

**INTERIM REPORT #22 - Analysis of Soil Samples**

**STUDY TITLE**

Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, Clams,  
Vegetation, Small Mammal Liver and Small Mammal Serum 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**

September 22, 2006

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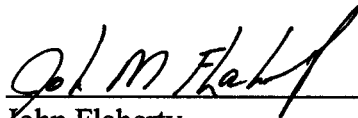
**PROJECT**

Protocol Number: P0000760  
Exygen Study Number: P0000760

Total Pages: 151

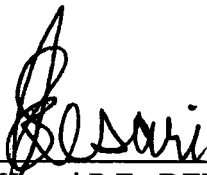
**GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT**

Exygen Study Number P0000760, entitled "Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, Clams, Vegetation, Small Mammal Liver and Small Mammal Serum 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 Exygen Research.



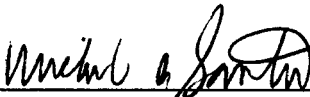
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Principal Investigator  
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9/22/06  
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Jaisimha Kesari P.E., DEE  
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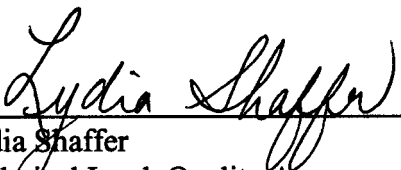
Michael A. Santoro  
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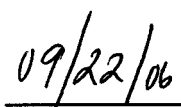
9/26/06  
Date

**QUALITY ASSURANCE STATEMENT**

Exygen Research's Quality Assurance Unit reviewed Exygen Study Number P0000760, entitled, "Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, Clams, Vegetation, Small Mammal Liver and Small Mammal Serum Using LC/MS/MS for the 3M Decatur Monitoring Program". All reviewed phases<sup>1</sup> 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> |
|----------------------------------------------------------|-----------------------|------------------------------------------------|-------------------------------------------|----------------------------------------|
| 32. Raw Data Review and Interim Analytical Report Review | 06/06-09/06           | 06/29/06                                       | 06/30/06                                  | 07/07/06                               |
| 33. Interim Analytical Report Review                     | 06/14/06              | 06/29/06                                       | 06/30/06                                  | 07/07/06                               |
| 34. Raw Data Review and Interim Analytical Report Review | 09/20-21/06           | 09/22/06                                       | 09/22/06                                  | 09/22/06                               |

  
Lydia Shaffer  
Technical Lead, Quality Assurance Unit

  
Date

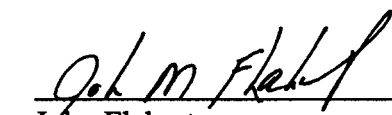
<sup>1</sup>Note: All in-lab inspections will be documented in the QA statement for the final analytical report at the conclusion of the study. This QA statement involves only the review of the interim report and associated raw data.

**CERTIFICATION OF AUTHENTICITY**

This interim report, for Exygen Study Number P0000760, is a true and complete representation of the raw data.

Submitted by: Exygen Research  
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\_\_\_\_\_  
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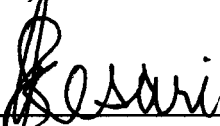
9/22/06  
\_\_\_\_\_  
Date

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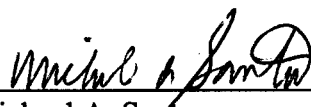
22-SEP-2006  
\_\_\_\_\_  
Date

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Director of Regulatory Affairs

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**STUDY IDENTIFICATION**

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

PROTOCOL NUMBER: P0000760

EXYGEN STUDY NUMBER: P0000760

TYPE OF STUDY: Residue

SAMPLE MATRIX: Soil and Water

TEST SUBSTANCE: Perfluorooctanoic acid (PFOA)

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PERFORMING LABORATORY: Exygen Research  
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ANALYTICAL PHASE  
TIMETABLE: Study Initiation Date: 11/05/04  
Interim Analytical Start Date: 04/15/06  
Interim Analytical Termination Date: 07/24/06  
Interim Report Completion Date: 09/22/06

**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:

| <u>Name</u>       | <u>Title</u>        |
|-------------------|---------------------|
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| Eric Edwards      | Sample Custodian    |
| Christine Edwards | Technician          |
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| Larissa Hoover    | Technician          |
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## 1.0 SUMMARY

Exygen Research extracted and analyzed soil samples for the determination of perfluorooctanoic acid (PFOA) according to Exygen Method V0001781 and water samples for the determination of perfluorooctanoic acid (PFOA) according to Exygen Method V0001780 (**Appendix A**).

Thirty-three of the analyzed soil samples were not reported (NR) due to quality control failure. Eight of the thirty-three samples failed because the PFOA spikes were too low relative to the endogenous level in the sample to allow assessment of matrix interference. Twenty-five of the thirty-three samples failed for unknown reasons. Additional analytical work was performed in an attempt to provide quantitative results for these thirty-three samples. Quantitative PFOA results were obtained for all samples except for nine soil samples that are not reported due to quality control failures.

The limit of quantitation for PFOA in soil was 0.2 ng/g. The LOQ provided is assuming wet weight values and the results for the actual samples are reported based on dry weight.

The limit of quantitation for PFOA in water was 25 ng/L.

Analytical results and assessed accuracies for the analysis of PFOA in soil samples based on dry weight are summarized in **Table I**. Analytical results and assessed accuracies for the analysis of PFOA in re-extracted soil samples based on dry weight are summarized in **Table II**. Analytical results and assessed accuracies for the analysis of PFOA in water samples are summarized in **Table III**. Field matrix spikes that were in the appropriate concentration range (primary concentration must be less than or equal to three times the spiking level) were used to calculate the assessed accuracies. When the field matrix spikes were not available or failed to meet recovery criteria, the laboratory spikes were used.

Fortification recoveries for the analysis of PFOA in soil samples based on wet weight are summarized in **Table IV**. The overall average percent recovery  $\pm$  standard deviation for PFOA in soil samples based on wet weight was  $80 \pm 19\%$ . Fortification recoveries for the analysis of PFOA in re-extracted soil samples based on wet weight are summarized in **Table V**. The overall average percent recovery  $\pm$  standard deviation for PFOA in re-extracted soil samples based on wet weight was  $74 \pm 18\%$ . Fortification recoveries for the analysis of PFOA in water samples are summarized in **Table VI**. The overall average percent recovery  $\pm$  standard deviation for PFOA in water samples was  $92 \pm 7\%$ .

Fortification recoveries for  $^{13}\text{C}$  PFOA in soil samples based on wet weight are summarized in **Table VII**. The average percent recovery  $\pm$  standard deviation for  $^{13}\text{C}$  PFOA in soil samples based on wet weight was  $91 \pm 32\%$ . Fortification recoveries for  $^{13}\text{C}$  PFOA in re-extracted soil samples based on wet weight are summarized in **Table VIII**. The average percent recovery  $\pm$  standard deviation for  $^{13}\text{C}$  PFOA in re-extracted soil

samples based on wet weight was  $48 \pm 30\%$ . Fortification recoveries for  $^{13}\text{C}$  PFOA in water samples are summarized in **Table IX**. The average percent recovery  $\pm$  standard deviation for  $^{13}\text{C}$  PFOA in water samples was  $107 \pm 14\%$ .

The total percent solids for soil samples are detailed in **Table X**.

## **2.0 OBJECTIVE**

The objective of the analytical part of this study was to determine levels of perfluorooctanoic acid (PFOA) in soil and water according to Protocol P0000760 (**Appendix A**).

## **3.0 INTRODUCTION**

This report details the results of the analysis for the determination of PFOA in soil using the analytical method entitled, "V0001781: Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS" and for the determination of PFOA 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 November 5, 2004, when the study director signed protocol number P0000760. The analytical start date for this interim report was April 15, 2006 and the analytical termination date for this interim report was July 24, 2006.

## **4.0 ANALYTICAL TEST SAMPLES**

Five water samples, representing one trip blank and its associated spikes and two rinse blanks (RB), and forty-seven soil samples (Exygen ID C0156344 – C0156395) were received on wet ice on March 2, 2006 from Tim Frinak at Weston Solutions, Inc. Six water samples, representing one trip blank and its associated spikes and three rinse blanks (RB), and twenty-three soil samples (Exygen ID C0159443 – C0159471) were received on wet ice on March 10, 2006 from Tim Frinak at Weston Solutions, Inc. Four water samples, representing one trip blank and its associated spikes and one rinse blank (RB), and thirty-four soil samples (Exygen ID C0160867 – C0160904) were received on wet ice on March 15, 2006 from Tim Frinak at Weston Solutions, Inc. Four water samples, representing one trip blank and its associated spikes and one rinse blank (RB), and thirteen soil samples (Exygen ID C0164222 – C0164260) were received on wet ice on March 24, 2006 from Tim Frinak at Weston Solutions, Inc. Please note that rinse blank (RB) samples do not have associated field spikes or laboratory spikes. The samples were logged in by Exygen personnel and placed in refrigerated storage.

The soil samples collected represent both soil boring samples and surface soil samples. Sample ID coding for the soil samples consisted of five sections or strings in the following format:

Dxxx-xx-xxxxxx-x(x)-xxxx.

The first string of code, for the soil boring samples, contains DAL, which represents Decatur, Alabama. For the surface soil samples, the first string begins with D for Decatur, Alabama and the xxx defines the sampling location within the study area where:

DF05 = Field 5

DF10 = Field 10

DF11 = Field 11

DF12 = Field 12

DF13 = Field 13

The second string defines the type of sample where:

SB = soil boring sample

SS = surface soil sample

The third string for the soil boring samples defines the soil boring identification. For the surface soil sample, the third string defines the location within the sampling area.

