	30
C0227581 Rep	DLI-F02-MSF002-0-061209*	ND	0.20	30
C0227582	DLI-F02-MSF003-0-061209	ND	0.20	40
C0227582 Rep	DLI-F02-MSF003-0-061209*	ND	0.20	40
C0227583	DLI-F02-MSF004-0-061209	0.201 <sup>*</sup>	0.20	30
C0227583 Rep	DLI-F02-MSF004-0-061209*	ND <sup>1</sup>	0.20	30
C0227584	DLI-F02-MSF005-0-061209	ND	0.20	30
C0227584 Rep	DLI-F02-MSF005-0-061209*	ND	0.20	30
C0227585	DLI-F02-IPF001-0-061212	ND	0.20	30
C0227585 Rep	DLI-F02-IPF001-0-061212*	ND	0.20	30
C0227586	DLI-F02-IPF002-0-061212	ND	0.20	30
C0227586 Rep	DLI-F02-IPF002-0-061212*	ND	0.20	30
C0227587	DLI-F02-IPF003-0-061212	ND	0.20	30
C0227587 Rep	DLI-F02-IPF003-0-061212*	ND	0.20	30
C0227588	DLI-F02-IPF004-0-061212	ND	0.20	30
C0227588 Rep	DLI-F02-IPF004-0-061212*	ND	0.20	30
C0227589	DLI-F02-IPF005-0-061212	ND	0.20	30
C0227589 Rep	DLI-F02-IPF005-0-061212*	ND	0.20	30
C0227590	DMC-F02-MSF001-0-061209	ND	0.20	30
C0227590 Rep	DMC-F02-MSF001-0-061209*	ND	0.20	30

<sup>1</sup>Laboratory Duplicate<sup>A</sup>Relative Percent Difference > 20%<sup>\*</sup>Relative Percent Difference was not calculated due to the presence of a nondetect and resulting uncertainty.

ND = Not detected at or above the acceptable LOQ.

NR<sup>\*</sup> = Not reported due to quality control failures; see Table II for re-extracted sample results.

**Table I. Summary of PFOA in Fish Fillet Samples (continued)**

ExyLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227591	DMC-F02-MSF002-0-061209	0.290 <sup>^</sup>	0.20	30
C0227591 Rep	DMC-F02-MSF002-0-061209*	0.218 <sup>^</sup>	0.20	30
C0227592	DMC-F02-MSF003-0-061209	0.221 <sup>^</sup>	0.20	30
C0227592 Rep	DMC-F02-MSF003-0-061209*	ND <sup>^</sup>	0.20	30
C0227593	DMC-F02-MSF004-0-061212	ND	0.20	30
C0227593 Rep	DMC-F02-MSF004-0-061212*	ND	0.20	30
C0227594	DMC-F02-MSF005-0-061212	ND	0.20	30
C0227594 Rep	DMC-F02-MSF005-0-061212*	ND	0.20	30
C0227595	DMC-F02-IPF001-0-061212	ND	0.20	40
C0227595 Rep	DMC-F02-IPF001-0-061212*	ND	0.20	40
C0227596	DMC-F02-IPF002-0-061212	0.284	0.20	30
C0227596 Rep	DMC-F02-IPF002-0-061212*	0.245	0.20	30
C0227597	DMC-F02-IPF003-0-061212	0.226 <sup>^</sup>	0.20	30
C0227597 Rep	DMC-F02-IPF003-0-061212*	0.381 <sup>^</sup>	0.20	30
C0227598	DMC-F02-IPF004-0-061212	ND	0.20	40
C0227598 Rep	DMC-F02-IPF004-0-061212*	ND	0.20	40
C0227599	DMC-F02-IPF005-0-061212	ND	0.20	30
C0227599 Rep	DMC-F02-IPF005-0-061212*	ND	0.20	30

\*Laboratory Duplicate

<sup>^</sup>Relative Percent Difference > 20%<sup>^</sup>Relative Percent Difference was not calculated due to the presence of a nondetect and resulting uncertainty.

ND = Not detected at or above the acceptable LOQ.

**Table II. Summary of PFOA in Re-extracted Fish Fillet Samples**

ExyLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227579	DOU-F02-IPF005-0-061211	NR	-	-
C0227579 Rep	DOU-F02-IPF005-0-061211*	NR	-	-

\*Laboratory Duplicate  
 NR = Not reported due to quality control failures.

**Table III. Summary of PFOA in Whole Body Fish Samples**

ExLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227600	DL3-F02-MSW001-0-061207	ND	0.20	30
C0227600 Rep	DL3-F02-MSW001-0-061207	ND	0.20	30
C0227601	DL3-F02-MSW002-0-061207	ND <sup>1</sup>	0.20	40
C0227601 Rep	DL3-F02-MSW002-0-061207	0.386 <sup>1</sup>	0.20	40
C0227602	DL3-F02-MSW003-0-061207	ND	0.20	30
C0227602 Rep	DL3-F02-MSW003-0-061207	ND	0.20	30
C0227603	DL3-F02-MSW004-0-061207	ND	0.20	30
C0227603 Rep	DL3-F02-MSW004-0-061207	ND	0.20	30
C0227604	DL3-F02-MSW005-0-061207	ND	0.20	30
C0227604 Rep	DL3-F02-MSW005-0-061207	ND	0.20	30
C0227605	DL3-F02-IPW001-0-061212	ND	0.20	30
C0227605 Rep	DL3-F02-IPW001-0-061212	ND	0.20	30
C0227606	DL3-F02-IPW002-0-061212	ND	0.20	30
C0227606 Rep	DL3-F02-IPW002-0-061212	ND	0.20	30
C0227607	DL3-F02-IPW003-0-061212	ND	0.20	30
C0227607 Rep	DL3-F02-IPW003-0-061212	ND	0.20	30
C0227608	DL3-F02-IPW004-0-061212	ND	0.20	30
C0227608 Rep	DL3-F02-IPW004-0-061212	ND	0.20	30
C0227609	DL3-F02-IPW005-0-061212	ND	0.20	30
C0227609 Rep	DL3-F02-IPW005-0-061212	ND	0.20	30
C0227610	DL2-F02-MSW001-0-061211	0.522 <sup>1</sup>	0.20	30
C0227610 Rep	DL2-F02-MSW001-0-061211	0.231 <sup>1</sup>	0.20	30
C0227611	DL2-F02-MSW002-0-061211	0.591	0.20	30
C0227611 Rep	DL2-F02-MSW002-0-061211	0.509	0.20	30
C0227612	DL2-F02-IPW001-0-061209	ND	0.20	30
C0227612 Rep	DL2-F02-IPW001-0-061209	ND	0.20	30
C0227613	DL2-F02-IPW002-0-061209	ND	0.20	30
C0227613 Rep	DL2-F02-IPW002-0-061209	ND	0.20	30
C0227614	DL2-F02-IPW003-0-061209	ND	0.20	30
C0227614 Rep	DL2-F02-IPW003-0-061209	ND	0.20	30
C0227615	DL2-F02-IPW004-0-061209	ND	0.20	30
C0227615 Rep	DL2-F02-IPW004-0-061209	ND	0.20	30
C0227616	DL2-F02-IPW005-0-061209	ND	0.20	30
C0227616 Rep	DL2-F02-IPW005-0-061209	ND	0.20	30
C0227617	DBC-F02-MSW001-0-061207	2.95	0.20	30
C0227617 Rep	DBC-F02-MSW001-0-061207	2.83	0.20	30
C0227618	DBC-F02-MSW002-0-061207	2.72	0.20	30
C0227618 Rep	DBC-F02-MSW002-0-061207	2.67	0.20	30
C0227619	DBC-F02-MSW003-0-061207	1.30	0.20	30
C0227619 Rep	DBC-F02-MSW003-0-061207	1.30	0.20	30
C0227620	DBC-F02-MSW004-0-061207	1.52	0.20	30
C0227620 Rep	DBC-F02-MSW004-0-061207	1.60	0.20	30
C0227621	DBC-F02-MSW005-0-061207	1.76	0.20	30
C0227621 Rep	DBC-F02-MSW005-0-061207	1.72	0.20	30
C0227622	DBC-F02-IPW001-0-061211	1.19	0.20	30
C0227622 Rep	DBC-F02-IPW001-0-061211	1.09	0.20	30

<sup>1</sup>Laboratory Duplicate<sup>1</sup>Relative Percent Difference > 20%<sup>1</sup>Relative Percent Difference was not calculated due to the presence of a nondetect and resulting uncertainty.

ND = Not detected at or above the acceptable LOQ.

**Table III. Summary of PFOA in Whole Body Fish Samples  
(continued)**

ExyLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227623	DBC-F02-IPW002-0-061211	0.694	0.20	30
C0227623 Rep	DBC-F02-IPW002-0-061211	0.586	0.20	30
C0227624	DBC-F02-IPW003-0-061211	2.08	0.20	30
C0227624 Rep	DBC-F02-IPW003-0-061211	1.94	0.20	30
C0227625	DBC-F02-IPW004-0-061211	ND	0.20	30
C0227625 Rep	DBC-F02-IPW004-0-061211	ND	0.20	30
C0227626	DBC-F02-IPW005-0-061211	1.47	0.20	30
C0227626 Rep	DBC-F02-IPW005-0-061211	1.49	0.20	30
C0227627	DOU-F02-MSW001-0-061209	1.23	0.20	30
C0227627 Rep	DOU-F02-MSW001-0-061209	1.05	0.20	30
C0227628	DOU-F02-MSW002-0-061212	2.90	0.20	30
C0227628 Rep	DOU-F02-MSW002-0-061212	3.20	0.20	30
C0227629	DOU-F02-MSW003-0-061212	NR*	-	-
C0227629 Rep	DOU-F02-MSW003-0-061212	NR*	-	-
C0227630	DOU-F02-MSW004-0-061212	1.04	0.20	30
C0227630 Rep	DOU-F02-MSW004-0-061212	1.10	0.20	30
C0227631	DOU-F02-MSW005-0-061212	3.75	0.20	30
C0227631 Rep	DOU-F02-MSW005-0-061212	4.00	0.20	30
C0227632	DOU-F02-IPW001-0-061212	0.634	0.20	30
C0227632 Rep	DOU-F02-IPW001-0-061212	0.619	0.20	30
C0227633	DOU-F02-IPW002-0-061212	0.876	0.20	30
C0227633 Rep	DOU-F02-IPW002-0-061212	0.874	0.20	30
C0227634	DOU-F02-IPW003-0-061212	1.72	0.20	30
C0227634 Rep	DOU-F02-IPW003-0-061212	1.79	0.20	30
C0227635	DOU-F02-IPW004-0-061212	ND	0.20	30
C0227635 Rep	DOU-F02-IPW004-0-061212	ND	0.20	30
C0227636	DOU-F02-IPW005-0-061212	1.11	0.20	30
C0227636 Rep	DOU-F02-IPW005-0-061212	1.10	0.20	30
C0227637	DL1-F02-MSW001-0-061209	0.291 <sup>A</sup>	0.20	30
C0227637 Rep	DL1-F02-MSW001-0-061209	0.387 <sup>A</sup>	0.20	30
C0227638	DL1-F02-MSW002-0-061209	0.333 <sup>A</sup>	0.20	30
C0227638 Rep	DL1-F02-MSW002-0-061209	0.485 <sup>A</sup>	0.20	30
C0227639	DL1-F02-MSW003-0-061209	0.342 <sup>A</sup>	0.20	30
C0227639 Rep	DL1-F02-MSW003-0-061209	0.468 <sup>A</sup>	0.20	30
C0227640	DL1-F02-MSW004-0-061209	0.351	0.20	30
C0227640 Rep	DL1-F02-MSW004-0-061209	0.310	0.20	30
C0227641	DL1-F02-MSW005-0-061209	0.497	0.20	30
C0227641 Rep	DL1-F02-MSW005-0-061209	0.482	0.20	30
C0227642	DL1-F02-IPW001-0-061212	0.262 <sup>A</sup>	0.20	30
C0227642 Rep	DL1-F02-IPW001-0-061212	0.350 <sup>A</sup>	0.20	30
C0227643	DL1-F02-IPW002-0-061212	0.371 <sup>A</sup>	0.20	30
C0227643 Rep	DL1-F02-IPW002-0-061212	0.301 <sup>A</sup>	0.20	30
C0227644	DL1-F02-IPW003-0-061212	0.285	0.20	40
C0227644 Rep	DL1-F02-IPW003-0-061212	0.334	0.20	40
C0227645	DL1-F02-IPW004-0-061215	0.252	0.20	30
C0227645 Rep	DL1-F02-IPW004-0-061215	0.275	0.20	30

\*Laboratory Duplicate

<sup>A</sup>Relative Percent Difference > 20%

ND = Not detected at or above the acceptable LOQ.

NR\* = Not reported due to quality control failures; see Table IV for re-extracted sample results.

**Table III. Summary of PFOA in Whole Body Fish Samples  
(continued)**

ExyLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227646	DL1-F02-IPW005-0-061215	0.557	0.20	40
C0227646 Rep	DL1-F02-IPW005-0-061215	0.492	0.20	40
C0227647	DMC-F02-MSW001-0-061209	ND	0.20	30
C0227647 Rep	DMC-F02-MSW001-0-061209	ND	0.20	30
C0227648	DMC-F02-MSW002-0-061209	0.536	0.20	30
C0227648 Rep	DMC-F02-MSW002-0-061209	0.603	0.20	30
C0227649	DMC-F02-MSW003-0-061209	0.334 <sup>^</sup>	0.20	30
C0227649 Rep	DMC-F02-MSW003-0-061209	0.512 <sup>^</sup>	0.20	30
C0227650	DMC-F02-MSW004-0-061212	1.48 <sup>^</sup>	0.20	40
C0227650 Rep	DMC-F02-MSW004-0-061212	0.202 <sup>^</sup>	0.20	40
C0227651	DMC-F02-MSW005-0-061212	0.502 <sup>^</sup>	0.20	30
C0227651 Rep	DMC-F02-MSW005-0-061212	ND <sup>^</sup>	0.20	30
C0227652	DMC-F02-IPW001-0-061212	ND	0.20	30
C0227652 Rep	DMC-F02-IPW001-0-061212	ND	0.20	30
C0227653	DMC-F02-IPW002-0-061212	ND	0.20	30
C0227653 Rep	DMC-F02-IPW002-0-061212	ND	0.20	30
C0227654	DMC-F02-IPW003-0-061212	ND	0.20	30
C0227654 Rep	DMC-F02-IPW003-0-061212	ND	0.20	30
C0227655	DMC-F02-IPW004-0-061212	ND	0.20	30
C0227655 Rep	DMC-F02-IPW004-0-061212	ND	0.20	30
C0227656	DMC-F02-IPW005-0-061212	ND	0.20	30
C0227656 Rep	DMC-F02-IPW005-0-061212	ND	0.20	30

<sup>\*</sup>Laboratory Duplicate

<sup>^</sup>Relative Percent Difference > 20%

<sup>^</sup>Relative Percent Difference was not calculated due to the presence of a nondetect and resulting uncertainty.