The fourth string more specifically defines the sample type where:

0 = primary field sample

DB = field duplicate sample

RB = equipment rinseate blank sample

The fifth string defines the collection depth of the soil samples where:

0000 = 0 to 3 inches

0003 = 3 to 6 inches

0005 = 6 inches to 1 foot

0010 = 1 to 1.5 feet

0050 = 5 to 5.5 feet

0100 = 10 to 11 feet

Sample login 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 (PFOA SP0005444), was purchased from Oakwood Products, Inc. and was received at Exygen on March 17, 2005. The analytical standard, PFOA (PFOA SP0007768), was received at Exygen on June 13, 2006 from the 3M Company. The surrogate spiking standard, <sup>13</sup>C labeled perfluorooctanoic acid (<sup>13</sup>C PFOA SP0004184), was received at Exygen on April 15, 2004 from the 3M Company. The surrogate spiking standard, <sup>13</sup>C labeled perfluorooctanoic acid (<sup>13</sup>C PFOA SP0006528), was received at Exygen on October 13, 2005 from the 3M Company. The surrogate

spiking standard,  $^{13}\text{C}$  labeled perfluorooctanoic acid ( $^{13}\text{C}$  PFOA SP0007767), was received at Exygen on June 13, 2006 from the 3M Company.

The available information for the reference materials is listed below. PFOA was stored ambient and  $^{13}\text{C}$  PFOA was stored frozen.

| <u>Compound</u>      | <u>Exygen Inventory</u><br><u>No.</u> | <u>Lot #</u> | <u>Purity (%)</u> | <u>Expiration</u><br><u>Date</u> |
|----------------------|---------------------------------------|--------------|-------------------|----------------------------------|
| PFOA                 | SP0005444                             | 203          | 99                | 03/31/07                         |
| PFOA                 | SP0007768                             | HU14401DU    | 96.7              | 12/15/06                         |
| $^{13}\text{C}$ PFOA | SP0006528                             | 3507-195     | 97                | 03/29/09                         |
| $^{13}\text{C}$ PFOA | SP0004184                             | 3507-195     | 97                | 03/29/09                         |
| $^{13}\text{C}$ PFOA | SP0007767                             | 3507-277     | 97.6              | 07/15/10                         |

The molecular structure of PFOA is given below:

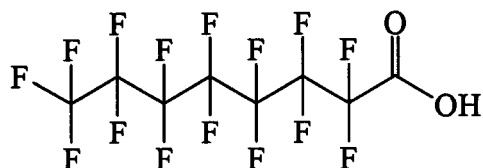
**PFOA**

Chemical Name: Perfluorooctanoic acid

Molecular Weight: 414

Transitions Monitored: 413  $\rightarrow$  369 (for quantification) and  
413  $\rightarrow$  219 (for confirmation)

Structure:

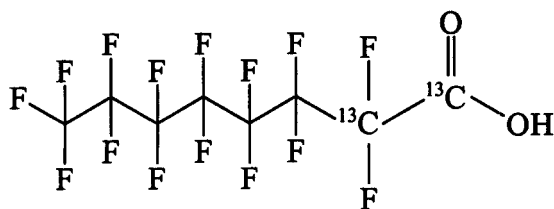
 **$^{13}\text{C}$  PFOA**

Chemical Name: 1,2- $^{13}\text{C}$  perfluorooctanoic acid

Molecular Weight: 416

Transition Monitored: 415  $\rightarrow$  370

Structure:



## **6.0 DESCRIPTION OF ANALYTICAL METHOD**

The analytical methods “V0001781: Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS” and “V0001780: Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS” were used for this study.

### **6.1 Extraction Procedure For Soil**

Before the samples were weighed for the extraction, they were placed into a new, clean Ziploc bag and mixed thoroughly. The samples were then transferred back to the sampling container. A 5-gram portion of soil was weighed into a fifty-milliliter centrifuge tube for the extraction. After fortification of appropriate samples, 5 mL of methanol was added to the samples. The samples were allowed to shake on a wrist action shaker for ~15 minutes and were then sonicated in an ultrasonic bath for ~15 minutes. The volume was taken to 40 mL with water and the samples were then centrifuged for ~10 minutes at ~3000 rpm. The supernatant was then loaded onto a C<sub>18</sub> 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 Percent Solids Procedure For Soil**

Percent solids were determined using the procedure indicated in Exygen method V0000427. Approximately 20 grams of sample was weighed into a pan. The weight of the sample plus the pan was recorded. The samples were then dried in an oven overnight at  $104 \pm 2$  °C. Then the samples were transferred to a dessicator and allowed to cool for ~15 minutes. Each sample was then weighed again, including the weight of the pan. The percent solid for each sample was then calculated. See **Table X** for percent solids results.

One soil sample, C00159446, was weighed at ~8g for percent solid analysis, rather than at ~20g which is stated in the method. See deviation 6. The percent solid calculation was altered to reflect this change in mass.

### **6.3 Extraction Procedure for Water**

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<sub>18</sub> 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.

#### 6.4 Preparation of Standards and Fortification Solutions

A mixed stock standard solution of PFOA and  $^{13}\text{C}$  PFOA was prepared at a concentration of 1000  $\mu\text{g/mL}$  by dissolving 100 mg of each of the standards (corrected for purity and salt content) in 100 mL of methanol. From this solution, the following fortification standards were prepared:

| Conc. of<br>Stock or Fort.<br>Solution<br>( $\mu\text{g/mL}$ ) <sup>1</sup> | Fort<br>Volume<br>(mL) | Final<br>Volume<br>(mL) | Final Conc. of<br>Fortification Std.<br>( $\mu\text{g/mL}$ ) |
|-----------------------------------------------------------------------------|------------------------|-------------------------|--------------------------------------------------------------|
| 1000                                                                        | 10                     | 100                     | 100                                                          |
| 100                                                                         | 10                     | 100                     | 10                                                           |
| 10                                                                          | 10                     | 100                     | 1.0                                                          |
| 1.0                                                                         | 10                     | 100                     | 0.1                                                          |
| 0.1                                                                         | 10                     | 100                     | 0.01                                                         |

<sup>1</sup> of PFOA and  $^{13}\text{C}$  PFOA

A stock solution of only  $^{13}\text{C}$  PFOA was also prepared at a concentration of 100  $\mu\text{g/mL}$  by dissolving 10 mg the standard (corrected for purity and salt content) in 100 mL of methanol. From this solution, the following fortification standards were prepared:

| Conc. of<br>Stock or Fort.<br>Solution<br>( $\mu\text{g/mL}$ ) <sup>1</sup> | Fort<br>Volume<br>(mL) | Final<br>Volume<br>(mL) | Final Conc. of<br>Fortification Std.<br>( $\mu\text{g/mL}$ ) |
|-----------------------------------------------------------------------------|------------------------|-------------------------|--------------------------------------------------------------|
| 100                                                                         | 10                     | 100                     | 10                                                           |
| 10                                                                          | 10                     | 100                     | 1.0                                                          |
| 1.0                                                                         | 10                     | 100                     | 0.1                                                          |
| 0.1                                                                         | 10                     | 100                     | 0.01                                                         |

<sup>1</sup> of  $^{13}\text{C}$  PFOA

A set of extracted calibration standards containing PFOA and  $^{13}\text{C}$  PFOA were also made. The following concentrations were prepared before extraction:

| Conc. of Fort<br>Solution<br>(ng/mL) <sup>1</sup> | Fort<br>Volume<br>(mL) | Volume of<br>Fortified Sample<br>(mL) | Final Conc. of<br>Calibration Std.<br>(ng/mL) |
|---------------------------------------------------|------------------------|---------------------------------------|-----------------------------------------------|
| -                                                 | -                      | 40                                    | 0                                             |
| 10                                                | 0.1                    | 40                                    | 25                                            |
| 10                                                | 0.2                    | 40                                    | 50                                            |
| 10                                                | 0.4                    | 40                                    | 100                                           |
| 100                                               | 0.1                    | 40                                    | 250                                           |
| 100                                               | 0.2                    | 40                                    | 500                                           |
| 100                                               | 0.4                    | 40                                    | 1000                                          |

<sup>1</sup> of PFOA and  $^{13}\text{C}$  PFOA

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.

## 6.5 Chromatography

Quantification of PFOA was accomplished by LC/MS/MS electrospray. The retention time of PFOA was ~11.2 min. Peaks above the LOQ were not detected in any of the reagent blank samples corresponding to the analyte retention time.

## 6.6 Instrument Sensitivity

The smallest standard amount injected during the chromatographic run had a concentration of 0.2 ng/g of PFOA for sediment samples and a concentration of 25 ng/L for water samples.

## 6.7 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:

| <u>Analyte</u>   | <u>Mode</u> | <u>Transition Monitored</u> | <u>Approximate Retention Time (min)</u> |
|------------------|-------------|-----------------------------|-----------------------------------------|
| PFOA             | negative    | 413 → 369                   | ~11.2 min.                              |
| PFOA Confirm Ion | negative    | 413 → 219                   | ~11.2 min.                              |
| 13C PFOA         | negative    | 415 → 370                   | ~11.2 min.                              |

## 6.8 Quantitation and Example Calculation

PFOA concentrations are quantitated in nanogram of PFOA per liter of extract and then converted to nanogram per gram. 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 and  $^{13}\text{C}$  PFOA prior to extraction, Equation 2 was used to calculate the percent recovery.

Equation 2:

Recovery (%) =

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

Note: For the PFOA recovery calculation, the “control” is the unspiked aliquot of the primary field sample.

Equation 3 was used to convert the amount of PFOA found in ng/L to ng/g (ppb) in the soil samples.

Equation 3:

$$\text{PFOA found (ppb)} = \frac{[\text{PFOA found (ng/L)} \times \text{volume extracted (0.04L)}]}{\text{sample weight (5 g)}}$$

Equation 4 was then used to calculate the amount of PFOA found in ppb based on dry weight.

Equation 4:

$$\text{PFOA found (ppb) dry weight} = \text{PFOA found (ppb)} \times [100\% / \text{total solids}(\%)]$$

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

An example of a calculation using an actual sample follows:

Soil sample Exygen ID C0160874 Spk C (Set: 041706B), fortified at 500 ng/L with PFOA where:

|                                     |   |        |
|-------------------------------------|---|--------|
| peak area                           | = | 121349 |
| intercept                           | = | 18700  |
| slope                               | = | 1060   |
| dilution factor                     | = | 10     |
| ng/L PFOA added (fort level)        | = | 500    |
| ng/L in corresponding sample (ng/L) | = | 502    |
| volume extracted (L)                | = | 0.04   |
| sample weight (g)                   | = | 5      |
| total solids (%)                    | = | 87.24  |

**From equation 1:**

$$\begin{aligned}\text{Analyte found (ng/L)} &= \frac{[121349 - 18700]}{1060} \times 10 \\ &= 968 \text{ ng/L}\end{aligned}$$

**From equation 2:**

$$\begin{aligned}\% \text{ Recovery} &= \frac{(968 \text{ ng/L} - 502 \text{ ng/L})}{500 \text{ ng/L}} \times 100\% \\ &= 93\%\end{aligned}$$

**From equation 3:**

$$\begin{aligned}\text{PFOA found (ppb)} &= \frac{(968 \text{ ng/L} \times 0.04\text{L})}{5 \text{ g}} \\ &= 7.74 \text{ ppb}\end{aligned}$$

**From equation 4:**

$$\begin{aligned}\text{PFOA found (ppb) dry weight} &= 7.74 \text{ ppb} \times (100\% / 87.24\%) \\ &= 8.87 \text{ ppb}\end{aligned}$$

Note: Numbers may vary slightly due to rounding.

## **7.0 EXPERIMENTAL DESIGN**

<sup>13</sup>C PFOA was used as a surrogate for all of the samples. <sup>13</sup>C PFOA was added to the soil samples in the laboratory after the samples were weighed for extraction. For soil samples designated as laboratory matrix spikes, PFOA was also added at a known concentration to the samples in the laboratory after the samples were weighed for extraction. For all of the water samples, <sup>13</sup>C PFOA was added to the sample collection bottles in the laboratory before being shipped to the field for sampling. For water samples designated as field matrix spikes, PFOA was 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 soil samples were extracted in thirty-two sets. Each set included one reagent blank and two reagent blanks fortified at known concentrations. Twenty-two of the sets contained five samples sites and one set contained two sample sites. One sample was collected for each sample site. For each site, two laboratory matrix spikes and a laboratory duplicate were extracted. The last eight sets were re-extraction sets. Four sets contained eight samples, three sets contained seven samples, and one set contained six samples. Each re-extraction set consisted of one sample and variable laboratory matrix spikes.

The water samples were extracted in two sets. Each set included one reagent blank and two reagent blanks fortified at known concentrations. Set one contained three rinse blanks, two trip blanks with the associated spikes. Set two contained four rinse blanks and two trip blanks with the associated spikes.

## 8.0 RESULTS

Analytical results and assessed accuracies for the analysis of PFOA in soil samples based on dry weight are summarized in **Table I**. Analytical results and assessed accuracies for the analysis of PFOA in re-extracted soil samples based on dry weight are summarized in **Table II**. Analytical results and assessed accuracies for the analysis of PFOA in water samples are summarized in **Table III**. Field matrix spikes that were in the appropriate concentration range (primary concentration must be less than or equal to three times the spiking level) were used to calculate the assessed accuracies. When the field matrix spikes were not available or failed to meet recovery criteria, the laboratory spikes were used. Quantitative PFOA results were obtained for all samples except for nine soil samples that are not reported due to quality control failures.

Fortification recoveries for the analysis of PFOA in soil samples based on wet weight are summarized in **Table IV**. The overall average percent recovery  $\pm$  standard deviation for PFOA in soil samples based on wet weight was  $80 \pm 19\%$ . Fortification recoveries for the analysis of PFOA in re-extracted soil samples based on wet weight are summarized in **Table V**. The overall average percent recovery  $\pm$  standard deviation for PFOA in re-extracted soil samples based on wet weight was  $74 \pm 18\%$ . Fortification recoveries for the analysis of PFOA in water samples are summarized in **Table VI**. The overall average percent recovery  $\pm$  standard deviation for PFOA in water samples was  $92 \pm 7\%$ .

Fortification recoveries for  $^{13}\text{C}$  PFOA in soil samples based on wet weight are summarized in **Table VII**. The average percent recovery  $\pm$  standard deviation for  $^{13}\text{C}$  PFOA in soil samples based on wet weight was  $91 \pm 32\%$ . Fortification recoveries for  $^{13}\text{C}$  PFOA in re-extracted soil samples based on wet weight are summarized in **Table VIII**. The average percent recovery  $\pm$  standard deviation for  $^{13}\text{C}$  PFOA in re-extracted soil samples based on wet weight was  $48 \pm 30\%$ . Fortification recoveries for  $^{13}\text{C}$  PFOA in water samples are summarized in **Table IX**. The average percent recovery  $\pm$  standard deviation for  $^{13}\text{C}$  PFOA in water samples was  $107 \pm 14\%$ .

The total percent solids for soil samples are detailed in **Table X**.

## 9.0 CONCLUSIONS

Except as noted above, the soil and water samples were successfully extracted and analyzed for PFOA according to analytical methods V0001781 and V0001780,

respectively. There were no circumstances that would have affected the quality or integrity of the data.

## **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 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 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 PFOA in Soil Samples**

| Exygen ID    | Client<br>Sample ID   | Analyte Found (ppb, ng/g) Dry Weight<br>C8 Acid PFOA<br>Perfluorooctanoic Acid | Assessed<br>Accuracy<br>(+/- %) |
|--------------|-----------------------|--------------------------------------------------------------------------------|---------------------------------|
| C0156344     | DF13-SS-SS03-0-0000   | NR                                                                             | -                               |
| C0156344 Rep | DF13-SS-SS03-0-0000*  | NR                                                                             | -                               |
| C0156345     | DF13-SS-SS03-0-0003   | NR                                                                             | -                               |
| C0156345 Rep | DF13-SS-SS03-0-0003*  | NR                                                                             | -                               |
| C0156346     | DF13-SS-SS04-0-0000   | 56.7                                                                           | 40                              |
| C0156346 Rep | DF13-SS-SS04-0-0000*  | 58.6                                                                           | 40                              |
| C0156347     | DF13-SS-SS04-0-0003   | 339                                                                            | 40                              |
| C0156347 Rep | DF13-SS-SS04-0-0003*  | 288                                                                            | 40                              |
| C0156351     | DF13-SS-SS05-0-0000   | NR                                                                             | -                               |
| C0156351 Rep | DF13-SS-SS05-0-0000*  | NR                                                                             | -                               |
| C0156352     | DF13-SS-SS05-0-0003   | NR                                                                             | -                               |
| C0156352 Rep | DF13-SS-SS05-0-0003*  | NR                                                                             | -                               |
| C0156353     | DF13-SS-SS05-DB-0003  | NR                                                                             | -                               |
| C0156353 Rep | DF13-SS-SS05-DB-0003* | NR                                                                             | -                               |
| C0156354     | DF10-SS-SS01-0-0000   | NR                                                                             | -                               |
| C0156354 Rep | DF10-SS-SS01-0-0000*  | NR                                                                             | -                               |
| C0156355     | DF10-SS-SS01-0-0003   | 191                                                                            | 50                              |
| C0156355 Rep | DF10-SS-SS01-0-0003*  | 220                                                                            | 50                              |
| C0156356     | DF10-SS-SS02-0-0000   | 119                                                                            | 50                              |
| C0156356 Rep | DF10-SS-SS02-0-0000*  | 116                                                                            | 50                              |
| C0156357     | DF10-SS-SS02-DB-0000  | 105                                                                            | 40                              |
| C0156357 Rep | DF10-SS-SS02-DB-0000* | 108                                                                            | 40                              |
| C0156358     | DF10-SS-SS02-0-0003   | 99.8                                                                           | 40                              |
| C0156358 Rep | DF10-SS-SS02-0-0003*  | 98.9                                                                           | 40                              |
| C0156360     | DF10-SS-SS03-0-0000   | 16.9                                                                           | 50                              |
| C0156360 Rep | DF10-SS-SS03-0-0000*  | 15.2                                                                           | 50                              |
| C0156361     | DF10-SS-SS03-0-0003   | 20.3                                                                           | 30                              |
| C0156361 Rep | DF10-SS-SS03-0-0003*  | 20.8                                                                           | 30                              |
| C0156362     | DF10-SS-SS04-0-0000   | 339                                                                            | 30                              |
| C0156362 Rep | DF10-SS-SS04-0-0000*  | 363                                                                            | 30                              |

\*Laboratory Duplicate

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table II for re-extract data.