ND = Not detected at or above the acceptable LOQ.

**Table IV. Summary of PFOA in Re-extracted Whole Body Fish Samples**

ExyLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227629	DOU-F02-MSW003-0-061212	1.27	0.20	30
C0227629 Rep	DOU-F02-MSW003-0-061212	1.20	0.20	30

\*Laboratory Duplicate



Table V. Summary of PFOA in Clam Samples

ExyLIMS ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid		
		Analyte Found (ppb, ng/g)	Acceptable LOQ (ng/g)	Assessed Accuracy (+/- %)
C0227658	DL3-I02-CFW001-0-061219	0.220	0.20	40
C0227658 Rep	DL3-I02-CFW001-0-061219*	0.218	0.20	40
C0227659	DL2-I02-CFW001-0-061219	0.424 <sup>^</sup>	0.20	30
C0227659 Rep	DL2-I02-CFW001-0-061219*	0.295 <sup>^</sup>	0.20	30
C0227660	DBC-I02-CFW001-0-061219	0.796 <sup>^</sup>	0.20	30
C0227680 Rep	DBC-I02-CFW001-0-061219*	1.00 <sup>^</sup>	0.20	30
C0227661	DOU-I02-CFW001-0-061219	0.832	0.20	30
C0227661 Rep	DOU-I02-CFW001-0-061219*	0.857	0.20	30
C0227662	DLI-I02-CFW001-0-061219	ND <sup>†</sup>	0.20	30
C0227662 Rep	DLI-I02-CFW001-0-061219*	0.221 <sup>†</sup>	0.20	30
C0227663	DMC-I02-CFW001-0-061219	ND	0.20	30
C0227663 Rep	DMC-I02-CFW001-0-061219*	ND	0.20	30

\*Laboratory Duplicate

<sup>^</sup>Relative Percent Difference > 20%<sup>†</sup>Relative Percent Difference was not calculated due to the presence of a nondetect and resulting uncertainty.

ND = Not detected at or above the acceptable LOQ.

**Table VI. Matrix Spike Recovery Summary of PFOA in Fish Fillet Samples**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DL3-F02-MSF001-0-061207 (C0227644 Spk D, 2.0 ppb spike)	2.0	0.469	1.95	74
DL3-F02-MSF001-0-061207 (C0227644 Spk E, 100 ppb spike)	100	0.469	NA	NA
DL3-F02-MSF002-0-061207 (C0227646 Spk F, 2.0 ppb spike)	2.0	ND	2.13	107
DL3-F02-MSF002-0-061207 (C0227646 Spk G, 100 ppb spike)	100	ND	NA	NA
DL3-F02-MSF003-0-061207 (C0227647 Spk H, 2.0 ppb spike)	2.0	0.370	2.05	84
DL3-F02-MSF003-0-061207 (C0227647 Spk I, 100 ppb spike)	100	0.370	NA	NA
DL3-F02-MSF004-0-061207 (C0227648 Spk J, 2.0 ppb spike)	2.0	0.295	1.97	64
DL3-F02-MSF004-0-061207 (C0227648 Spk K, 100 ppb spike)	100	0.295	NA	NA
DL3-F02-MSF005-0-061207 (C0227649 Spk L, 2.0 ppb spike)	2.0	ND	2.30	115
DL3-F02-MSF005-0-061207 (C0227649 Spk M, 100 ppb spike)	100	ND	NA	NA
DL3-F02-IPF001-0-061211 (C0227650 Spk D, 2.0 ppb spike)	2.0	ND	2.59	130
DL3-F02-IPF001-0-061211 (C0227650 Spk E, 100 ppb spike)	100	ND	NA	NA
DL3-F02-IPF002-0-061212 (C0227651 Spk F, 2.0 ppb spike)	2.0	ND	2.34	117
DL3-F02-IPF002-0-061212 (C0227651 Spk G, 100 ppb spike)	100	ND	NA	NA
DL3-F02-IPF003-0-061212 (C0227652 Spk H, 2.0 ppb spike)	2.0	ND	2.54	127
DL3-F02-IPF003-0-061212 (C0227652 Spk I, 100 ppb spike)	100	ND	NA	NA
DL3-F02-IPF004-0-061212 (C0227653 Spk J, 2.0 ppb spike)	2.0	ND	2.80	140
DL3-F02-IPF004-0-061212 (C0227653 Spk K, 100 ppb spike)	100	ND	NA	NA
DL3-F02-IPF005-0-061212 (C0227654 Spk L, 2.0 ppb spike)	2.0	ND	2.34	117
DL3-F02-IPF005-0-061212 (C0227654 Spk M, 100 ppb spike)	100	ND	NA	NA
DL2-F02-IPF001-0-061209 (C0227655 Spk D, 2.0 ppb spike)	2.0	ND	2.58	129
DL2-F02-IPF001-0-061209 (C0227655 Spk E, 100 ppb spike)	100	ND	NA	NA
DL2-F02-IPF002-0-061209 (C0227656 Spk F, 2.0 ppb spike)	2.0	ND	2.51	126
DL2-F02-IPF002-0-061209 (C0227656 Spk G, 100 ppb spike)	100	ND	NA	NA
DL2-F02-IPF003-0-061209 (C0227657 Spk H, 2.0 ppb spike)	2.0	ND	2.48	124
DL2-F02-IPF003-0-061209 (C0227657 Spk I, 100 ppb spike)	100	ND	NA	NA

ND = Not detected at or above the acceptable LOQ reported in Table I.  
 NA = Not applicable. This matrix spike concentration was not used to assess the accuracy for this analysis.  
 Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.

**Table VI. Matrix Spike Recovery Summary of PFOA in Fish Fillet Samples (continued)**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DL2-F02-IPF004-0-061209 (C0227688 Spk J, 2.0 ppb spike)	2.0	ND	2.84	142
DL2-F02-IPF004-0-061209 (C0227688 Spk K, 100 ppb spike)	100	ND	NA	NA
DL2-F02-IPF005-0-061209 (C0227689 Spk L, 2.0 ppb spike)	2.0	ND	2.94	147
DL2-F02-IPF005-0-061209 (C0227689 Spk M, 100 ppb spike)	100	ND	NA	NA
DBC-F02-MSF001-0-061207 (C0227660 Spk D, 2.0 ppb spike)	2.0	0.920	2.38	73
DBC-F02-MSF001-0-061207 (C0227660 Spk E, 100 ppb spike)	100	0.920	NA	NA
DBC-F02-MSF002-0-061207 (C0227661 Spk F, 2.0 ppb spike)	2.0	0.354	2.23	94
DBC-F02-MSF002-0-061207 (C0227661 Spk G, 100 ppb spike)	100	0.354	NA	NA
DBC-F02-MSF003-0-061207 (C0227662 Spk H, 2.0 ppb spike)	2.0	0.204	2.28	104
DBC-F02-MSF003-0-061207 (C0227662 Spk I, 100 ppb spike)	100	0.204	NA	NA
DBC-F02-MSF004-0-061207 (C0227663 Spk J, 2.0 ppb spike)	2.0	ND	1.77	89
DBC-F02-MSF004-0-061207 (C0227663 Spk K, 100 ppb spike)	100	ND	NA	NA
DBC-F02-MSF005-0-061207 (C0227664 Spk L, 2.0 ppb spike)	2.0	0.308	2.27	98
DBC-F02-MSF005-0-061207 (C0227664 Spk M, 100 ppb spike)	100	0.308	NA	NA
DBC-F02-IPF001-0-061211 (C0227665 Spk F, 2.0 ppb spike)	2.0	1.42	3.85	122
DBC-F02-IPF001-0-061211 (C0227665 Spk E, 100 ppb spike)	100	1.42	NA	NA
DBC-F02-IPF002-0-061211 (C0227666 Spk F, 2.0 ppb spike)	2.0	0.470	3.08	130
DBC-F02-IPF002-0-061211 (C0227666 Spk G, 100 ppb spike)	100	0.470	NA	NA
DBC-F02-IPF003-0-061211 (C0227667 Spk H, 2.0 ppb spike)	2.0	0.402	2.95	127
DBC-F02-IPF003-0-061211 (C0227667 Spk I, 100 ppb spike)	100	0.402	NA	NA
DBC-F02-IPF004-0-061211 (C0227668 Spk J, 2.0 ppb spike)	2.0	0.432	2.78	116
DBC-F02-IPF004-0-061211 (C0227668 Spk K, 100 ppb spike)	100	0.432	NA	NA
DBC-F02-IPF005-0-061211 (C0227669 Spk L, 2.0 ppb spike)	2.0	0.743	3.60	143
DBC-F02-IPF005-0-061211 (C0227669 Spk M, 100 ppb spike)	100	0.743	NA	NA
DOU-F02-MSF001-0-061209 (C0227870 Spk D, 2.0 ppb spike)	2.0	1.01	3.01	100
DOU-F02-MSF001-0-061209 (C0227870 Spk E, 100 ppb spike)	100	1.01	NA	NA

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

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

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

**Table VI. Matrix Spike Recovery Summary of PFOA in Fish Fillet Samples (continued)**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DOU-F02-MSF002-0-061212 (C0227671 Spk F, 2.0 ppb spike)	2.0	0.538	2.33	90
DOU-F02-MSF002-0-061212 (C0227671 Spk G, 100 ppb spike)	100	0.538	NA	NA
DOU-F02-MSF003-0-061212 (C0227672 Spk H, 2.0 ppb spike)	2.0	0.559	2.05	75
DOU-F02-MSF003-0-061212 (C0227672 Spk I, 100 ppb spike)	100	0.559	NA	NA
DOU-F02-MSF004-0-061212 (C0227673 Spk J, 2.0 ppb spike)	2.0	0.543	1.94	70
DOU-F02-MSF004-0-061212 (C0227673 Spk K, 100 ppb spike)	100	0.543	NA	NA
DOU-F02-MSF005-0-061212 (C0227674 Spk L, 2.0 ppb spike)	2.0	0.383	1.84	73
DOU-F02-MSF005-0-061212 (C0227674 Spk M, 100 ppb spike)	100	0.383	NA	NA
DOU-F02-IPF001-0-061212 (C0227675 Spk N, 2.0 ppb spike)	2.0	0.307	2.83	110
DOU-F02-IPF001-0-061212 (C0227675 Spk O, 100 ppb spike)	100	0.307	NA	NA
DOU-F02-IPF002-0-061212 (C0227676 Spk P, 2.0 ppb spike)	2.0	0.533	1.99	73
DOU-F02-IPF002-0-061212 (C0227676 Spk Q, 100 ppb spike)	100	0.533	NA	NA
DOU-F02-IPF003-0-061212 (C0227677 Spk R, 2.0 ppb spike)	2.0	0.481	2.01	76
DOU-F02-IPF003-0-061212 (C0227677 Spk S, 100 ppb spike)	100	0.481	NA	NA
DOU-F02-IPF004-0-061212 (C0227678 Spk T, 2.0 ppb spike)	2.0	0.871	1.90	51
DOU-F02-IPF004-0-061212 (C0227678 Spk U, 100 ppb spike)	100	0.871	NA	NA
DOU-F02-IPF005-0-061211 (C0227679 Spk V, 2.0 ppb spike)	2.0	NR*	NR*	NR*
DOU-F02-IPF005-0-061211 (C0227679 Spk W, 100 ppb spike)	100	NR*	NR*	NR*
DLI-F02-MSF001-0-061209 (C0227680 Spk D, 2.0 ppb spike)	2.0	ND	2.25	113
DLI-F02-MSF001-0-061209 (C0227680 Spk E, 100 ppb spike)	100	ND	NA	NA
DLI-F02-MSF002-0-061209 (C0227681 Spk F, 2.0 ppb spike)	2.0	ND	2.36	118
DLI-F02-MSF002-0-061209 (C0227681 Spk G, 100 ppb spike)	100	ND	NA	NA
DLI-F02-MSF003-0-061209 (C0227682 Spk H, 2.0 ppb spike)	2.0	ND	2.71	136
DLI-F02-MSF003-0-061209 (C0227682 Spk I, 100 ppb spike)	100	ND	NA	NA
DLI-F02-MSF004-0-061209 (C0227683 Spk J, 2.0 ppb spike)	2.0	0.201	2.19	99
DLI-F02-MSF004-0-061209 (C0227683 Spk K, 100 ppb spike)	100	0.201	NA	NA

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

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

NR\* = Not reported due to quality control failures; see Table VII for re-extracted sample results.