**Table I. Summary of PFOA in Soil Samples Continued**

| Exygen ID    | Client<br>Sample ID   | Analyte Found (ppb, ng/g) Dry Weight |                        | Assessed<br>Accuracy<br>(+/- %) |
|--------------|-----------------------|--------------------------------------|------------------------|---------------------------------|
|              |                       | C8 Acid PFOA                         | Perfluorooctanoic Acid |                                 |
| C0156363     | DF10-SS-SS04-0-0003   | 387                                  |                        | 30                              |
| C0156363 Rep | DF10-SS-SS04-0-0003*  | 435                                  |                        | 30                              |
| C0156364     | DF10-SS-SS05-0-0000   | 338                                  |                        | 50                              |
| C0156364 Rep | DF10-SS-SS05-0-0000*  | 313                                  |                        | 50                              |
| C0156365     | DF10-SS-SS05-0-0003   | 699                                  |                        | 50                              |
| C0156365 Rep | DF10-SS-SS05-0-0003*  | 560                                  |                        | 50                              |
| C0156366     | DF11-SS-SS01-0-0000   | 257                                  |                        | 30                              |
| C0156366 Rep | DF11-SS-SS01-0-0000*  | 250                                  |                        | 30                              |
| C0156367     | DF11-SS-SS01-0-0003   | NR                                   |                        | -                               |
| C0156367 Rep | DF11-SS-SS01-0-0003*  | NR                                   |                        | -                               |
| C0156368     | DF11-SS-SS02-0-0000   | NR                                   |                        | -                               |
| C0156368 Rep | DF11-SS-SS02-0-0000*  | NR                                   |                        | -                               |
| C0156370     | DF11-SS-SS02-0-0003   | NR                                   |                        | -                               |
| C0156370 Rep | DF11-SS-SS02-0-0003*  | NR                                   |                        | -                               |
| C0156371     | DF11-SS-SS03-0-0000   | NR                                   |                        | -                               |
| C0156371 Rep | DF11-SS-SS03-0-0000*  | NR                                   |                        | -                               |
| C0156372     | DF11-SS-SS03-DB-0000  | 138                                  |                        | 50                              |
| C0156372 Rep | DF11-SS-SS03-DB-0000* | 170                                  |                        | 50                              |
| C0156373     | DF11-SS-SS03-0-0003   | NR                                   |                        | -                               |
| C0156373 Rep | DF11-SS-SS03-0-0003*  | NR                                   |                        | -                               |
| C0156374     | DF11-SS-SS04-0-0000   | 28.7                                 |                        | 40                              |
| C0156374 Rep | DF11-SS-SS04-0-0000*  | 32.2                                 |                        | 40                              |
| C0156375     | DF11-SS-SS04-0-0003   | 102                                  |                        | 40                              |
| C0156375 Rep | DF11-SS-SS04-0-0003*  | 97.6                                 |                        | 40                              |
| C0156376     | DF11-SS-SS05-0-0000   | 48.9                                 |                        | 30                              |
| C0156376 Rep | DF11-SS-SS05-0-0000*  | 47.4                                 |                        | 30                              |
| C0156377     | DF11-SS-SS05-0-0003   | 53.4                                 |                        | 40                              |
| C0156377 Rep | DF11-SS-SS05-0-0003*  | 58.5                                 |                        | 40                              |
| C0156378     | DF05-SS-SS01-0-0000   | 119                                  |                        | 50                              |
| C0156378 Rep | DF05-SS-SS01-0-0000*  | 121                                  |                        | 50                              |
| C0156379     | DF05-SS-SS01-0-0003   | 51.6                                 |                        | 40                              |
| C0156379 Rep | DF05-SS-SS01-0-0003*  | 40.5                                 |                        | 40                              |
| C0156380     | DF05-SS-SS02-0-0000   | 346                                  |                        | 30                              |
| C0156380 Rep | DF05-SS-SS02-0-0000*  | 366                                  |                        | 30                              |
| C0156381     | DF05-SS-SS02-0-0003   | NR                                   |                        | -                               |
| C0156381 Rep | DF05-SS-SS02-0-0003*  | NR                                   |                        | -                               |

\*Laboratory Duplicate

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table II for re-extract data.

**Table I. Summary of PFOA in Soil Samples Continued**

| Exygen ID    | Client<br>Sample ID  | Analyte Found (ppb, ng/g) Dry Weight |                        | Assessed<br>Accuracy<br>(+/- %) |
|--------------|----------------------|--------------------------------------|------------------------|---------------------------------|
|              |                      | C8 Acid PFOA                         | Perfluorooctanoic Acid |                                 |
| C0156382     | DF12-SS-SS01-0-0000  | 49.8                                 |                        | 30                              |
| C0156382 Rep | DF12-SS-SS01-0-0000* | 82.8                                 |                        | 30                              |
| C0156383     | DF12-SS-SS01-0-0003  | NR                                   |                        | -                               |
| C0156383 Rep | DF12-SS-SS01-0-0003* | NR                                   |                        | -                               |
| C0156384     | DF12-SS-SS02-0-0000  | NR                                   |                        | -                               |
| C0156384 Rep | DF12-SS-SS02-0-0000* | NR                                   |                        | -                               |
| C0156385     | DF12-SS-SS02-0-0003  | NR                                   |                        | -                               |
| C0156385 Rep | DF12-SS-SS02-0-0003* | NR                                   |                        | -                               |
| C0156386     | DF12-SS-SS03-0-0000  | NR                                   |                        | -                               |
| C0156386 Rep | DF12-SS-SS03-0-0000* | NR                                   |                        | -                               |
| C0156387     | DF12-SS-SS03-0-0003  | 29.9                                 |                        | 50                              |
| C0156387 Rep | DF12-SS-SS03-0-0003* | 40.7                                 |                        | 50                              |
| C0156388     | DF12-SS-SS04-0-0000  | NR                                   |                        | -                               |
| C0156388 Rep | DF12-SS-SS04-0-0000* | NR                                   |                        | -                               |
| C0156389     | DF12-SS-SS04-0-0003  | NR                                   |                        | -                               |
| C0156389 Rep | DF12-SS-SS04-0-0003* | NR                                   |                        | -                               |
| C0156390     | DF12-SS-SS05-0-0000  | 42.1                                 |                        | 30                              |
| C0156390 Rep | DF12-SS-SS05-0-0000* | 48.8                                 |                        | 30                              |
| C0156391     | DF12-SS-SS05-0-0003  | 176                                  |                        | 40                              |
| C0156391 Rep | DF12-SS-SS05-0-0003* | 174                                  |                        | 40                              |
| C0156392     | DF13-SS-SS01-0-0000  | 310                                  |                        | 50                              |
| C0156392 Rep | DF13-SS-SS01-0-0000* | 315                                  |                        | 50                              |
| C0156393     | DF13-SS-SS01-0-0003  | NR                                   |                        | -                               |
| C0156393 Rep | DF13-SS-SS01-0-0003* | NR                                   |                        | -                               |
| C0156394     | DF13-SS-SS02-0-0000  | NR                                   |                        | -                               |
| C0156394 Rep | DF13-SS-SS02-0-0000* | NR                                   |                        | -                               |
| C0156395     | DF13-SS-SS02-0-0003  | NR                                   |                        | -                               |
| C0156395 Rep | DF13-SS-SS02-0-0003* | NR                                   |                        | -                               |
| C0159446     | DAL-SB-603R-0-0000   | NR                                   |                        | -                               |
| C0159446 Rep | DAL-SB-603R-0-0000*  | NR                                   |                        | -                               |
| C0159447     | DAL-SB-603R-0-0005   | 4.02                                 |                        | 50                              |
| C0159447 Rep | DAL-SB-603R-0-0005*  | 4.04                                 |                        | 50                              |

\*Laboratory Duplicate

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table II for re-extract data.

**Table I. Summary of PFOA in Soil Samples Continued**

| Exygen ID    | Client<br>Sample ID  | Analyte Found (ppb, ng/g) Dry Weight |                        | Assessed<br>Accuracy<br>(+/- %) |
|--------------|----------------------|--------------------------------------|------------------------|---------------------------------|
|              |                      | C8 Acid PFOA                         | Perfluorooctanoic Acid |                                 |
| C0159448     | DAL-SB-603R-0-0010   | 5.34                                 |                        | 40                              |
| C0159448 Rep | DAL-SB-603R-0-0010*  | 4.78                                 |                        | 40                              |
| C0159449     | DAL-SB-603R-0-0050   | NR                                   |                        | -                               |
| C0159449 Rep | DAL-SB-603R-0-0050*  | NR                                   |                        | -                               |
| C0159450     | DAL-SB-603R-0-0100   | 1.45                                 |                        | 40                              |
| C0159450 Rep | DAL-SB-603R-0-0100*  | 1.10                                 |                        | 40                              |
| C0159451     | DAL-SB-603R-DB-0100  | 0.936                                |                        | 30                              |
| C0159451 Rep | DAL-SB-603R-DB-0100* | 0.990                                |                        | 30                              |
| C0159453     | DAL-SB-604R-0-0000   | NR                                   |                        | -                               |
| C0159453 Rep | DAL-SB-604R-0-0000*  | NR                                   |                        | -                               |
| C0159454     | DAL-SB-604R-0-0005   | NR                                   |                        | -                               |
| C0159454 Rep | DAL-SB-604R-0-0005*  | NR                                   |                        | -                               |
| C0159455     | DAL-SB-604R-0-0010   | 1.46                                 |                        | 30                              |
| C0159455 Rep | DAL-SB-604R-0-0010*  | 1.62                                 |                        | 30                              |
| C0159456     | DAL-SB-604R-0-0050   | 5.71                                 |                        | 30                              |
| C0159456 Rep | DAL-SB-604R-0-0050*  | 5.52                                 |                        | 30                              |
| C0159457     | DAL-SB-604R-0-0100   | ND                                   |                        | 30                              |
| C0159457 Rep | DAL-SB-604R-0-0100*  | 0.356                                |                        | 30                              |
| C0159458     | DAL-SB-605R-0-0000   | 7.95                                 |                        | 40                              |
| C0159458 Rep | DAL-SB-605R-0-0000*  | 7.86                                 |                        | 40                              |
| C0159459     | DAL-SB-605R-DB-0000  | 7.61                                 |                        | 30                              |
| C0159459 Rep | DAL-SB-605R-DB-0000* | 7.98                                 |                        | 30                              |
| C0159460     | DAL-SB-605R-0-0005   | 7.82                                 |                        | 30                              |
| C0159460 Rep | DAL-SB-605R-0-0005*  | 7.46                                 |                        | 30                              |
| C0159461     | DAL-SB-605R-0-0010   | 2.64                                 |                        | 30                              |
| C0159461 Rep | DAL-SB-605R-0-0010*  | 3.33                                 |                        | 30                              |
| C0159462     | DAL-SB-605R-0-0050   | 1.01                                 |                        | 30                              |
| C0159462 Rep | DAL-SB-605R-0-0050*  | 1.03                                 |                        | 30                              |
| C0159463     | DAL-SB-605R-0-0100   | 0.849                                |                        | 30                              |
| C0159463 Rep | DAL-SB-605R-0-0100*  | 0.862                                |                        | 30                              |
| C0159464     | DAL-SB-138R-0-0000   | 27.6                                 |                        | 30                              |
| C0159464 Rep | DAL-SB-138R-0-0000*  | 29.1                                 |                        | 30                              |
| C0159466     | DAL-SB-138R-0-0005   | 48.8                                 |                        | 30                              |
| C0159466 Rep | DAL-SB-138R-0-0005*  | 57.2                                 |                        | 30                              |
| C0159467     | DAL-SB-138R-0-0010   | 40.7                                 |                        | 30                              |
| C0159467 Rep | DAL-SB-138R-0-0010*  | 47.4                                 |                        | 30                              |
| C0159468     | DAL-SB-138R-0-0050   | NR                                   |                        | -                               |
| C0159468 Rep | DAL-SB-138R-0-0050*  | NR                                   |                        | -                               |

\*Laboratory Duplicate

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table II for re-extract data.