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

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

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DLI-F02-MSF005-0-061209 (C0227684 Spk L, 2.0 ppb spike)	2.0	ND	1.96	98
DLI-F02-MSF005-0-061209 (C0227684 Spk M, 100 ppb spike)	100	ND	NA	NA
DLI-F02-IPF001-0-061212 (C0227688 Spk D, 2.0 ppb spike)	2.0	ND	1.99	100
DLI-F02-IPF001-0-061212 (C0227688 Spk E, 100 ppb spike)	100	ND	NA	NA
DLI-F02-IPF002-0-061212 (C0227686 Spk F, 2.0 ppb spike)	2.0	ND	2.18	109
DLI-F02-IPF002-0-061212 (C0227686 Spk G, 100 ppb spike)	100	ND	NA	NA
DLI-F02-IPF003-0-061212 (C0227687 Spk H, 2.0 ppb spike)	2.0	ND	1.92	96
DLI-F02-IPF003-0-061212 (C0227687 Spk I, 100 ppb spike)	100	ND	NA	NA
DLI-F02-IPF004-0-061212 (C0227688 Spk J, 2.0 ppb spike)	2.0	ND	2.07	104
DLI-F02-IPF004-0-061212 (C0227688 Spk K, 100 ppb spike)	100	ND	NA	NA
DLI-F02-IPF005-0-061212 (C0227689 Spk L, 2.0 ppb spike)	2.0	ND	2.08	104
DLI-F02-IPF005-0-061212 (C0227689 Spk M, 100 ppb spike)	100	ND	NA	NA
DMC-F02-MSF001-0-061209 (C0227690 Spk D, 2.0 ppb spike)	2.0	ND	1.61	81
DMC-F02-MSF001-0-061209 (C0227690 Spk E, 100 ppb spike)	100	ND	NA	NA
DMC-F02-MSF002-0-061209 (C0227691 Spk F, 2.0 ppb spike)	2.0	0.264	1.84	79
DMC-F02-MSF002-0-061209 (C0227691 Spk G, 100 ppb spike)	100	0.264	NA	NA
DMC-F02-MSF003-0-061209 (C0227692 Spk H, 2.0 ppb spike)	2.0	0.211	2.21	100
DMC-F02-MSF003-0-061209 (C0227692 Spk I, 100 ppb spike)	100	0.211	NA	NA
DMC-F02-MSF004-0-061212 (C0227693 Spk J, 2.0 ppb spike)	2.0	ND	1.61	91
DMC-F02-MSF004-0-061212 (C0227693 Spk K, 100 ppb spike)	100	ND	NA	NA
DMC-F02-MSF005-0-061212 (C0227694 Spk L, 2.0 ppb spike)	2.0	ND	1.40	70
DMC-F02-MSF005-0-061212 (C0227694 Spk M, 100 ppb spike)	100	ND	NA	NA
DMC-F02-IPF001-0-061212 (C0227695 Spk D, 2.0 ppb spike)	2.0	ND	2.61	131
DMC-F02-IPF001-0-061212 (C0227695 Spk E, 100 ppb spike)	100	ND	NA	NA
DMC-F02-IPF002-0-061212 (C0227696 Spk F, 2.0 ppb spike)	2.0	0.264	2.36	105
DMC-F02-IPF002-0-061212 (C0227696 Spk G, 100 ppb spike)	100	0.264	NA	NA

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

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

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

**Table VI. Matrix Spike Recovery Summary of PFOA in Fish Fillet Samples (continued)**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DMC-F02-IPF003-0-061212 (C0227597 Spk H, 2.0 ppb spike)	2.0	0.304	2.17	93
DMC-F02-IPF003-0-061212 (C0227597 Spk I, 100 ppb spike)	100	0.304	NA	NA
DMC-F02-IPF004-0-061212 (C0227598 Spk J, 2.0 ppb spike)	2.0	ND	2.70	135
DMC-F02-IPF004-0-061212 (C0227598 Spk K, 100 ppb spike)	100	ND	NA	NA
DMC-F02-IPF005-0-061212 (C0227599 Spk L, 2.0 ppb spike)	2.0	ND	2.53	127
DMC-F02-IPF005-0-061212 (C0227599 Spk M, 100 ppb spike)	100	ND	NA	NA
			<b>Average:</b>	<b>105</b>
			<b>Standard Deviation:</b>	<b>23</b>

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

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

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

**Table VII. Matrix Spike Recovery Summary of PFOA in Re-extracted Fish Fillet Samples**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DOU-F02-IPF005-0-061211 (C0227579 Spk F, 2.0 ppb spike)	2.0	NR	NR	NR

NR = Not reported due to quality control failures.

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

Table VIII. Matrix Spike Recovery Summary of PFOA in Whole Body Fish Samples

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DL3-F02-MSW001-0-061207 (C0227600 Spk D, 2.0 ppb spike)	2.0	ND	2.08	105
DL3-F02-MSW001-0-061207 (C0227600 Spk E, 400 ppb spike)	400	ND	NA	NA
DL3-F02-MSW002-0-061207 (C0227601 Spk F, 2.0 ppb spike)	2.0	0.293	1.82	66
DL3-F02-MSW002-0-061207 (C0227601 Spk G, 400 ppb spike)	400	0.293	NA	NA
DL3-F02-MSW003-0-061207 (C0227602 Spk H, 2.0 ppb spike)	2.0	ND	2.45	123
DL3-F02-MSW003-0-061207 (C0227602 Spk I, 400 ppb spike)	400	ND	NA	NA
DL3-F02-MSW004-0-061207 (C0227603 Spk J, 2.0 ppb spike)	2.0	ND	2.26	113
DL3-F02-MSW004-0-061207 (C0227603 Spk K, 400 ppb spike)	400	ND	NA	NA
DL3-F02-MSW005-0-061207 (C0227604 Spk L, 2.0 ppb spike)	2.0	ND	2.42	121
DL3-F02-MSW005-0-061207 (C0227604 Spk M, 400 ppb spike)	400	ND	NA	NA
DL3-F02-IPW001-0-061212 (C0227606 Spk O, 2.0 ppb spike)	2.0	ND	2.13	107
DL3-F02-IPW001-0-061212 (C0227606 Spk P, 50 ppb spike)	50	ND	NA	NA
DL3-F02-IPW002-0-061212 (C0227606 Spk F, 2.0 ppb spike)	2.0	ND	1.96	98
DL3-F02-IPW002-0-061212 (C0227606 Spk G, 50 ppb spike)	50	ND	NA	NA
DL3-F02-IPW003-0-061212 (C0227607 Spk H, 2.0 ppb spike)	2.0	ND	2.23	112
DL3-F02-IPW003-0-061212 (C0227607 Spk I, 50 ppb spike)	50	ND	NA	NA
DL3-F02-IPW004-0-061212 (C0227608 Spk J, 2.0 ppb spike)	2.0	ND	1.81	91
DL3-F02-IPW004-0-061212 (C0227608 Spk K, 50 ppb spike)	50	ND	NA	NA
DL3-F02-IPW005-0-061212 (C0227609 Spk L, 2.0 ppb spike)	2.0	ND	1.69	85
DL3-F02-IPW005-0-061212 (C0227609 Spk M, 50 ppb spike)	50	ND	NA	NA
DL2-F02-MSW001-0-061211 (C0227610 Spk D, 2.0 ppb spike)	2.0	0.377	2.70	118
DL2-F02-MSW001-0-061211 (C0227610 Spk E, 400 ppb spike)	400	0.377	NA	NA
DL2-F02-MSW002-0-061211 (C0227611 Spk F, 2.0 ppb spike)	2.0	0.350	2.04	75
DL2-F02-MSW002-0-061211 (C0227611 Spk G, 400 ppb spike)	400	0.350	NA	NA

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

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

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



**Table VIII. Matrix Spike Recovery Summary of PFOA in Whole Body Fish Samples (continued)**

Sample Description	C8 Acid PFOA Perfluorooctanoic Acid			
	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	Amount Recovered (ng/g)	Recovery (%)
DL2-F02-IPW001-0-061209 (C0227612 Spk D, 2.0 ppb spike)	2.0	ND	1.85	93
DL2-F02-IPW001-0-061209 (C0227612 Spk E, 200 ppb spike)	200	ND	NA	NA
DL2-F02-IPW002-0-061209 (C0227613 Spk F, 2.0 ppb spike)	2.0	ND	2.07	104
DL2-F02-IPW002-0-061209 (C0227613 Spk G, 200 ppb spike)	200	ND	NA	NA
DL2-F02-IPW003-0-061209 (C0227614 Spk H, 2.0 ppb spike)	2.0	ND	1.44	72
DL2-F02-IPW003-0-061209 (C0227614 Spk I, 200 ppb spike)	200	ND	NA	NA
DL2-F02-IPW004-0-061209 (C0227615 Spk J, 2.0 ppb spike)	2.0	ND	1.79	90
DL2-F02-IPW004-0-061209 (C0227615 Spk K, 200 ppb spike)	200	ND	NA	NA
DL2-F02-IPW005-0-061209 (C0227616 Spk L, 2.0 ppb spike)	2.0	ND	1.68	84
DL2-F02-IPW005-0-061209 (C0227616 Spk M, 200 ppb spike)	200	ND	NA	NA
DBC-F02-MSW001-0-061207 (C0227617 Spk D, 2.0 ppb spike)	2.0	2.89	5.13	112
DBC-F02-MSW001-0-061207 (C0227617 Spk E, 10000 ppb spike)	10000	2.89	NA	NA
DBC-F02-MSW002-0-061207 (C0227618 Spk F, 2.0 ppb spike)	2.0	2.69	4.63	107
DBC-F02-MSW002-0-061207 (C0227618 Spk G, 10000 ppb spike)	10000	2.69	NA	NA
DBC-F02-MSW003-0-061207 (C0227619 Spk H, 2.0 ppb spike)	2.0	1.30	3.21	98
DBC-F02-MSW003-0-061207 (C0227619 Spk I, 10000 ppb spike)	10000	1.30	NA	NA
DBC-F02-MSW004-0-061207 (C0227620 Spk J, 2.0 ppb spike)	2.0	1.56	4.07	126
DBC-F02-MSW004-0-061207 (C0227620 Spk K, 10000 ppb spike)	10000	1.56	NA	NA
DBC-F02-MSW005-0-061207 (C0227621 Spk L, 2.0 ppb spike)	2.0	1.74	3.95	111
DBC-F02-MSW005-0-061207 (C0227621 Spk M, 10000 ppb spike)	10000	1.74	NA	NA
DBC-F02-IPW001-0-061211 (C0227622 Spk D, 2.0 ppb spike)	2.0	1.14	3.25	108
DBC-F02-IPW001-0-061211 (C0227622 Spk E, 5000 ppb spike)	5000	1.14	NA	NA
DBC-F02-IPW002-0-061211 (C0227623 Spk F, 2.0 ppb spike)	2.0	0.640	2.67	102
DBC-F02-IPW002-0-061211 (C0227623 Spk G, 5000 ppb spike)	5000	0.640	NA	NA

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

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

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

**Table VIII. Matrix Spike Recovery Summary of PFOA in Whole Body Fish Samples (continued)**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic acid	
			Amount Recovered (ng/g)	Recovery (%)
DBC-F02-IPW003-0-061211 (C0227624 Spk M, 2.0 ppb spike)	2.0	2.01	4.23	111
DBC-F02-IPW003-0-061211 (C0227624 Spk I, 5000 ppb spike)	5000	2.01	NA	NA
DBC-F02-IPW004-0-061211 (C0227625 Spk J, 2.0 ppb spike)	2.0	ND	2.43	122
DBC-F02-IPW004-0-061211 (C0227625 Spk K, 5000 ppb spike)	5000	ND	NA	NA
DBC-F02-IPW005-0-061211 (C0227626 Spk L, 2.0 ppb spike)	2.0	1.48	2.93	73
DBC-F02-IPW005-0-061211 (C0227626 Spk M, 5000 ppb spike)	5000	1.48	NA	NA
DOU-F02-MSW001-0-061209 (C0227627 Spk O, 2.0 ppb spike)	2.0	1.14	3.17	102
DOU-F02-MSW001-0-061209 (C0227627 Spk E, 25000 ppb spike)	25000	1.14	NA	NA
DOU-F02-MSW002-0-061212 (C0227628 Spk F, 2.0 ppb spike)	2.0	3.05	4.95	95
DOU-F02-MSW002-0-061212 (C0227628 Spk G, 25000 ppb spike)	25000	3.05	NA	NA
DOU-F02-MSW003-0-061212 (C0227629 Spk H, 2.0 ppb spike)	2.0	NR*	NR*	NR*
DOU-F02-MSW003-0-061212 (C0227629 Spk I, 25000 ppb spike)	25000	NR*	NR*	NR*
DOU-F02-MSW004-0-061212 (C0227630 Spk J, 2.0 ppb spike)	2.0	1.07	3.62	128
DOU-F02-MSW004-0-061212 (C0227630 Spk K, 25000 ppb spike)	25000	1.07	NA	NA
DOU-F02-MSW005-0-061212 (C0227631 Spk L, 2.0 ppb spike)	2.0	3.87	5.41	77
DOU-F02-MSW005-0-061212 (C0227631 Spk M, 25000 ppb spike)	25000	3.87	NA	NA
DOU-F02-IPW001-0-061212 (C0227632 Spk O, 2.0 ppb spike)	2.0	0.626	2.85	111
DOU-F02-IPW001-0-061212 (C0227632 Spk E, 5000 ppb spike)	5000	0.626	NA	NA
DOU-F02-IPW002-0-061212 (C0227633 Spk F, 2.0 ppb spike)	2.0	0.875	2.30	71
DOU-F02-IPW002-0-061212 (C0227633 Spk G, 5000 ppb spike)	5000	0.875	NA	NA
DOU-F02-IPW003-0-061212 (C0227634 Spk H, 2.0 ppb spike)	2.0	1.76	4.22	123
DOU-F02-IPW003-0-061212 (C0227634 Spk I, 5000 ppb spike)	5000	1.76	NA	NA
DOU-F02-IPW004-0-061212 (C0227635 Spk J, 2.0 ppb spike)	2.0	ND	2.21	111
DOU-F02-IPW004-0-061212 (C0227635 Spk K, 5000 ppb spike)	5000	ND	NA	NA

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

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

NR\* = Not reported due to quality control failures; see Table IX for re-extracted sample results.