**Table I. Summary of PFOA in Soil Samples Continued**

| Exygen ID    | Client<br>Sample ID   | Analyte Found (ppb, ng/g) Dry Weight<br>C8 Acid PFOA<br>Perfluorooctanoic Acid | Assessed<br>Accuracy<br>(+/- %) |
|--------------|-----------------------|--------------------------------------------------------------------------------|---------------------------------|
| C0159470     | DAL-SB-138R-0-0100    | 354                                                                            | 40                              |
| C0159470 Rep | DAL-SB-138R-0-0100*   | 269                                                                            | 40                              |
| C0159471     | DAL-SB-138R-DB-0100   | 308                                                                            | 30                              |
| C0159471 Rep | DAL-SB-138R-DB-0100*  | 309                                                                            | 30                              |
| C0160871     | DF05-SS-SS03-0-0000   | NR                                                                             | -                               |
| C0160871 Rep | DF05-SS-SS03-0-0000*  | NR                                                                             | -                               |
| C0160872     | DF05-SS-SS03-0-0003   | NR                                                                             | -                               |
| C0160872 Rep | DF05-SS-SS03-0-0003*  | NR                                                                             | -                               |
| C0160873     | DF05-SS-SS04-0-0000   | NR                                                                             | -                               |
| C0160873 Rep | DF05-SS-SS04-0-0000*  | NR                                                                             | -                               |
| C0160874     | DF05-SS-SS04-0-0003   | 4.60                                                                           | 30                              |
| C0160874 Rep | DF05-SS-SS04-0-0003*  | 4.62                                                                           | 30                              |
| C0160875     | DF05-SS-SS05-0-0000   | 5.79                                                                           | 40                              |
| C0160875 Rep | DF05-SS-SS05-0-0000*  | 5.71                                                                           | 40                              |
| C0160876     | DF05-SS-SS05-0-0003   | 7.23                                                                           | 30                              |
| C0160876 Rep | DF05-SS-SS05-0-0003*  | 6.76                                                                           | 30                              |
| C0160877     | DF05-SS-SS05-DB-0003  | 7.41                                                                           | 50                              |
| C0160877 Rep | DF05-SS-SS05-DB-0003* | 7.12                                                                           | 50                              |
| C0160878     | DF09-SS-0006-0-0000   | 14.8                                                                           | 30                              |
| C0160878 Rep | DF09-SS-0006-0-0000*  | 14.9                                                                           | 30                              |
| C0160879     | DF09-SS-0006-0-0003   | 16.3                                                                           | 30                              |
| C0160879 Rep | DF09-SS-0006-0-0003*  | 15.4                                                                           | 30                              |
| C0160880     | DF09-SS-0006-0-0010   | 8.91                                                                           | 40                              |
| C0160880 Rep | DF09-SS-0006-0-0010*  | 8.92                                                                           | 40                              |
| C0160881     | DF09-SS-0007-0-0000   | 173                                                                            | 50                              |
| C0160881 Rep | DF09-SS-0007-0-0000*  | 182                                                                            | 50                              |
| C0160882     | DF09-SS-0007-0-0003   | 124                                                                            | 50                              |
| C0160882 Rep | DF09-SS-0007-0-0003*  | 112                                                                            | 50                              |

\*Laboratory Duplicate

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table II for re-extract data.

**Table I. Summary of PFOA in Soil Samples Continued**

| Exygen ID    | Client Sample ID      | Analyte Found (ppb, ng/g) Dry Weight<br>C8 Acid PFOA<br>Perfluorooctanoic Acid | Assessed Accuracy<br>(+/- %) |
|--------------|-----------------------|--------------------------------------------------------------------------------|------------------------------|
| C0160883     | DF09-SS-0007-0-0010   | 46.7                                                                           | 50                           |
| C0160883 Rep | DF09-SS-0007-0-0010*  | 51.4                                                                           | 50                           |
| C0160884     | DF09-SS-0007-DB-0010  | 38.9                                                                           | 30                           |
| C0160884 Rep | DF09-SS-0007-DB-0010* | 47.9                                                                           | 30                           |
| C0160885     | DF09-SS-0008-0-0000   | 176                                                                            | 30                           |
| C0160885 Rep | DF09-SS-0008-0-0000*  | 149                                                                            | 30                           |
| C0160886     | DF09-SS-0008-0-0003   | 149                                                                            | 40                           |
| C0160886 Rep | DF09-SS-0008-0-0003*  | 135                                                                            | 40                           |
| C0160887     | DF09-SS-0008-0-0010   | 122                                                                            | 30                           |
| C0160887 Rep | DF09-SS-0008-0-0010*  | 108                                                                            | 30                           |
| C0160888     | DF09-SS-0009-0-0000   | 10.1                                                                           | 40                           |
| C0160888 Rep | DF09-SS-0009-0-0000*  | 11.1                                                                           | 40                           |
| C0160889     | DF09-SS-0009-0-0003   | 12.2                                                                           | 30                           |
| C0160889 Rep | DF09-SS-0009-0-0003*  | 14.6                                                                           | 30                           |
| C0160890     | DF09-SS-0009-DB-0003  | 12.3                                                                           | 30                           |
| C0160890 Rep | DF09-SS-0009-DB-0003* | 13.5                                                                           | 30                           |
| C0160891     | DF09-SS-0009-0-0010   | 19.0                                                                           | 30                           |
| C0160891 Rep | DF09-SS-0009-0-0010*  | 23.1                                                                           | 30                           |
| C0160892     | DF09-SS-0010-0-0000   | 886                                                                            | 30                           |
| C0160892 Rep | DF09-SS-0010-0-0000*  | 934                                                                            | 30                           |
| C0160893     | DF09-SS-0010-0-0003   | 511                                                                            | 30                           |
| C0160893 Rep | DF09-SS-0010-0-0003*  | 498                                                                            | 30                           |
| C0160894     | DF09-SS-0010-0-0010   | 461                                                                            | 30                           |
| C0160894 Rep | DF09-SS-0010-0-0010*  | 357                                                                            | 30                           |
| C0160895     | DF09-SS-0011-0-0000   | 253                                                                            | 30                           |
| C0160895 Rep | DF09-SS-0011-0-0000*  | 297                                                                            | 30                           |
| C0160896     | DF09-SS-0011-0-0003   | 284                                                                            | 30                           |
| C0160896 Rep | DF09-SS-0011-0-0003*  | 360                                                                            | 30                           |

\*Laboratory Duplicate

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table II for re-extract data.

**Table I. Summary of PFOA in Soil Samples Continued**

| Exygen ID    | Client<br>Sample ID   | Analyte Found (ppb, ng/g) Dry Weight<br>C8 Acid PFOA<br>Perfluorooctanoic Acid | Assessed<br>Accuracy<br>(+/- %) |
|--------------|-----------------------|--------------------------------------------------------------------------------|---------------------------------|
| C0160897     | DF09-SS-0011-0-0010   | NR                                                                             | -                               |
| C0160897 Rep | DF09-SS-0011-0-0010*  | NR                                                                             | -                               |
| C0160898     | DF09-SS-0012-0-0000   | NR                                                                             | -                               |
| C0160898 Rep | DF09-SS-0012-0-0000*  | NR                                                                             | -                               |
| C0160899     | DF09-SS-0012-DB-0000  | 535                                                                            | 30                              |
| C0160899 Rep | DF09-SS-0012-DB-0000* | 500                                                                            | 30                              |
| C0160900     | DF09-SS-0012-0-0003   | NR                                                                             | -                               |
| C0160900 Rep | DF09-SS-0012-0-0003*  | NR                                                                             | -                               |
| C0160901     | DF09-SS-0012-0-0010   | 481                                                                            | 40                              |
| C0160901 Rep | DF09-SS-0012-0-0010*  | 454                                                                            | 40                              |
| C0160902     | DF09-SS-0013-0-0000   | 21.1                                                                           | 30                              |
| C0160902 Rep | DF09-SS-0013-0-0000*  | 21.0                                                                           | 30                              |
| C0160903     | DF09-SS-0013-0-0003   | 25.4                                                                           | 30                              |
| C0160903 Rep | DF09-SS-0013-0-0003*  | 29.3                                                                           | 30                              |
| C0160904     | DF09-SS-0013-0-0010   | 50.5                                                                           | 40                              |
| C0160904 Rep | DF09-SS-0013-0-0010*  | 42.7                                                                           | 40                              |
| C0164247     | DAL-SB-602R-0-0000    | 6.11                                                                           | 30                              |
| C0164247 Rep | DAL-SB-602R-0-0000*   | 6.58                                                                           | 30                              |
| C0164248     | DAL-SB-602R-0-0005    | 6.91                                                                           | 30                              |
| C0164248 Rep | DAL-SB-602R-0-0005*   | 6.73                                                                           | 30                              |
| C0164249     | DAL-SB-602R-0-0010    | 4.03                                                                           | 30                              |
| C0164249 Rep | DAL-SB-602R-0-0010*   | 3.24                                                                           | 30                              |
| C0164250     | DAL-SB-602R-0-0050    | 0.766                                                                          | 30                              |
| C0164250 Rep | DAL-SB-602R-0-0050*   | 0.674                                                                          | 30                              |
| C0164251     | DAL-SB-602R-0-0100    | 0.665                                                                          | 40                              |
| C0164251 Rep | DAL-SB-602R-0-0100*   | 0.561                                                                          | 40                              |
| C0164252     | DAL-SB-601R-0-0000    | 1.75                                                                           | 30                              |
| C0164252 Rep | DAL-SB-601R-0-0000*   | 2.11                                                                           | 30                              |
| C0164253     | DAL-SB-601R-DB-0000   | 2.22                                                                           | 30                              |
| C0164253 Rep | DAL-SB-601R-DB-0000   | 1.97                                                                           | 30                              |
| C0164254     | DAL-SB-601R-0-0005    | 1.91                                                                           | 30                              |
| C0164254 Rep | DAL-SB-601R-0-0005    | 1.87                                                                           | 30                              |