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

**Table VIII. Matrix Spike Recovery Summary of PFOA in Whole Body Fish Samples (continued)**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found In Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DOU-F02-IPW005-0-061212 (C0227636 Spk L, 2.0 ppb spike)	2.0	1.10	2.62	76
DOU-F02-IPW005-0-061212 (C0227636 Spk M, 5000 ppb spike)	5000	1.10	NA	NA
DL1-F02-MSW001-0-061209 (C0227637 Spk D, 2.0 ppb spike)	2.0	0.339	2.37	102
DL1-F02-MSW001-0-061209 (C0227637 Spk E, 1000 ppb spike)	1000	0.339	NA	NA
DL1-F02-MSW002-0-061209 (C0227638 Spk F, 2.0 ppb spike)	2.0	0.399	2.99	130
DL1-F02-MSW002-0-061208 (C0227638 Spk G, 1000 ppb spike)	1000	0.399	NA	NA
DL1-F02-MSW003-0-061209 (C0227639 Spk H, 2.0 ppb spike)	2.0	0.405	3.01	130
DL1-F02-MSW003-0-061209 (C0227639 Spk I, 1000 ppb spike)	1000	0.405	NA	NA
DL1-F02-MSW004-0-061209 (C0227640 Spk J, 2.0 ppb spike)	2.0	0.330	2.75	121
DL1-F02-MSW004-0-061209 (C0227640 Spk K, 1000 ppb spike)	1000	0.330	NA	NA
DL1-F02-MSW005-0-061209 (C0227641 Spk L, 2.0 ppb spike)	2.0	0.490	3.05	128
DL1-F02-MSW005-0-061209 (C0227641 Spk M, 1000 ppb spike)	1000	0.490	NA	NA
DL1-F02-IPW001-0-061212 (C0227642 Spk D, 2.0 ppb spike)	2.0	0.305	1.74	72
DL1-F02-IPW001-0-061212 (C0227642 Spk E, 200 ppb spike)	200	0.305	NA	NA
DL1-F02-IPW002-0-061212 (C0227643 Spk F, 2.0 ppb spike)	2.0	0.336	2.29	98
DL1-F02-IPW002-0-061212 (C0227643 Spk G, 200 ppb spike)	200	0.336	NA	NA
DL1-F02-IPW003-0-061212 (C0227644 Spk H, 2.0 ppb spike)	2.0	0.309	1.56	63
DL1-F02-IPW003-0-061212 (C0227644 Spk I, 200 ppb spike)	200	0.309	NA	NA
DL1-F02-IPW004-0-061215 (C0227645 Spk J, 2.0 ppb spike)	2.0	0.263	2.30	102
DL1-F02-IPW004-0-061215 (C0227645 Spk K, 200 ppb spike)	200	0.263	NA	NA
DL1-F02-IPW005-0-061215 (C0227646 Spk L, 2.0 ppb spike)	2.0	0.524	1.78	63
DL1-F02-IPW005-0-061215 (C0227646 Spk M, 200 ppb spike)	200	0.524	NA	NA
DMC-F02-MSW001-0-061209 (C0227647 Spk D, 2.0 ppb spike)	2.0	ND	2.39	120
DMC-F02-MSW001-0-061209 (C0227647 Spk E, 1000 ppb spike)	1000	ND	NA	NA

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

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

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

**Table VIII. Matrix Spike Recovery Summary of PFOA in Whole Body Fish Samples (continued)**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DMC-F02-MSW002-0-061209 (C0227648 Spk F, 2.0 ppb spike)	2.0	0.570	2.29	86
DMC-F02-MSW002-0-061209 (C0227648 Spk G, 1000 ppb spike)	1000	0.570	NA	NA
DMC-F02-MSW003-0-061209 (C0227649 Spk H, 2.0 ppb spike)	2.0	0.423	2.47	102
DMC-F02-MSW003-0-061209 (C0227649 Spk I, 1000 ppb spike)	1000	0.423	NA	NA
DMC-F02-MSW004-0-061212 (C0227650 Spk J, 2.0 ppb spike)	2.0	0.842	2.23	69
DMC-F02-MSW004-0-061212 (C0227650 Spk K, 1000 ppb spike)	1000	0.842	NA	NA
DMC-F02-MSW005-0-061212 (C0227651 Spk L, 2.0 ppb spike)	2.0	0.351	2.56	110
DMC-F02-MSW005-0-061212 (C0227651 Spk M, 1000 ppb spike)	1000	0.351	NA	NA
DMC-F02-IPW001-0-061212 (C0227652 Spk D, 2.0 ppb spike)	2.0	ND	1.72	86
DMC-F02-IPW001-0-061212 (C0227652 Spk E, 200 ppb spike)	200	ND	NA	NA
DMC-F02-IPW002-0-061212 (C0227653 Spk F, 2.0 ppb spike)	2.0	ND	1.83	92
DMC-F02-IPW002-0-061212 (C0227653 Spk G, 200 ppb spike)	200	ND	NA	NA
DMC-F02-IPW003-0-061212 (C0227654 Spk H, 2.0 ppb spike)	2.0	ND	1.50	75
DMC-F02-IPW003-0-061212 (C0227654 Spk I, 200 ppb spike)	200	ND	NA	NA
DMC-F02-IPW004-0-061212 (C0227655 Spk J, 2.0 ppb spike)	2.0	ND	2.42	121
DMC-F02-IPW004-0-061212 (C0227655 Spk K, 200 ppb spike)	200	ND	NA	NA
DMC-F02-IPW005-0-061212 (C0227656 Spk L, 2.0 ppb spike)	2.0	ND	1.42	71
DMC-F02-IPW005-0-061212 (C0227656 Spk M, 200 ppb spike)	200	ND	NA	NA

Average: 99  
Standard Deviation: 20

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

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

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

**Table IX. Matrix Spike Recovery Summary of PFOA in Re-extracted Whole Body Fish Samples**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	C8 Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DOU-F02-MSW003-0-061212 (CP227628 Spk E, 2.0 ppb spike)	2.0	1.23	3.01	89
			Average:	89
			Standard Deviation:	NA

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

**Table X. Matrix Spike Recovery Summary of PFOA in Clam Samples**

Sample Description	Amount Spiked (ng/g)	Avg Amt Found in Sample (ng/g)	CB Acid PFOA Perfluorooctanoic Acid	
			Amount Recovered (ng/g)	Recovery (%)
DL3-I02-CFW001-0-061219 (C0227658 Spk D, 2.0 ppb Spike)	2.0	0.219	2.95	137
DL3-I02-CFW001-0-061219 (C0227658 Spk E, 20 ppb Spike)	20	0.219	NA	NA
DL2-I02-CFW001-0-061219 (C0227659 Spk F, 2.0 ppb Spike)	2.0	0.360	2.78	121
DL2-I02-CFW001-0-061219 (C0227659 Spk G, 20 ppb Spike)	20	0.360	NA	NA
DBC-I02-CFW001-0-061219 (C0227660 Spk H, 2.0 ppb Spike)	2.0	0.899	3.25	118
DBC-I02-CFW001-0-061219 (C0227660 Spk I, 20 ppb Spike)	20	0.899	NA	NA
DOU-I02-CFW001-0-061219 (C0227661 Spk J, 2.0 ppb Spike)	2.0	0.845	3.04	110
DOU-I02-CFW001-0-061219 (C0227661 Spk K, 20 ppb Spike)	20	0.845	NA	NA
DLI-I02-CFW001-0-061219 (C0227662 Spk L, 2.0 ppb Spike)	2.0	0.211	2.08	93
DLI-I02-CFW001-0-061219 (C0227662 Spk M, 20 ppb Spike)	20	0.211	NA	NA
DMC-I02-CFW001-0-061219 (C0227663 Spk N, 2.0 ppb Spike)	2.0	ND	1.73	87
DMC-I02-CFW001-0-061219 (C0227663 Spk O, 20 ppb Spike)	20	ND	NA	NA
<b>Average:</b>				111
<b>Standard Deviation:</b>				19

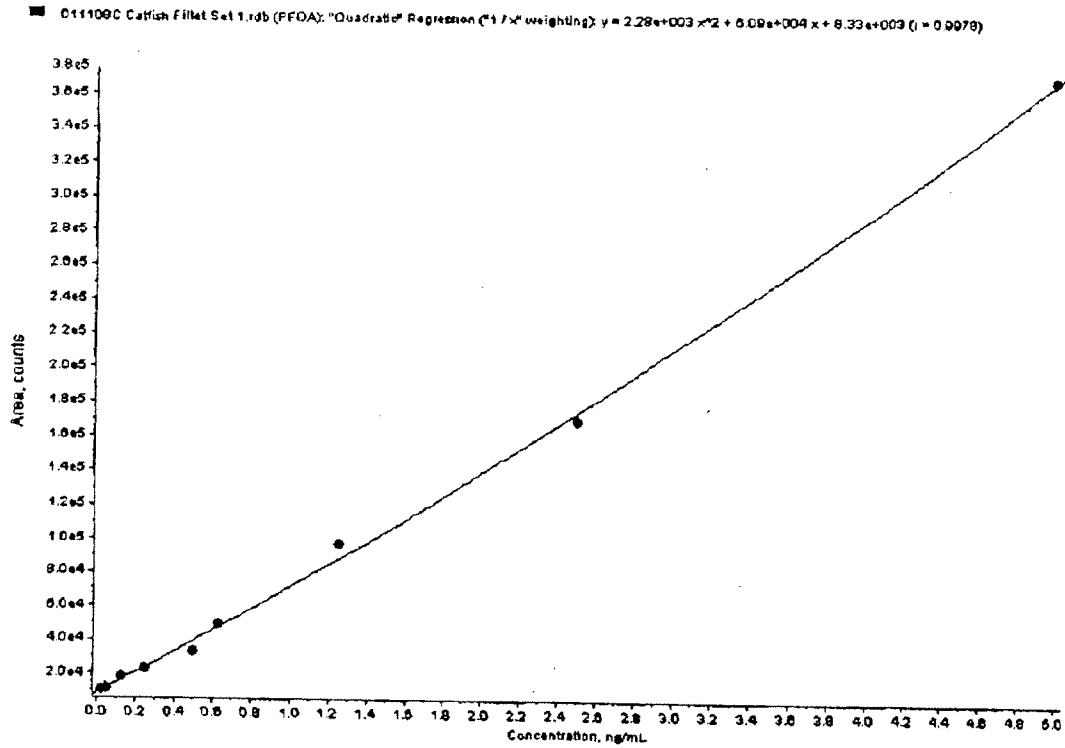
ND = Not detected at or above the acceptable LOQ reported in Table V.