\*Laboratory Duplicate

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table II for re-extract data.

**Table I. Summary of PFOA in Soil Samples Continued**

| Exygen ID    | Client<br>Sample ID  | Analyte Found (ppb, ng/g) Dry Weight |                        | Assessed<br>Accuracy<br>(+/- %) |
|--------------|----------------------|--------------------------------------|------------------------|---------------------------------|
|              |                      | C8 Acid PFOA                         | Perfluorooctanoic Acid |                                 |
| C0164255     | DAL-SB-601R-0-0010   | NR                                   |                        | -                               |
| C0164255 Rep | DAL-SB-601R-0-0010   | NR                                   |                        | -                               |
| C0164256     | DAL-SB-601R-DB-0010  | 2.15                                 |                        | 40                              |
| C0164256 Rep | DAL-SB-601R-DB-0010  | 1.66                                 |                        | 40                              |
| C0164257     | DAL-SB-601R-0-0050   | 0.895                                |                        | 30                              |
| C0164257 Rep | DAL-SB-601R-0-0050   | 1.42                                 |                        | 30                              |
| C0164259     | DAL-SB-601R-0-0100   | 0.477                                |                        | 30                              |
| C0164259 Rep | DAL-SB-601R-0-0100*  | 0.438                                |                        | 30                              |
| C0164260     | DAL-SB-601R-DB-0100  | 0.485                                |                        | 30                              |
| C0164260 Rep | DAL-SB-601R-DB-0100* | 0.399                                |                        | 30                              |

\*Laboratory Duplicate

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table II for re-extract data.

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

| Exygen ID | Client<br>Sample ID  | Analyte Found (ppb, ng/g) Dry Weight |                        | Assessed<br>Accuracy<br>(+/- %) |
|-----------|----------------------|--------------------------------------|------------------------|---------------------------------|
|           |                      | C8 Acid PFOA                         | Perfluorooctanoic Acid |                                 |
| C0156344  | DF13-SS-SS03-0-0000  | 525                                  |                        | 40                              |
| C0156345  | DF13-SS-SS03-0-0003  | 551                                  |                        | 30                              |
| C0156351  | DF13-SS-SS05-0-0000  | 1130                                 |                        | 40                              |
| C0156352  | DF13-SS-SS05-0-0003  | 1020                                 |                        | 30                              |
| C0156353  | DF13-SS-SS05-DB-0003 | 857                                  |                        | 50                              |
| C0156354  | DF10-SS-SS01-0-0000  | NR                                   |                        | -                               |
| C0156367  | DF11-SS-SS01-0-0003  | 86.8                                 |                        | 30                              |
| C0156368  | DF11-SS-SS02-0-0000  | NR                                   |                        | -                               |
| C0156370  | DF11-SS-SS02-0-0003  | 818                                  |                        | 30                              |
| C0156371  | DF11-SS-SS03-0-0000  | 102                                  |                        | 40                              |
| C0156373  | DF11-SS-SS03-0-0003  | NR                                   |                        | -                               |
| C0156381  | DF05-SS-SS02-0-0003  | 335                                  |                        | 50                              |
| C0156383  | DF12-SS-SS01-0-0003  | 197                                  |                        | 30                              |
| C0156384  | DF12-SS-SS02-0-0000  | 2140                                 |                        | 50                              |
| C0156385  | DF12-SS-SS02-0-0003  | 3910                                 |                        | 40                              |
| C0156386  | DF12-SS-SS03-0-0000  | 28.6                                 |                        | 50                              |
| C0156388  | DF12-SS-SS04-0-0000  | 4390                                 |                        | 30                              |
| C0156389  | DF12-SS-SS04-0-0003  | 11900                                |                        | 30                              |
| C0156393  | DF13-SS-SS01-0-0003  | 561                                  |                        | 30                              |
| C0156394  | DF13-SS-SS02-0-0000  | 3350                                 |                        | 50                              |
| C0156395  | DF13-SS-SS02-0-0003  | 5930                                 |                        | 30                              |
| C0159449  | DAL-SB-603R-0-0050   | 3.71                                 |                        | 30                              |
| C0159453  | DAL-SB-604R-0-0000   | 2.29                                 |                        | 50                              |
| C0159454  | DAL-SB-604R-0-0005   | 3.75                                 |                        | 30                              |
| C0159468  | DAL-SB-138R-0-0050   | 104                                  |                        | 30                              |
| C0160871  | DF05-SS-SS03-0-0000  | 227                                  |                        | 40                              |
| C0160872  | DF05-SS-SS03-0-0003  | NR                                   |                        | -                               |
| C0160873  | DF05-SS-SS04-0-0000  | NR                                   |                        | -                               |
| C0160897  | DF09-SS-0011-0-0010  | 238                                  |                        | 40                              |
| C0160898  | DF09-SS-0012-0-0000  | NR                                   |                        | -                               |
| C0160900  | DF09-SS-0012-0-0003  | NR                                   |                        | -                               |
| C0164255  | DAL-SB-601R-0-0010   | NR                                   |                        | -                               |

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g.

NR = Not reported due to quality control failures.

**Table III. Summary of PFOA in Associated Rinse Blanks and Trip Blanks**

| Exygen ID | Client<br>Sample ID    | Analyte Found (ppt, ng/L) |                        | Assessed Accuracy<br>(+/- %) |
|-----------|------------------------|---------------------------|------------------------|------------------------------|
|           |                        | C8 Acid PFOA              | Perfluorooctanoic Acid |                              |
| C0156348  | DF13-SS-TRIP1-0-060221 | ND                        |                        | 30                           |
| C0156359  | DF10-SS-SS03-RB-0000   | ND                        |                        | 30                           |
| C0156369  | DF11-SS-SS02-RB-0003   | ND                        |                        | 30                           |
| C0159443  | DAL-SB-TRIP1-0-060302  | ND                        |                        | 30                           |
| C0159452  | DAL-SB-603R-RB-0100    | ND                        |                        | 30                           |
| C0159465  | DAL-SB-138R-RB-0000    | ND                        |                        | 30                           |
| C0159469  | DAL-SB-138R-RB-0100    | ND                        |                        | 30                           |
| C0160867  | DF05-SS-TRIP2-0-060308 | ND                        |                        | 30                           |
| C0160870  | DF05-SS-SS03-RB-0000   | ND                        |                        | 30                           |
| C0164222  | DAL-SB-TRIP-0-060316   | ND                        |                        | 30                           |
| C0164258  | DAL-SB-601R-RB-0100    | ND                        |                        | 30                           |

ND = Not detected at or above the Limit of Quantitation (LOQ) of 25 ng/L.