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

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

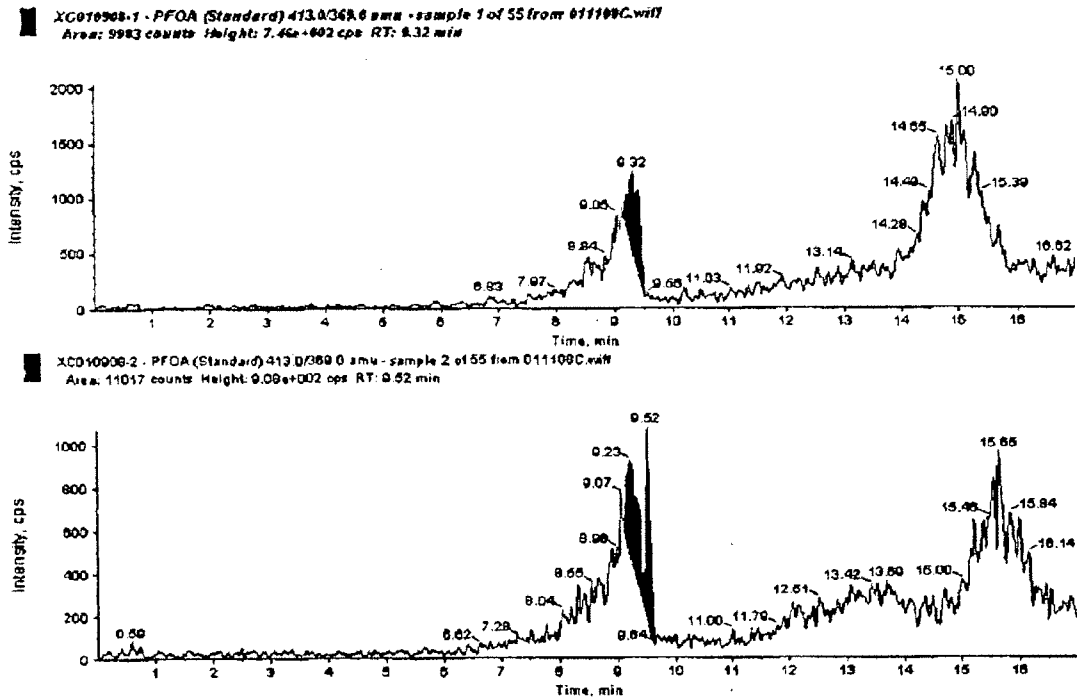
# FIGURES

**Figure 1. Typical Extracted Calibration Curve for PFOA in Catfish Fillet Matrix**

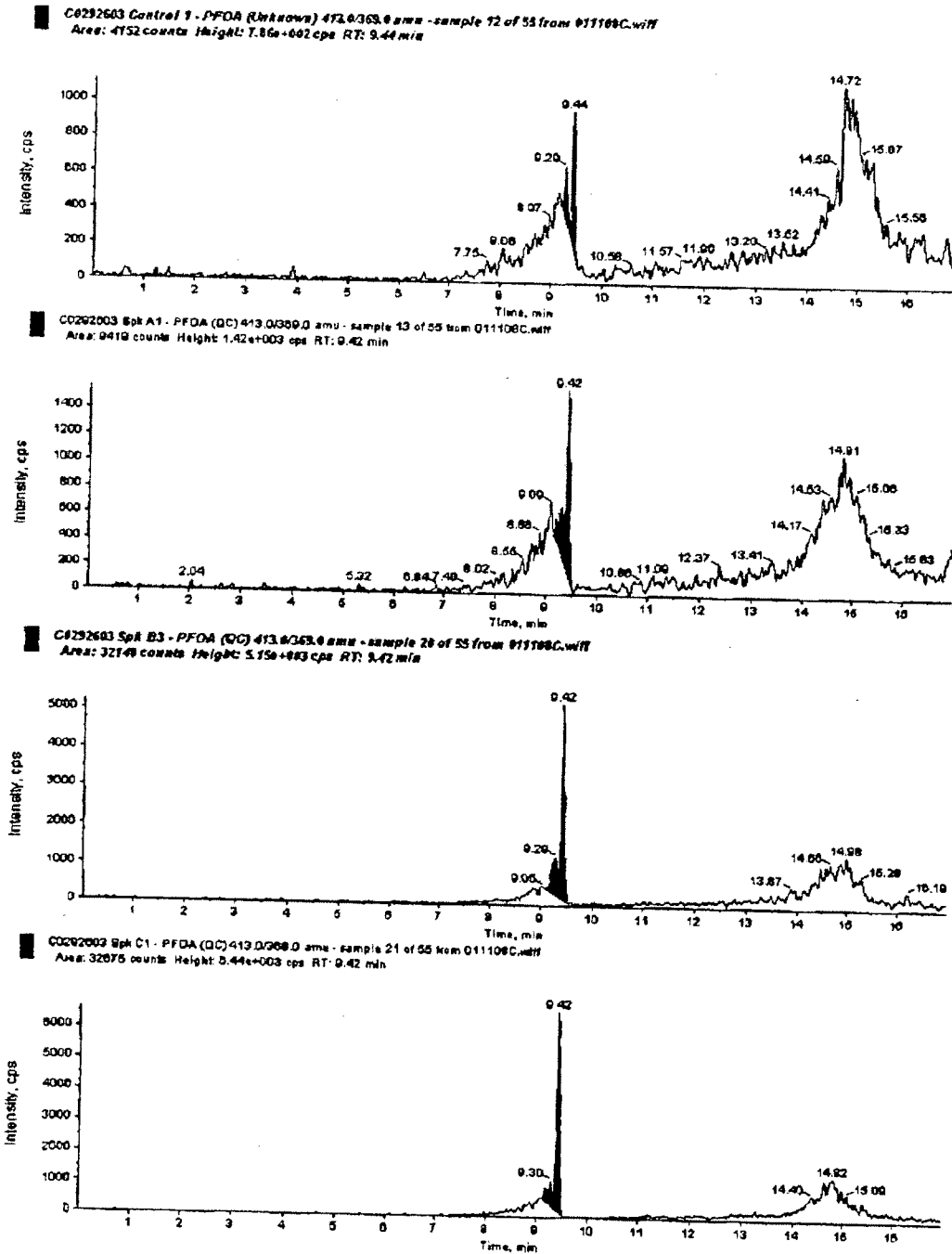




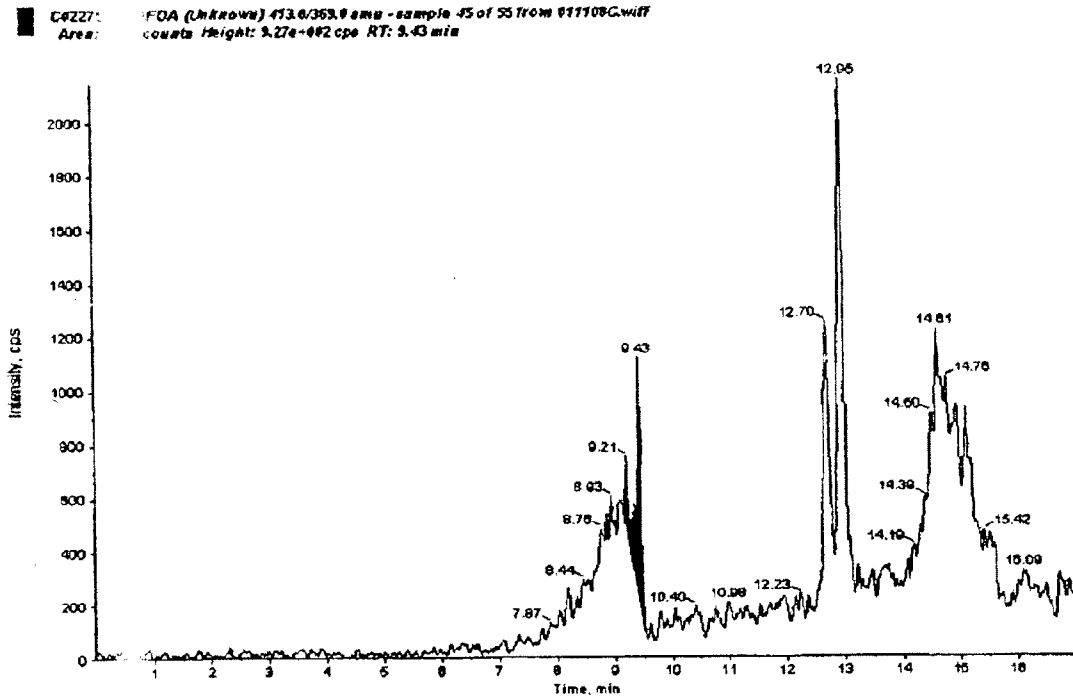
**Figure 2. Extracted Standards of PFOA in Catfish Fillet Matrix, 0.025 ng/mL (0.10 ng/g) and 0.050 ng/mL (0.20 ng/g), Respectively**



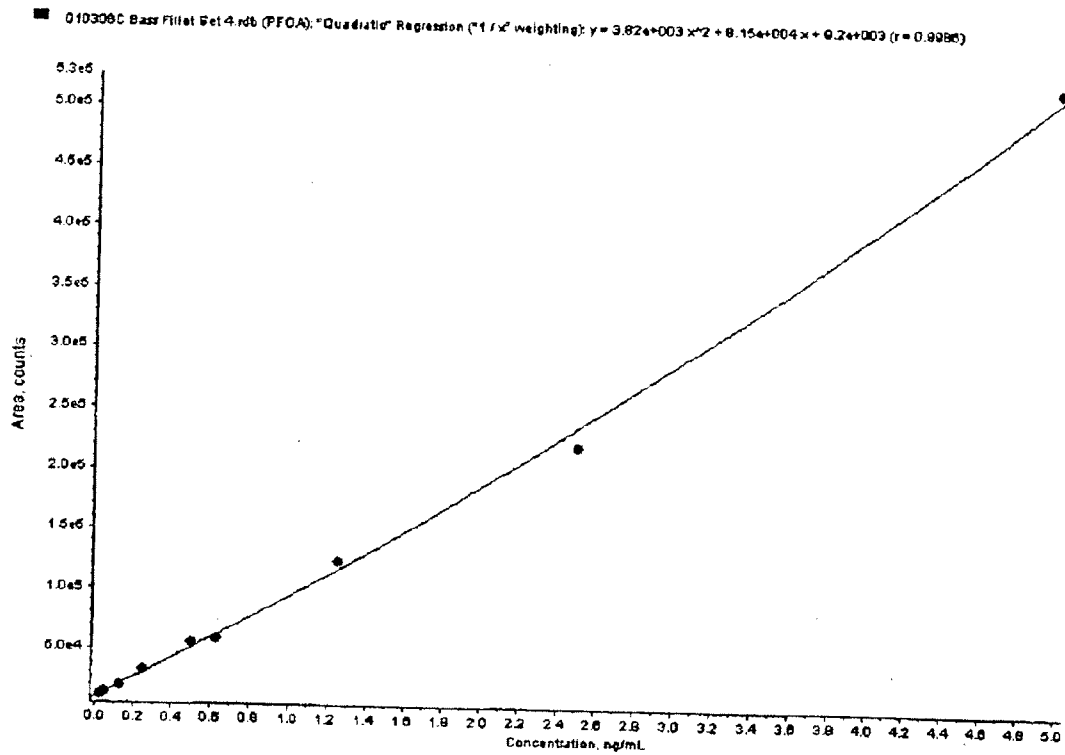
**Figure 3. PFOA in a Control Catfish Fillet, a 0.075 ng/mL (0.30 ng/g) Fortified Catfish Fillet Spike A, a 0.50 ng/mL (2.0 ng/g) Fortified Catfish Fillet Spike B, and a 4.0 ng/mL (16 ng/g) Fortified Catfish Fillet Spike C, Respectively**



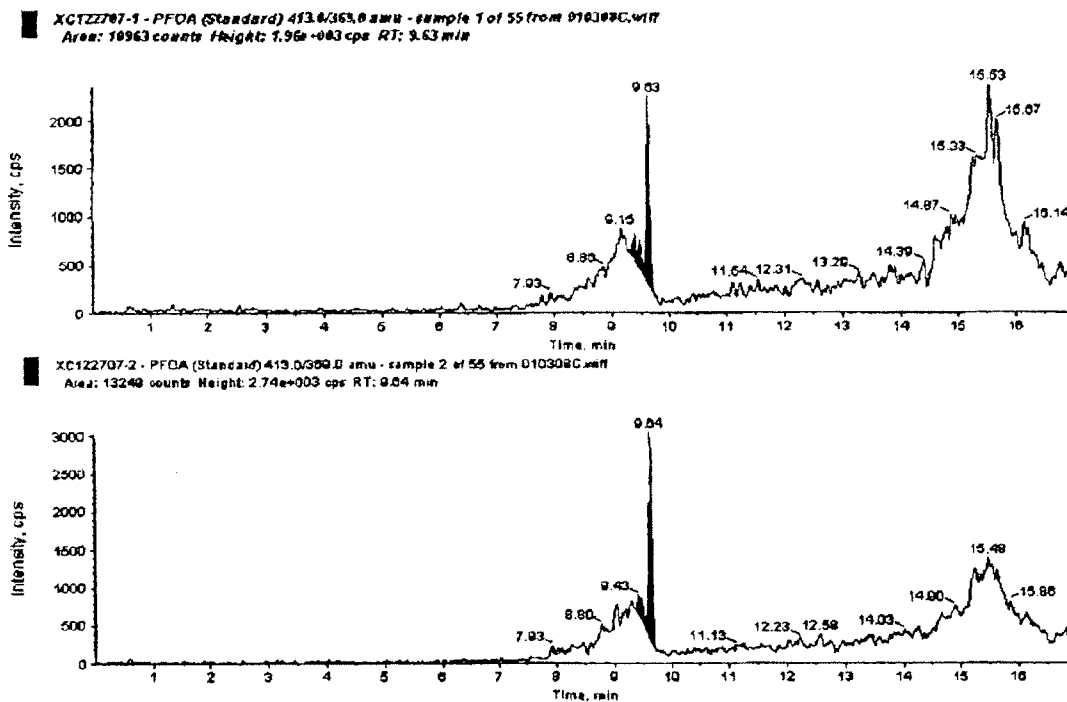
**Figure** Chromatogram Representing a Catfish Fillet Sample Analyzed for PFOA (ExyLIMS ID: C0227553, Data Set: 011108C)



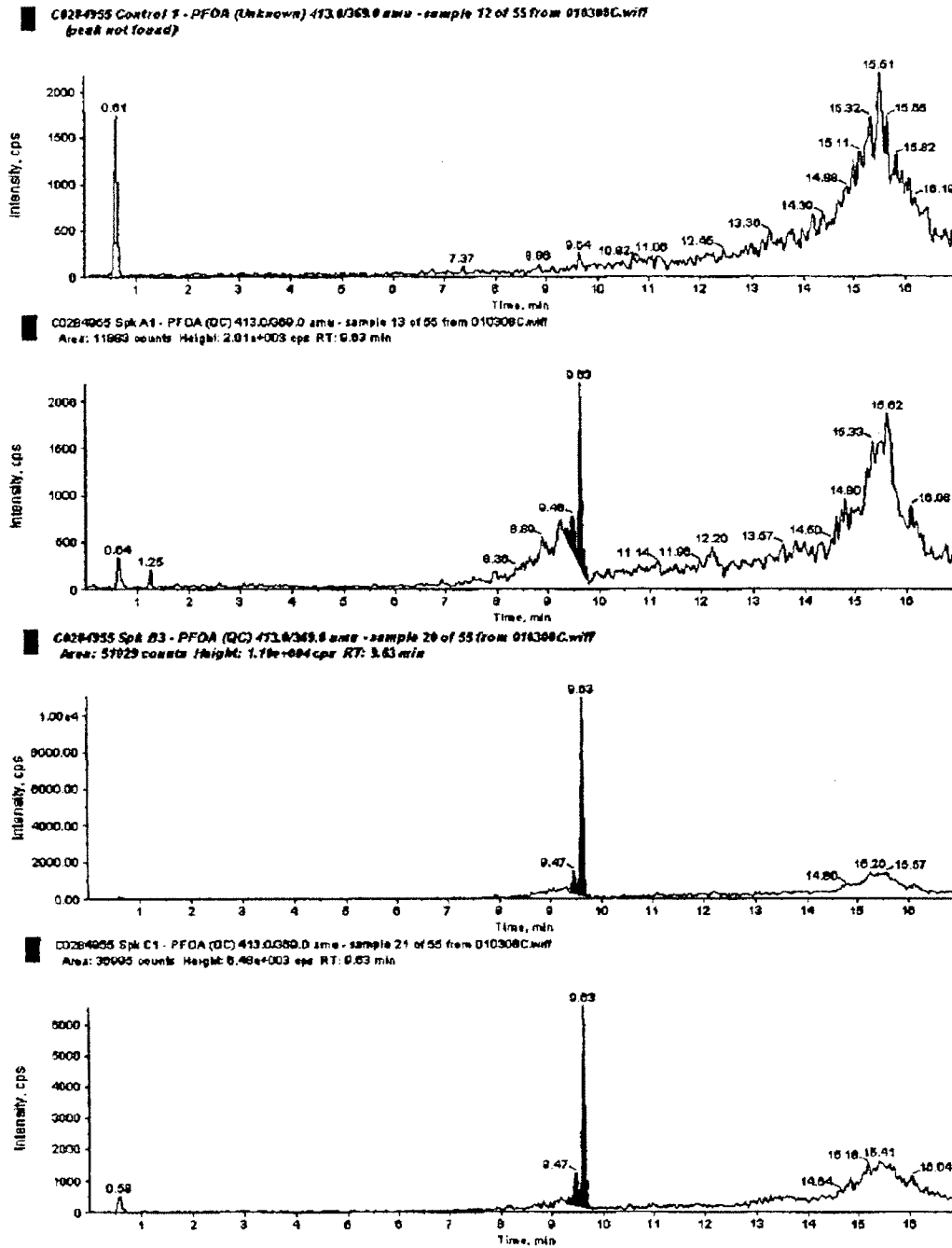
**Figure 5. Typical Extracted Calibration Curve for PFOA in Bass Fillet Matrix**



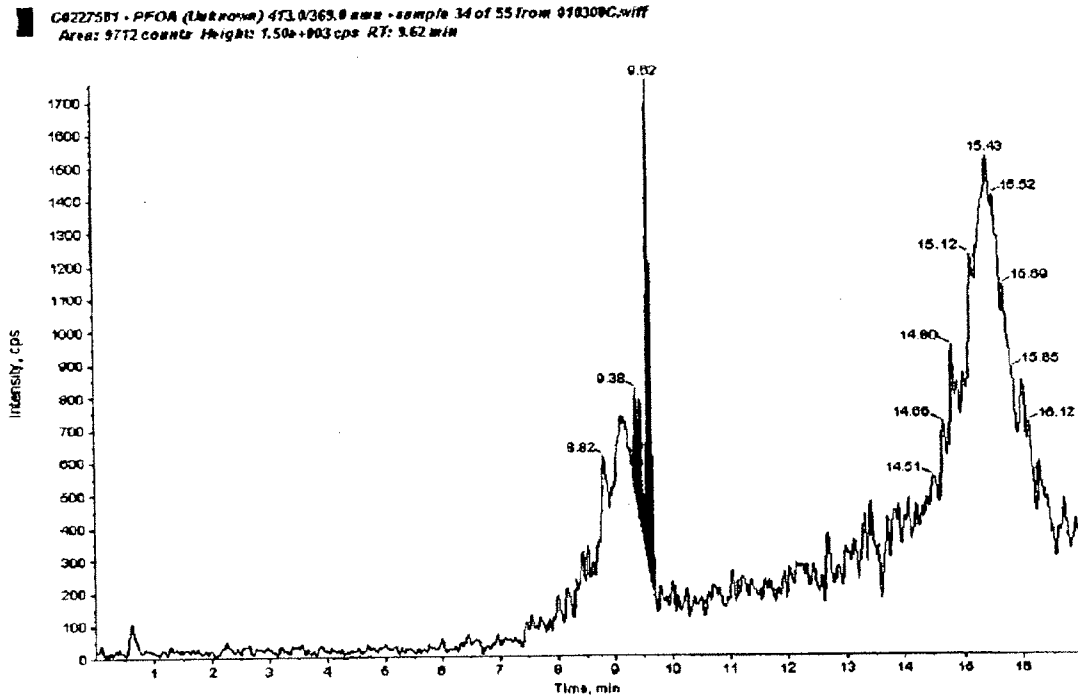
**Figure 6. Extracted Standards of PFOA in Bass Fillet Matrix, 0.025 ng/mL (0.10 ng/g) and 0.050 ng/mL (0.20 ng/g), Respectively**



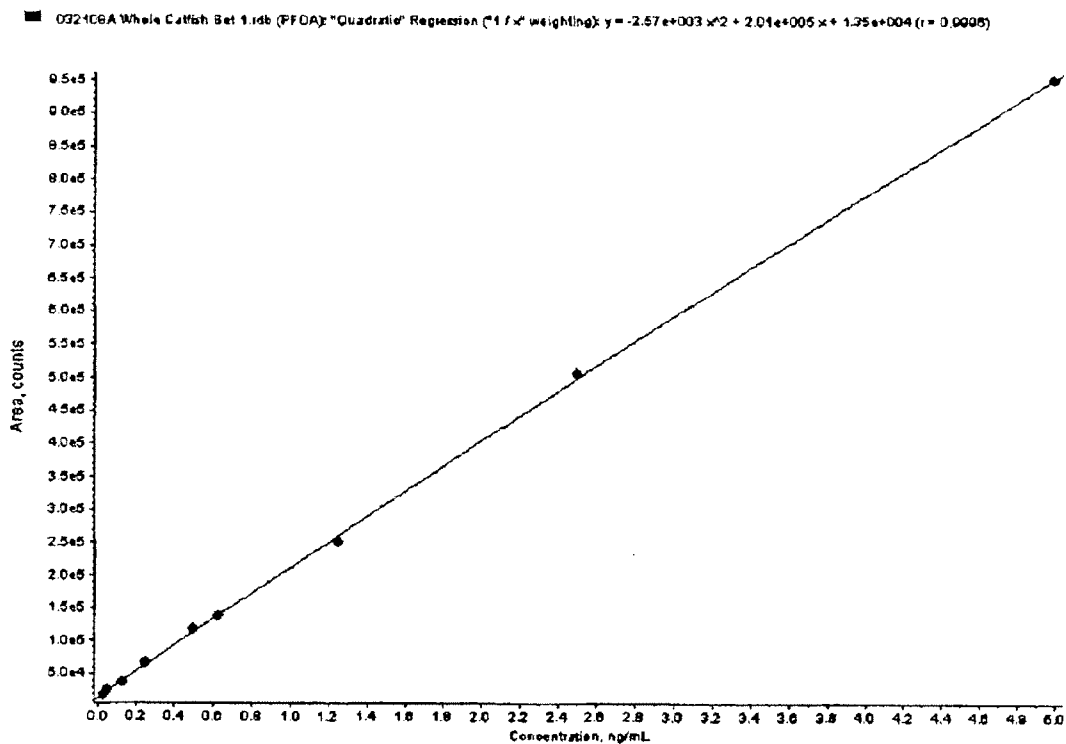
**Figure 7. PFOA in a Control Bass Fillet, a 0.075 ng/mL (0.30 ng/g) Fortified Bass Fillet Spike A, a 0.50 ng/mL (2.0 ng/g) Fortified Bass Fillet Spike B, and a 4.0 ng/mL (16 ng/g) Fortified Bass Fillet Spike C, Respectively**



**Figure 8. Chromatogram Representing a Bass Fillet Sample  
Analyzed for PFOA (ExyLIMS ID: C0227581, Data Set:  
010308C)**



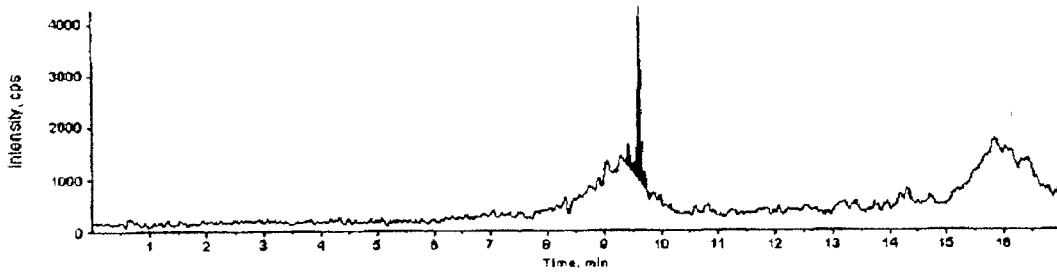
**Figure 9. Typical Extracted Calibration Curve for PFOA in Whole Body Catfish Matrix**



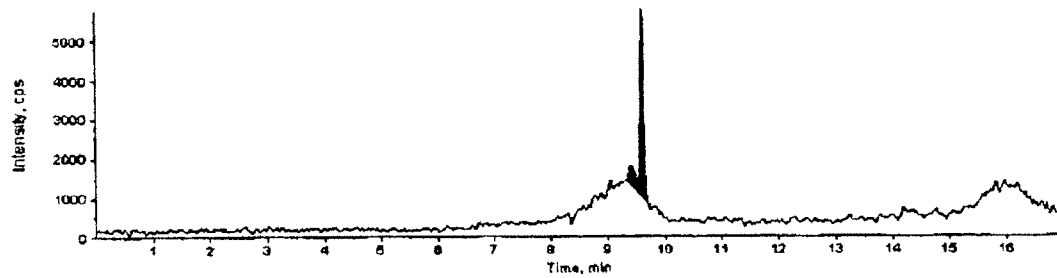


**Figure 10. Extracted Standards of PFOA in Whole Body Catfish Matrix, 0.025 ng/mL (0.10 ng/g) and 0.050 ng/mL (0.20 ng/g), Respectively**

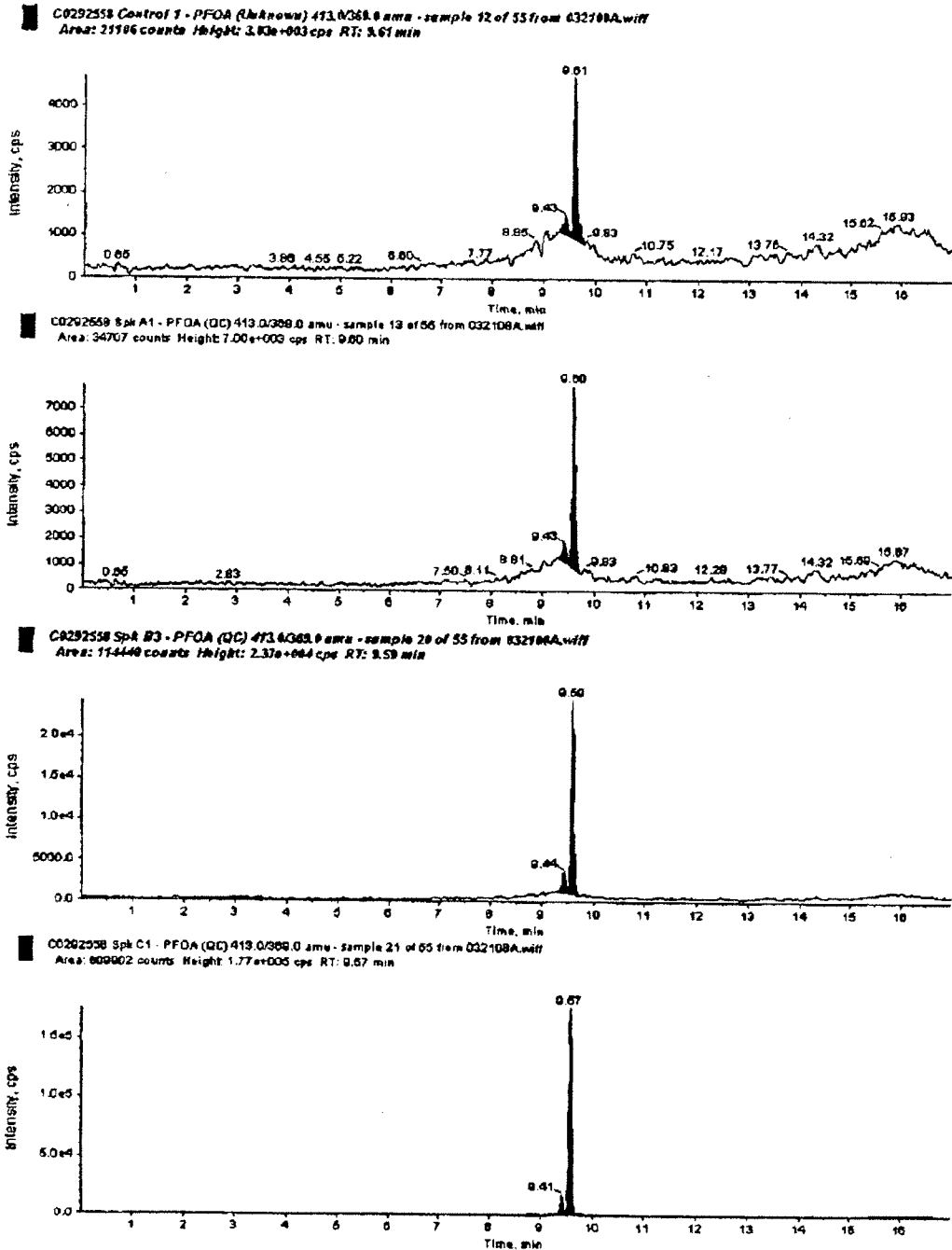
XG032109-1 - PFOA (Standard) 413.0369.6 amu - sample 1 of 55 from 032109A.wiff  
Area: 17684 counts Height: 3.38e+003 cps RT: 9.62 min



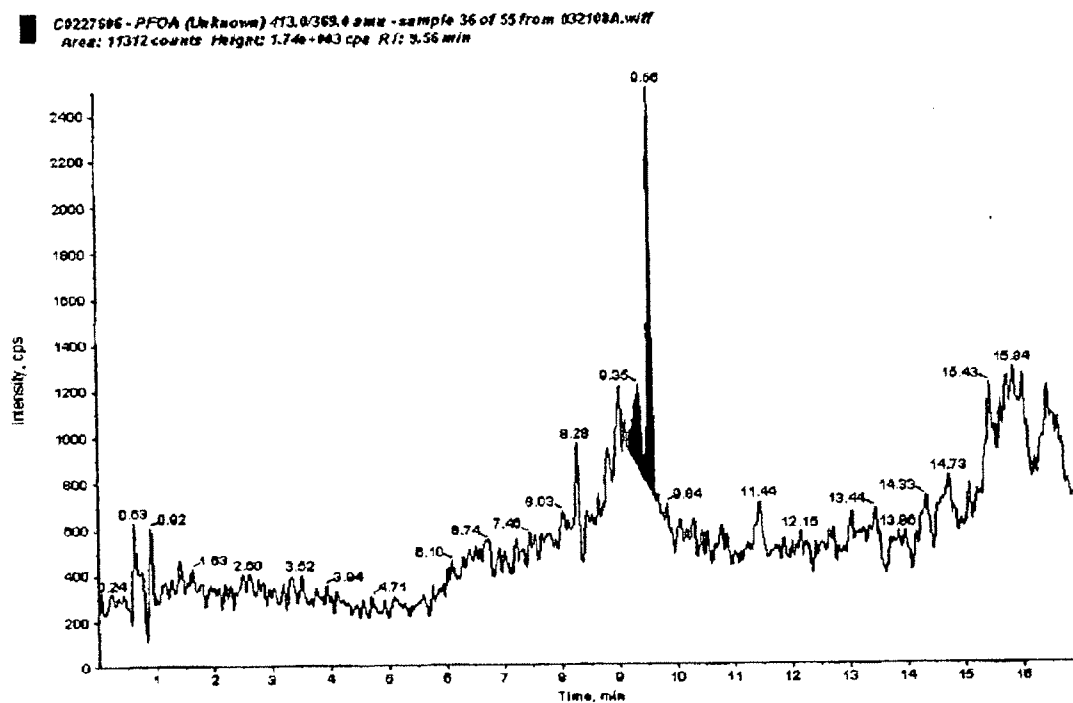
XG032109-2 - PFOA (Standard) 413.0369.0 amu - sample 2 of 65 from 032109A.wiff  
Area: 26409 counts Height: 4.70e+003 cps RT: 9.60 min



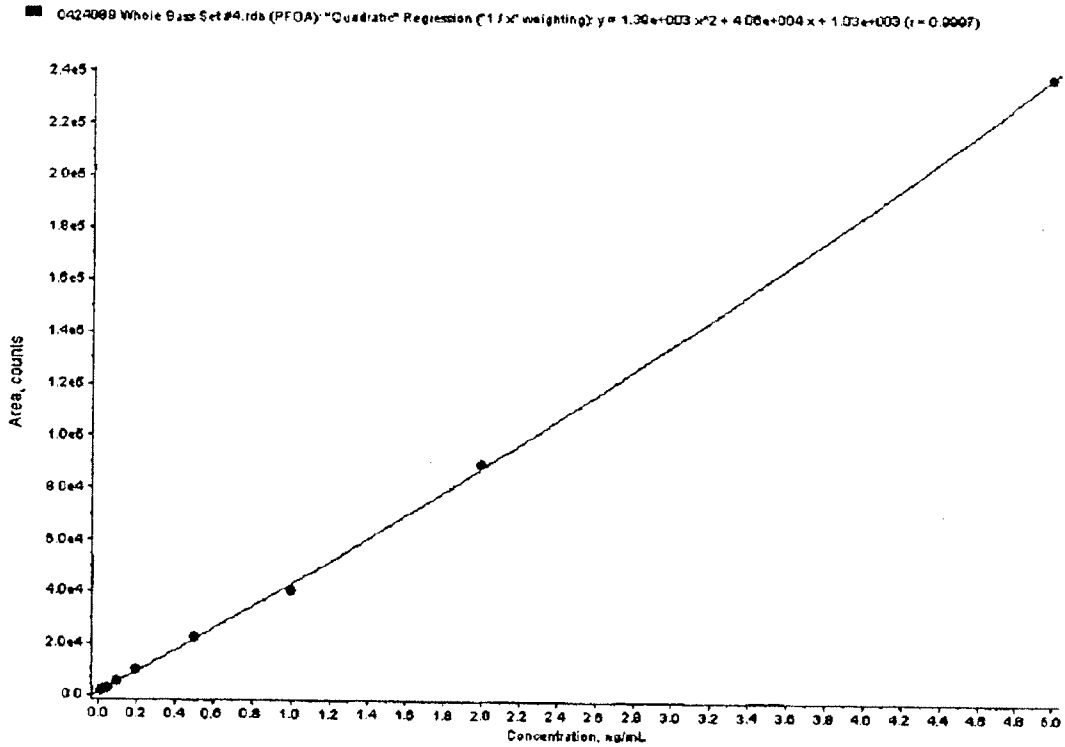
**Figure 11. PFOA in a Control Whole Body Catfish, a 0.075 ng/mL (0.30 ng/g) Fortified Whole Body Catfish Spike A, a 0.50 ng/mL (2.0 ng/g) Fortified Whole Body Catfish Spike B, and a 4.0 ng/mL (16 ng/g) Fortified Whole Body Catfish Spike C, Respectively**