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples**

| Sample Description                                      | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|---------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                         |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF13-SS-SS03-0-0000<br>(C0156344 Spk C, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF13-SS-SS03-0-0000<br>(C0156344 Spk D, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF13-SS-SS03-0-0003<br>(C0156345 Spk E, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF13-SS-SS03-0-0003<br>(C0156345 Spk F, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF13-SS-SS04-0-0000<br>(C0156346 Spk G, 4 ppb Spike)    | 4                    | 47.3                                  | 52.7                               | *            |
| DF13-SS-SS04-0-0000<br>(C0156346 Spk H, 400 ppb Spike)  | 400                  | 47.3                                  | 298                                | 63           |
| DF13-SS-SS04-0-0003<br>(C0156347 Spk I, 4 ppb Spike)    | 4                    | 296                                   | 284                                | *            |
| DF13-SS-SS04-0-0003<br>(C0156347 Spk J, 400 ppb Spike)  | 400                  | 296                                   | 570                                | 69           |
| DF13-SS-SS05-0-0000<br>(C0156351 Spk K, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF13-SS-SS05-0-0000<br>(C0156351 Spk L, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF13-SS-SS05-0-0003<br>(C0156352 Spk C, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF13-SS-SS05-0-0003<br>(C0156352 Spk D, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF13-SS-SS05-DB-0003<br>(C0156353 Spk E, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF13-SS-SS05-DB-0003<br>(C0156353 Spk F, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF10-SS-SS01-0-0000<br>(C0156354 Spk G, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF10-SS-SS01-0-0000<br>(C0156354 Spk H, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF10-SS-SS01-0-0003<br>(C0156355 Spk I, 4 ppb Spike)    | 4                    | 166                                   | 161                                | *            |
| DF10-SS-SS01-0-0003<br>(C0156355 Spk J, 400 ppb Spike)  | 400                  | 166                                   | 397                                | 58           |
| DF10-SS-SS02-0-0000<br>(C0156356 Spk K, 4 ppb Spike)    | 4                    | 102                                   | 105                                | *            |
| DF10-SS-SS02-0-0000<br>(C0156356 Spk L, 400 ppb Spike)  | 400                  | 102                                   | 330                                | 57           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                      | C8 Acid PFOA         |                                       |                                    |              |
|---------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                         | Amount Spiked (ng/g) | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF10-SS-SS02-DB-0000<br>(C0156357 Spk C, 4 ppb Spike)   | 4                    | 91.2                                  | 102                                | *            |
| DF10-SS-SS02-DB-0000<br>(C0156357 Spk D, 400 ppb Spike) | 400                  | 91.2                                  | 364                                | 68           |
| DF10-SS-SS02-0-0003<br>(C0156358 Spk E, 4 ppb Spike)    | 4                    | 86.4                                  | 82.4                               | *            |
| DF10-SS-SS02-0-0003<br>(C0156358 Spk F, 400 ppb Spike)  | 400                  | 86.4                                  | 330                                | 61           |
| DF10-SS-SS03-0-0000<br>(C0156360 Spk G, 4 ppb Spike)    | 4                    | 13.8                                  | 14.8                               | *            |
| DF10-SS-SS03-0-0000<br>(C0156360 Spk H, 400 ppb Spike)  | 400                  | 13.8                                  | 246                                | 58           |
| DF10-SS-SS03-0-0003<br>(C0156361 Spk I, 4 ppb Spike)    | 4                    | 17.1                                  | 19.4                               | *            |
| DF10-SS-SS03-0-0003<br>(C0156361 Spk J, 400 ppb Spike)  | 400                  | 17.1                                  | 304                                | 72           |
| DF10-SS-SS04-0-0000<br>(C0156362 Spk K, 4 ppb Spike)    | 4                    | 242                                   | 196                                | *            |
| DF10-SS-SS04-0-0000<br>(C0156362 Spk L, 400 ppb Spike)  | 400                  | 242                                   | 530                                | 72           |
| DF10-SS-SS04-0-0003<br>(C0156363 Spk C, 4 ppb Spike)    | 4                    | 293                                   | 310                                | *            |
| DF10-SS-SS04-0-0003<br>(C0156363 Spk D, 400 ppb Spike)  | 400                  | 293                                   | 598                                | 76           |
| DF10-SS-SS05-0-0000<br>(C0156364 Spk E, 4 ppb Spike)    | 4                    | 278                                   | 263                                | *            |
| DF10-SS-SS05-0-0000<br>(C0156364 Spk F, 400 ppb Spike)  | 400                  | 278                                   | 490                                | 53           |
| DF10-SS-SS05-0-0003<br>(C0156365 Spk G, 4 ppb Spike)    | 4                    | 577                                   | 596                                | *            |
| DF10-SS-SS05-0-0003<br>(C0156365 Spk H, 400 ppb Spike)  | 400                  | 577                                   | 800                                | 56           |
| DF11-SS-SS01-0-0000<br>(C0156366 Spk I, 4 ppb Spike)    | 4                    | 209                                   | 228                                | *            |
| DF11-SS-SS01-0-0000<br>(C0156366 Spk J, 400 ppb Spike)  | 400                  | 209                                   | 498                                | 72           |
| DF11-SS-SS01-0-0003<br>(C0156367 Spk K, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF11-SS-SS01-0-0003<br>(C0156367 Spk L, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                      | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|---------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                         |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF11-SS-SS02-0-0000<br>(C0156368 Spk C, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF11-SS-SS02-0-0000<br>(C0156368 Spk D, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF11-SS-SS02-0-0003<br>(C0156370 Spk E, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF11-SS-SS02-0-0003<br>(C0156370 Spk F, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF11-SS-SS03-0-0000<br>(C0156371 Spk G, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF11-SS-SS03-0-0000<br>(C0156371 Spk H, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF11-SS-SS03-DB-0000<br>(C0156372 Spk I, 4 ppb Spike)   | 4                    | 110                                   | 113                                | *            |
| DF11-SS-SS03-DB-0000<br>(C0156372 Spk J, 400 ppb Spike) | 400                  | 110                                   | 316                                | 52           |
| DF11-SS-SS03-0-0003<br>(C0156373 Spk K, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF11-SS-SS03-0-0003<br>(C0156373 Spk L, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF11-SS-SS04-0-0000<br>(C0156374 Spk C, 4 ppb Spike)    | 4                    | 24.9                                  | 29.4                               | *            |
| DF11-SS-SS04-0-0000<br>(C0156374 Spk D, 400 ppb Spike)  | 400                  | 24.9                                  | 298                                | 68           |
| DF11-SS-SS04-0-0003<br>(C0156375 Spk E, 4 ppb Spike)    | 4                    | 91.2                                  | 96.0                               | *            |
| DF11-SS-SS04-0-0003<br>(C0156375 Spk F, 400 ppb Spike)  | 400                  | 91.2                                  | 342                                | 63           |
| DF11-SS-SS05-0-0000<br>(C0156376 Spk G, 4 ppb Spike)    | 4                    | 40.9                                  | 34.6                               | *            |
| DF11-SS-SS05-0-0000<br>(C0156376 Spk H, 400 ppb Spike)  | 400                  | 40.9                                  | 322                                | 70           |
| DF11-SS-SS05-0-0003<br>(C0156377 Spk I, 4 ppb Spike)    | 4                    | 45.5                                  | 44.9                               | *            |
| DF11-SS-SS05-0-0003<br>(C0156377 Spk J, 400 ppb Spike)  | 400                  | 45.5                                  | 317                                | 68           |
| DF05-SS-SS01-0-0000<br>(C0156378 Spk K, 4 ppb Spike)    | 4                    | 96.8                                  | 126                                | *            |
| DF05-SS-SS01-0-0000<br>(C0156378 Spk L, 400 ppb Spike)  | 400                  | 96.8                                  | 318                                | 55           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                     | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|--------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                        |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF05-SS-SS01-0-0003<br>(C0156379 Spk C, 4 ppb Spike)   | 4                    | 43.4                                  | 45.0                               | *            |
| DF05-SS-SS01-0-0003<br>(C0156379 Spk D, 400 ppb Spike) | 400                  | 43.4                                  | 303                                | 65           |
| DF05-SS-SS02-0-0000<br>(C0156380 Spk E, 4 ppb Spike)   | 4                    | 285                                   | 410                                | *            |
| DF05-SS-SS02-0-0000<br>(C0156380 Spk F, 400 ppb Spike) | 400                  | 285                                   | 681                                | 99           |
| DF05-SS-SS02-0-0003<br>(C0156381 Spk G, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF05-SS-SS02-0-0003<br>(C0156381 Spk H, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF12-SS-SS01-0-0000<br>(C0156382 Spk I, 4 ppb Spike)   | 4                    | 41.2                                  | 80.8                               | *            |
| DF12-SS-SS01-0-0000<br>(C0156382 Spk J, 400 ppb Spike) | 400                  | 41.2                                  | 326                                | 71           |
| DF12-SS-SS01-0-0003<br>(C0156383 Spk K, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF12-SS-SS01-0-0003<br>(C0156383 Spk L, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF12-SS-SS02-0-0000<br>(C0156384 Spk C, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF12-SS-SS02-0-0000<br>(C0156384 Spk D, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF12-SS-SS02-0-0003<br>(C0156385 Spk E, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF12-SS-SS02-0-0003<br>(C0156385 Spk F, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF12-SS-SS03-0-0000<br>(C0156386 Spk G, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF12-SS-SS03-0-0000<br>(C0156386 Spk H, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF12-SS-SS03-0-0003<br>(C0156387 Spk I, 4 ppb Spike)   | 4                    | 26.2                                  | 22.3                               | *            |
| DF12-SS-SS03-0-0003<br>(C0156387 Spk J, 400 ppb Spike) | 400                  | 26.2                                  | 254                                | 57           |
| DF12-SS-SS04-0-0000<br>(C0156388 Spk K, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF12-SS-SS04-0-0000<br>(C0156388 Spk L, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                     | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|--------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                        |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF12-SS-SS04-0-0003<br>(C0156389 Spk C, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF12-SS-SS04-0-0003<br>(C0156389 Spk D, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF12-SS-SS05-0-0000<br>(C0156390 Spk E, 4 ppb Spike)   | 4                    | 33.0                                  | 37.3                               | *            |
| DF12-SS-SS05-0-0000<br>(C0156390 Spk F, 400 ppb Spike) | 400                  | 33.0                                  | 406                                | 93           |
| DF12-SS-SS05-0-0003<br>(C0156391 Spk G, 4 ppb Spike)   | 4                    | 143                                   | 148                                | *            |