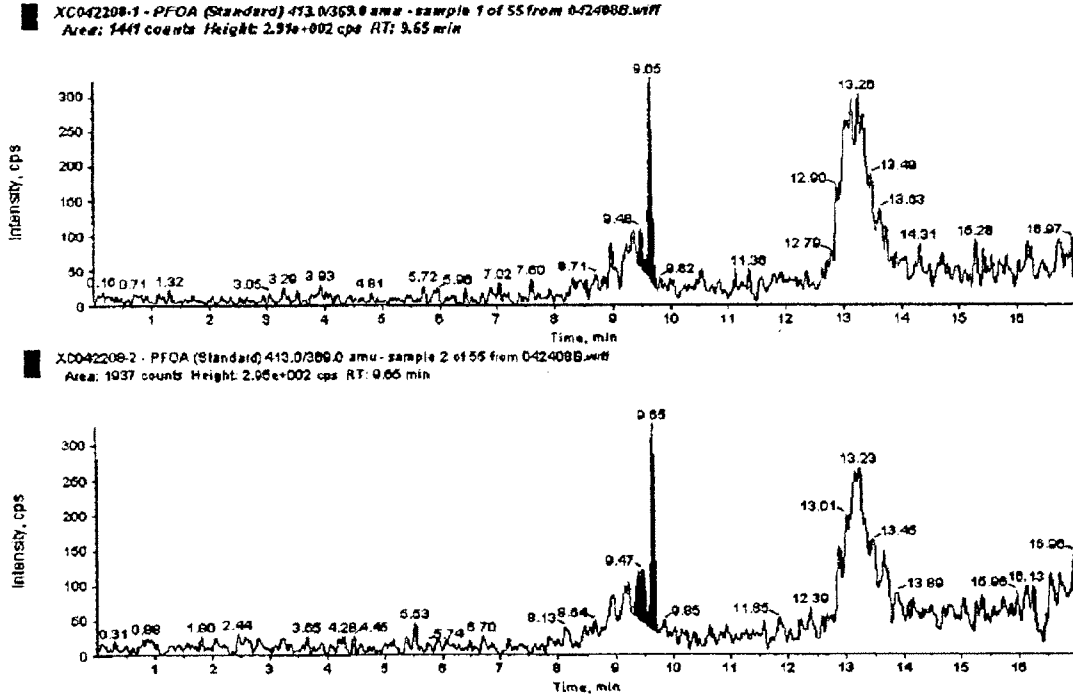
**Figure 12. Chromatogram Representing a Whole Body Catfish Sample Analyzed for PFOA (ExyLIMS ID: C0227606, Data Set: 032108A)**



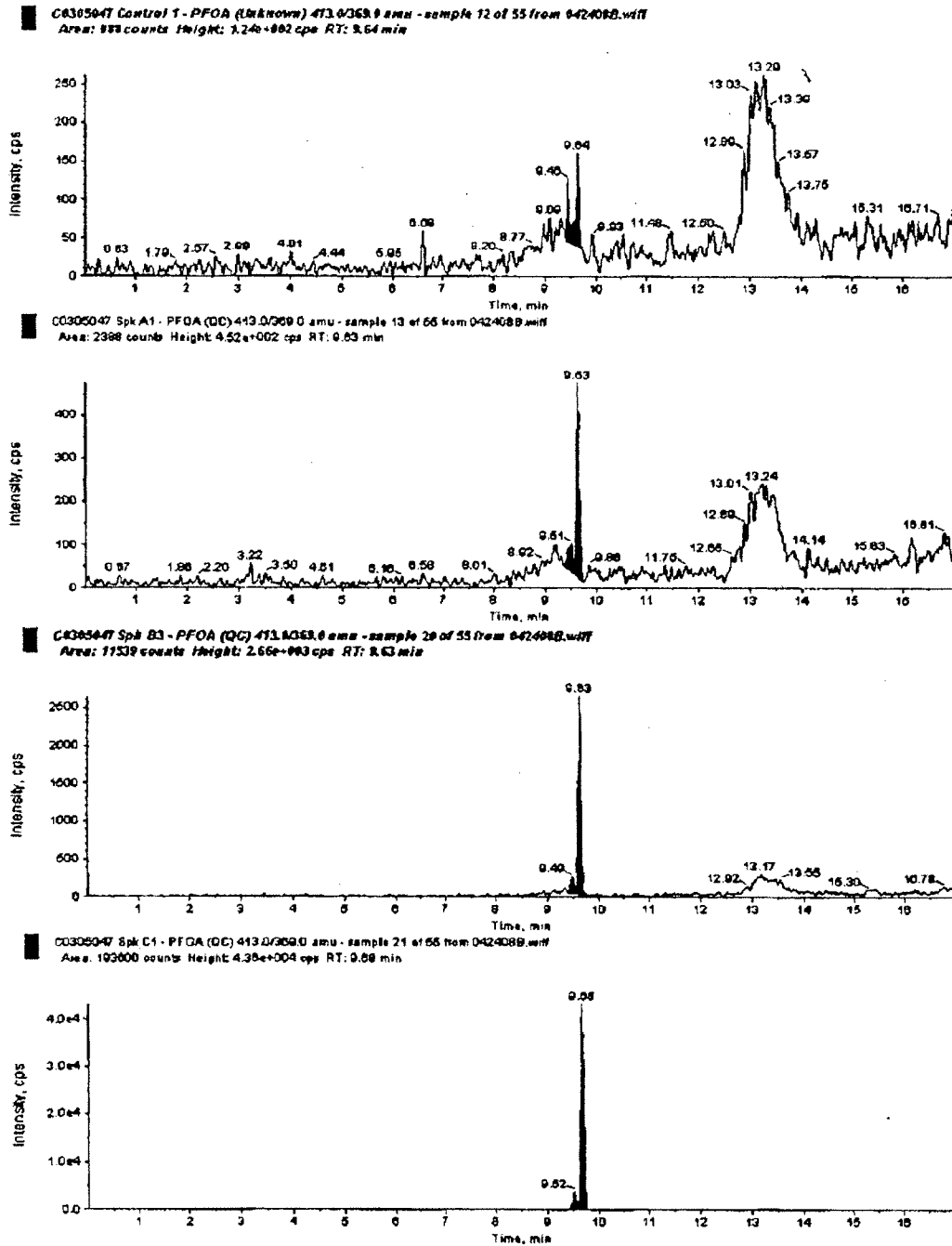
**Figure 13. Typical Extracted Calibration Curve for PFOA in Whole Body Bass Matrix**



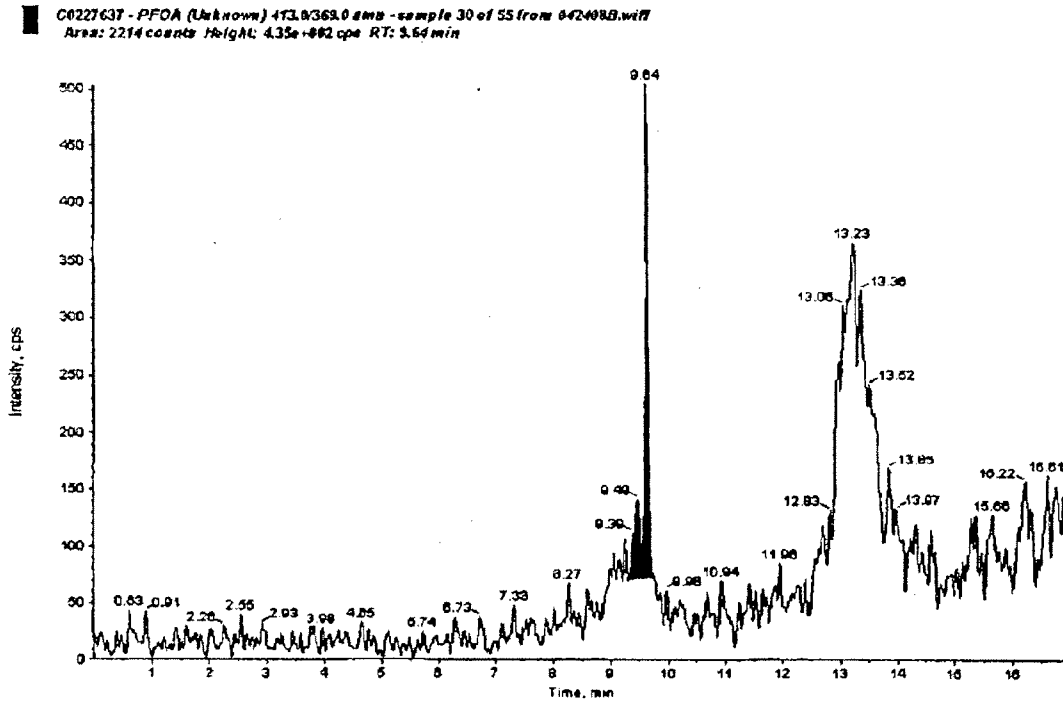
**Figure 14. Extracted Standards of PFOA in Whole Body Bass Matrix, 0.01 ng/mL (0.10 ng/g) and 0.02 ng/mL (0.20 ng/g), Respectively**



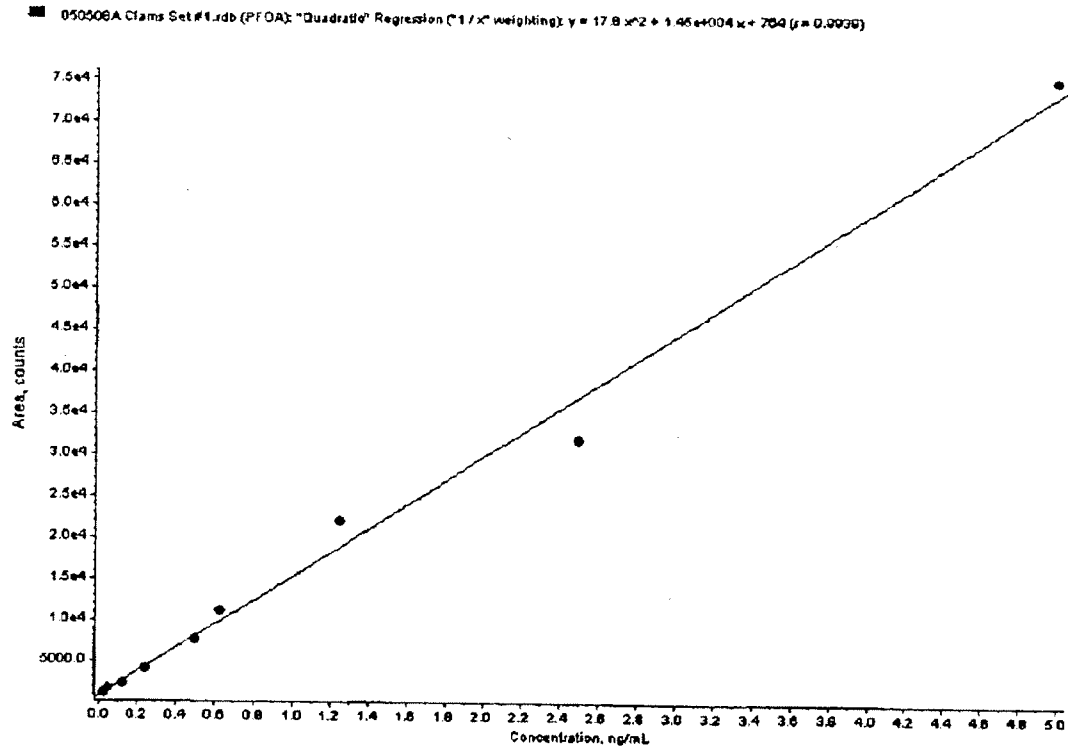
**Figure 15. PFOA in a Control Whole Body Bass, a 0.03 ng/mL (0.30 ng/g) Fortified Whole Body Bass Spike A, a 0.2 ng/mL (2.0 ng/g) Fortified Whole Body Bass Spike B, and a 4.0 ng/mL (40 ng/g) Fortified Whole Body Bass Spike C, Respectively**



**Figure 16. Chromatogram Representing a Whole Body Bass Sample Analyzed for PFOA (ExyLIMS ID: C0227637, Data Set: 042408B)**

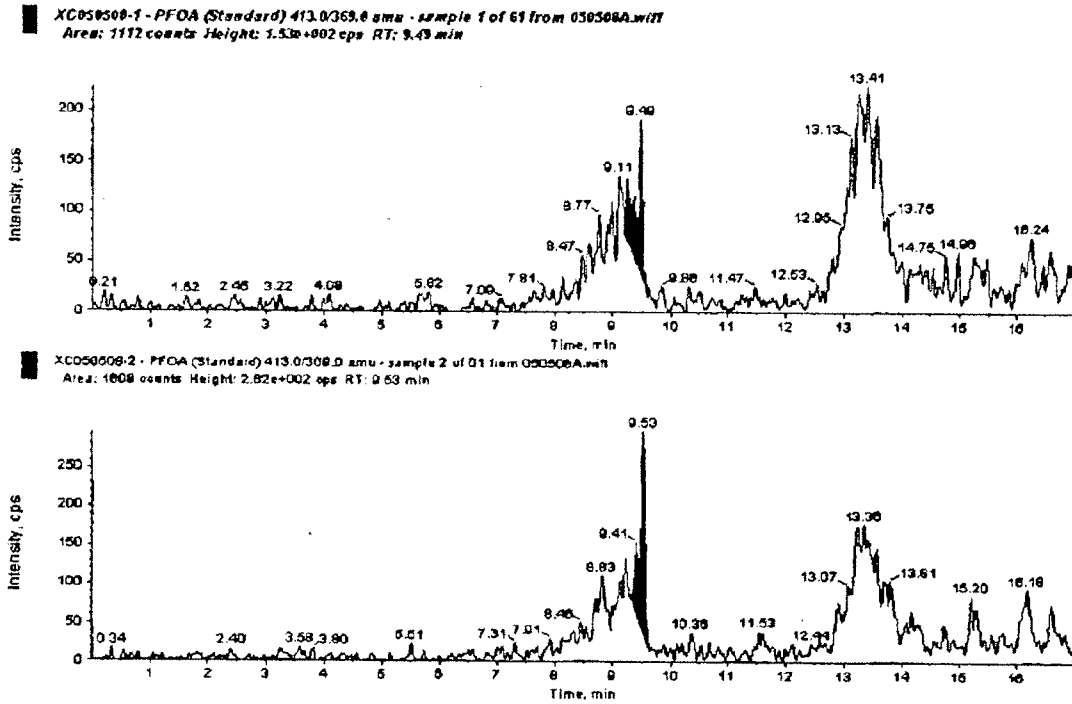


**Figure 17. Typical Extracted Calibration Curve for PFOA in Clam Matrix**

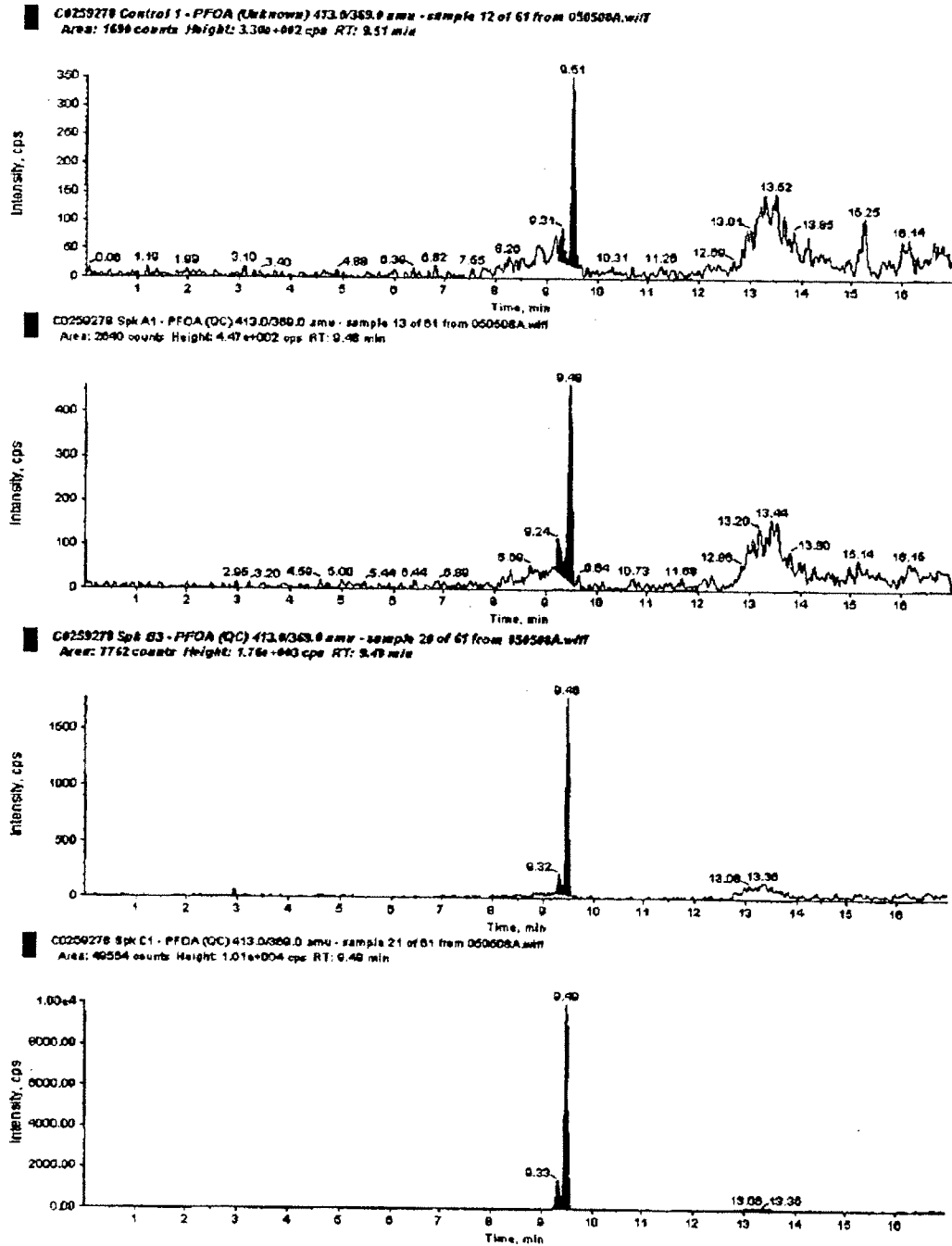




**Figure 18. Extracted Standards of PFOA in Clam Matrix, 0.025 ng/mL (0.10 ng/g) and 0.050 ng/mL (0.20 ng/g), Respectively**

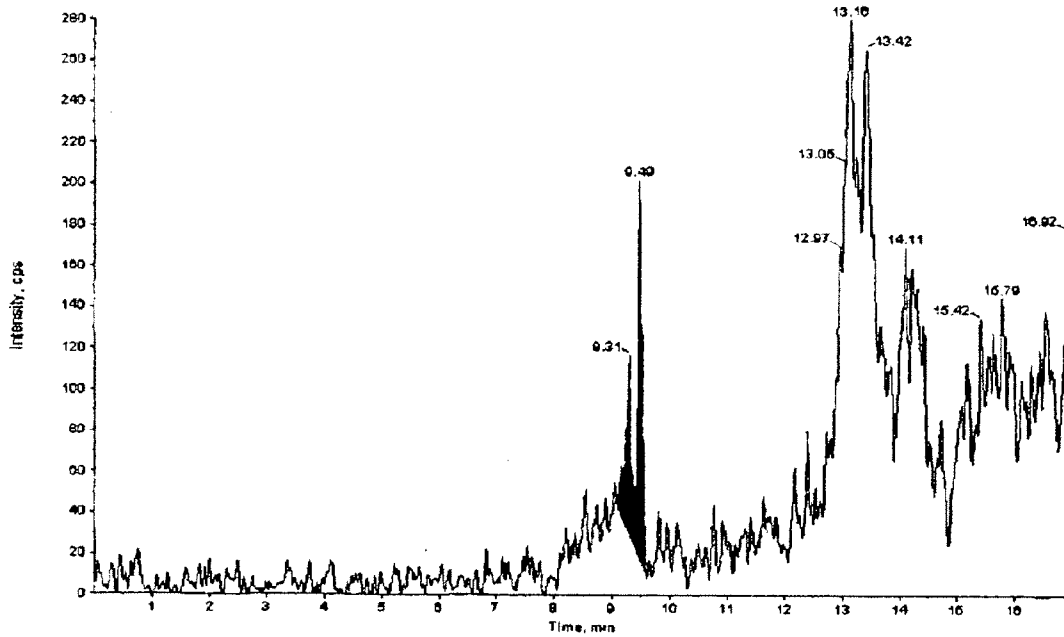


**Figure 19. PFOA in a Control Clam, a 0.075 ng/mL (0.30 ng/g) Fortified Clam Spike A, a 0.50 ng/mL (2.0 ng/g) Fortified Clam Spike B, and a 4.0 ng/mL (16 ng/g) Fortified Clam Spike C, Respectively**



**Figure 20. Chromatogram Representing a Clam Sample Analyzed for PFOA (ExyLIMS ID: C0227658, Data Set: 050508A)**

■ C0227658 - PFOA (Unknown) 413.0369.0 amu - sample 38 of 51 from 050508A.wiff  
Area: 1561 counts Height: 1.86e+002 cps RT: 9.49 min





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**ATTACHMENT 2**

**GROUNDWATER SAMPLING  
TECHNICAL PROGRESS REPORT**

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**August 2008**



**GROUNDWATER SAMPLING  
TECHNICAL PROGRESS REPORT  
3M AND DYNEON DECATUR, ALABAMA**

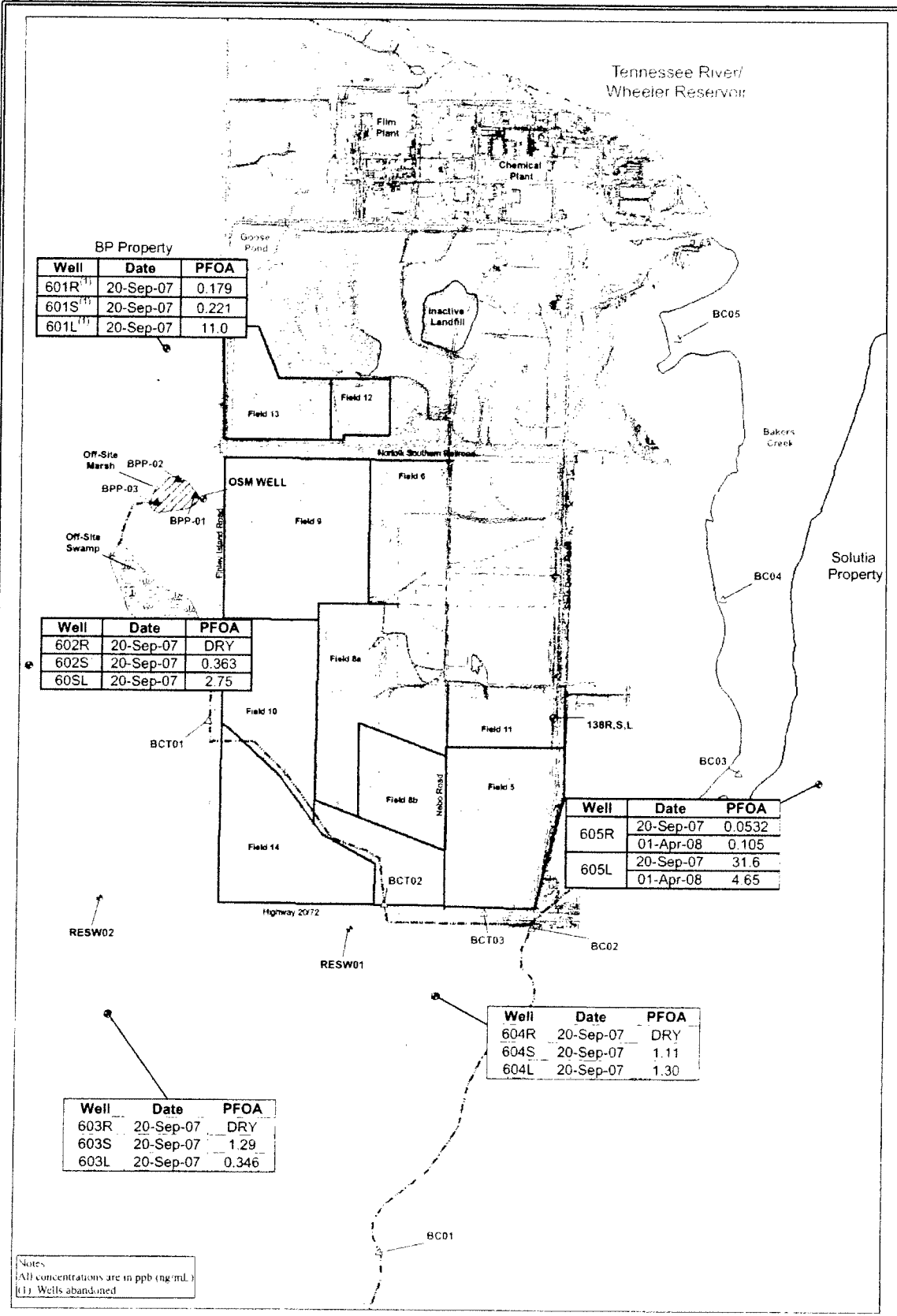
As part of the groundwater assessment associated with the former sludge incorporation area, 17 monitoring wells were constructed from November 2005 through April 2006 to evaluate groundwater conditions associated with the perimeter of the property. The locations include two well clusters located west of the former sludge incorporation area (monitoring wells 601R/601S/601L and 602R/602S/602L), two well clusters south of the former sludge incorporation area (monitoring wells 603R/603S/603L and 601R/601S/601L), one well cluster at the east side of the former sludge incorporation area (monitoring wells 138R/138S/138L in Field 11), and one well cluster on the east side of Bakers Creek (monitoring wells 605R/605L).

Initial sampling and analysis of groundwater for PFOA was performed in April 2006 and reported in the October 25, 2006 *Quarterly Status Report* and the 2008 *Data Assessment Report*. A second round of groundwater sampling of the off-site monitoring well clusters 601 through 605 was performed in September 2006 prior to the abandonment of the 601R/601S/601L wells before commencement of construction activities at that location. In addition, groundwater samples were collected from the 605R and 605L wells in April 2008. Analytical results for the September 2007 and April 2008 sampling rounds are tabulated in Table 1 and shown in Figure 1. The groundwater laboratory data packages for PFOA (Interim Reports 4 and 5) are provided in an appendix to this attachment.



**Table 1 Off-Site Groundwater PFOA Concentrations  
September 2007/April 2008**

Sample ID	Well	Sample Date	Average PFOA (ng/mL, ppb)
DAL-GW-601R-0-070920	601R	20-Sep-07	0.179
DAL-GW-601S-0-070920	601S	20-Sep-07	0.221
DAL-GW-601L-0-070920	601L	20-Sep-07	11.0
DAL-GW-602S-0-070920	602S	20-Sep-07	0.363
DAL-GW-602L-0-070920	602L	20-Sep-07	2.75
DAL-GW-603S-0-070920	603S	20-Sep-07	1.29
DAL-GW-603L-0-070920	603L	20-Sep-07	0.346
DAL-GW604S-0-070920	604S	20-Sep-07	1.11
DAL-GW-604L-0-070920	604L	20-Sep-07	1.30
DAL-GW-605R-0-070920	605R	20-Sep-07	0.0532
DAL-GW-605R-0-080401		01-Apr-08	0.105
DAL-GW-605L-0-070920	605L	20-Sep-07	31.6
DAL-GW-605L-0-080401		01-Apr-08	4.65



**BP Property**

Well	Date	PFOA
601R <sup>(1)</sup>	20-Sep-07	0.179
601S <sup>(1)</sup>	20-Sep-07	0.221
601L <sup>(1)</sup>	20-Sep-07	11.0

Well	Date	PFOA
602R	20-Sep-07	DRY
602S	20-Sep-07	0.363
602L	20-Sep-07	2.75

Well	Date	PFOA
605R	20-Sep-07	0.0532
	01-Apr-08	0.105
605L	20-Sep-07	31.6
	01-Apr-08	4.65

Well	Date	PFOA
604R	20-Sep-07	DRY
604S	20-Sep-07	1.11
604L	20-Sep-07	1.30

Well	Date	PFOA
603R	20-Sep-07	DRY
603S	20-Sep-07	1.29
603L	20-Sep-07	0.346

Notes:  
 (All concentrations are in ppb (ng/mL).)  
 (1) Wells abandoned

**Legend**

- Monitoring Well Locations
  - R - Residuum Well
  - S - Epikarst Well
  - L - Bedrock Well
- Daikin Property
- Surface Water Sediment
- Surface Water
- Residential Well

WESTON CONSULTANTS

**Figure 1**  
 Off-Site Groundwater PFOA Concentrations  
 September 2007/April 2008  
 3M Decatur, AL Facility