| DF12-SS-SS05-0-0003<br>(C0156391 Spk H, 400 ppb Spike) | 400                  | 143                                   | 407                                | 66           |
| DF13-SS-SS01-0-0000<br>(C0156392 Spk I, 4 ppb Spike)   | 4                    | 240                                   | 251                                | *            |
| DF13-SS-SS01-0-0000<br>(C0156392 Spk J, 400 ppb Spike) | 400                  | 240                                   | 438                                | 50           |
| DF13-SS-SS01-0-0003<br>(C0156393 Spk K, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF13-SS-SS01-0-0003<br>(C0156393 Spk L, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF13-SS-SS02-0-0000<br>(C0156394 Spk C, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF13-SS-SS02-0-0000<br>(C0156394 Spk D, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DF13-SS-SS02-0-0003<br>(C0156395 Spk E, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF13-SS-SS02-0-0003<br>(C0156395 Spk F, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |
| DAL-SB-603R-0-0000<br>(C0159446 Spk E, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DAL-SB-603R-0-0000<br>(C0159446 Spk F, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DAL-SB-603R-0-0005<br>(C0159447 Spk G, 4 ppb Spike)    | 4                    | 3.45                                  | 5.57                               | 53           |
| DAL-SB-603R-0-0005<br>(C0159447 Spk H, 400 ppb Spike)  | 400                  | 3.45                                  | 268                                | 66           |
| DAL-SB-603R-0-0010<br>(C0159448 Spk I, 4 ppb Spike)    | 4                    | 4.36                                  | 6.81                               | 61           |
| DAL-SB-603R-0-0010<br>(C0159448 Spk J, 400 ppb Spike)  | 400                  | 4.36                                  | 282                                | 69           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                     | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|--------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                        |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DAL-SB-603R-0-0050<br>(C0159449 Spk K, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DAL-SB-603R-0-0050<br>(C0159449 Spk L, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DAL-SB-603R-0-0100<br>(C0159450 Spk C, 4 ppb Spike)    | 4                    | 1.21                                  | 3.80                               | 65           |
| DAL-SB-603R-0-0100<br>(C0159450 Spk D, 400 ppb Spike)  | 400                  | 1.21                                  | 303                                | 75           |
| DAL-SB-603R-DB-0100<br>(C0159451 Spk E, 4 ppb Spike)   | 4                    | 0.733                                 | 3.72                               | 75           |
| DAL-SB-603R-DB-0100<br>(C0159451 Spk F, 400 ppb Spike) | 400                  | 0.733                                 | 295                                | 74           |
| DAL-SB-604R-0-0000<br>(C0159453 Spk G, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DAL-SB-604R-0-0000<br>(C0159453 Spk H, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DAL-SB-604R-0-0005<br>(C0159454 Spk I, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DAL-SB-604R-0-0005<br>(C0159454 Spk J, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DAL-SB-604R-0-0010<br>(C0159455 Spk K, 4 ppb Spike)    | 4                    | 1.20                                  | 4.14                               | 74           |
| DAL-SB-604R-0-0010<br>(C0159455 Spk L, 400 ppb Spike)  | 400                  | 1.20                                  | 343                                | 85           |
| DAL-SB-604R-0-0050<br>(C0159456 Spk C, 4 ppb Spike)    | 4                    | 5.04                                  | 9.20                               | 104          |
| DAL-SB-604R-0-0050<br>(C0159456 Spk D, 400 ppb Spike)  | 400                  | 5.04                                  | 294                                | 72           |
| DAL-SB-604R-0-0100<br>(C0159457 Spk E, 4 ppb Spike)    | 4                    | ND                                    | 3.93                               | 98           |
| DAL-SB-604R-0-0100<br>(C0159457 Spk F, 400 ppb Spike)  | 400                  | ND                                    | 328                                | 82           |
| DAL-SB-605R-0-0000<br>(C0159458 Spk G, 4 ppb Spike)    | 4                    | 6.47                                  | 11.7                               | 131          |
| DAL-SB-605R-0-0000<br>(C0159458 Spk H, 400 ppb Spike)  | 400                  | 6.47                                  | 361                                | 89           |
| DAL-SB-605R-DB-0000<br>(C0159459 Spk I, 4 ppb Spike)   | 4                    | 6.22                                  | 10.6                               | 110          |
| DAL-SB-605R-DB-0000<br>(C0159459 Spk J, 400 ppb Spike) | 400                  | 6.22                                  | 387                                | 95           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                     | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|--------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                        |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DAL-SB-605R-0-0005<br>(C0159460 Spk K, 4 ppb Spike)    | 4                    | 6.63                                  | 10.5                               | 97           |
| DAL-SB-605R-0-0005<br>(C0159460 Spk L, 400 ppb Spike)  | 400                  | 6.63                                  | 326                                | 80           |
| DAL-SB-605R-0-0010<br>(C0159461 Spk C, 4 ppb Spike)    | 4                    | 2.17                                  | 6.00                               | 96           |
| DAL-SB-605R-0-0010<br>(C0159461 Spk D, 400 ppb Spike)  | 400                  | 2.17                                  | 281                                | 70           |
| DAL-SB-605R-0-0050<br>(C0159462 Spk E, 4 ppb Spike)    | 4                    | 0.808                                 | 3.99                               | 80           |
| DAL-SB-605R-0-0050<br>(C0159462 Spk F, 400 ppb Spike)  | 400                  | 0.808                                 | 369                                | 92           |
| DAL-SB-605R-0-0100<br>(C0159463 Spk G, 4 ppb Spike)    | 4                    | 0.627                                 | 3.67                               | 76           |
| DAL-SB-605R-0-0100<br>(C0159463 Spk H, 400 ppb Spike)  | 400                  | 0.627                                 | 286                                | 71           |
| DAL-SB-138R-0-0000<br>(C0159464 Spk I, 4 ppb Spike)    | 4                    | 23.8                                  | 29.0                               | *            |
| DAL-SB-138R-0-0000<br>(C0159464 Spk J, 400 ppb Spike)  | 400                  | 23.8                                  | 366                                | 86           |
| DAL-SB-138R-0-0005<br>(C0159466 Spk K, 4 ppb Spike)    | 4                    | 42.9                                  | 48.7                               | *            |
| DAL-SB-138R-0-0005<br>(C0159466 Spk L, 400 ppb Spike)  | 400                  | 42.9                                  | 407                                | 91           |
| DAL-SB-138R-0-0010<br>(C0159467 Spk C, 4 ppb Spike)    | 4                    | 35.0                                  | 46.7                               | *            |
| DAL-SB-138R-0-0010<br>(C0159467 Spk D, 400 ppb Spike)  | 400                  | 35.0                                  | 337                                | 76           |
| DAL-SB-138R-0-0050<br>(C0159468 Spk E, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DAL-SB-138R-0-0050<br>(C0159468 Spk F, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DAL-SB-138R-0-0100<br>(C0159470 Spk G, 4 ppb Spike)    | 4                    | 278                                   | 345                                | *            |
| DAL-SB-138R-0-0100<br>(C0159470 Spk H, 400 ppb Spike)  | 400                  | 278                                   | 546                                | 67           |
| DAL-SB-138R-DB-0100<br>(C0159471 Spk I, 4 ppb Spike)   | 4                    | 247                                   | 378                                | *            |
| DAL-SB-138R-DB-0100<br>(C0159471 Spk J, 400 ppb Spike) | 400                  | 247                                   | 543                                | 74           |
| DF05-SS-SS03-0-0000<br>(C0160871 Spk G, 4 ppb Spike)   | 4                    | NR                                    | NR                                 | NR           |
| DF05-SS-SS03-0-0000<br>(C0160871 Spk H, 400 ppb Spike) | 400                  | NR                                    | NR                                 | NR           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                      | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|---------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                         |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF05-SS-SS03-0-0003<br>(C0160872 Spk I, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF05-SS-SS03-0-0003<br>(C0160872 Spk J, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF05-SS-SS04-0-0000<br>(C0160873 Spk K, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF05-SS-SS04-0-0000<br>(C0160873 Spk L, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF05-SS-SS04-0-0003<br>(C0160874 Spk C, 4 ppb Spike)    | 4                    | 4.02                                  | 7.72                               | 93           |
| DF05-SS-SS04-0-0003<br>(C0160874 Spk D, 400 ppb Spike)  | 400                  | 4.02                                  | 333                                | 82           |
| DF05-SS-SS05-0-0000<br>(C0160875 Spk E, 4 ppb Spike)    | 4                    | 5.01                                  | 7.69                               | 67           |
| DF05-SS-SS05-0-0000<br>(C0160875 Spk F, 400 ppb Spike)  | 400                  | 5.01                                  | 310                                | 76           |
| DF05-SS-SS05-0-0003<br>(C0160876 Spk G, 4 ppb Spike)    | 4                    | 6.26                                  | 9.60                               | 84           |
| DF05-SS-SS05-0-0003<br>(C0160876 Spk H, 400 ppb Spike)  | 400                  | 6.26                                  | 314                                | 77           |
| DF05-SS-SS05-DB-0003<br>(C0160877 Spk I, 4 ppb Spike)   | 4                    | 6.44                                  | 8.56                               | 53           |
| DF05-SS-SS05-DB-0003<br>(C0160877 Spk J, 400 ppb Spike) | 400                  | 6.44                                  | 298                                | 73           |
| DF09-SS-0006-0-0000<br>(C0160878 Spk K, 4 ppb Spike)    | 4                    | 13.2                                  | 16.0                               | *            |
| DF09-SS-0006-0-0000<br>(C0160878 Spk L, 400 ppb Spike)  | 400                  | 13.2                                  | 392                                | 95           |
| DF09-SS-0006-0-0003<br>(C0160879 Spk C, 4 ppb Spike)    | 4                    | 14.2                                  | 19.8                               | *            |
| DF09-SS-0006-0-0003<br>(C0160879 Spk D, 400 ppb Spike)  | 400                  | 14.2                                  | 332                                | 79           |
| DF09-SS-0006-0-0010<br>(C0160880 Spk E, 4 ppb Spike)    | 4                    | 7.68                                  | 13.0                               | 133          |
| DF09-SS-0006-0-0010<br>(C0160880 Spk F, 400 ppb Spike)  | 400                  | 7.68                                  | 335                                | 82           |
| DF09-SS-0007-0-0000<br>(C0160881 Spk G, 4 ppb Spike)    | 4                    | 150                                   | 136                                | *            |
| DF09-SS-0007-0-0000<br>(C0160881 Spk H, 400 ppb Spike)  | 400                  | 150                                   | 357                                | 52           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                      | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|---------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                         |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF09-SS-0007-0-0003<br>(C0160882 Spk I, 4 ppb Spike)    | 4                    | 111                                   | 109                                | *            |
| DF09-SS-0007-0-0003<br>(C0160882 Spk J, 400 ppb Spike)  | 400                  | 111                                   | 319                                | 52           |
| DF09-SS-0007-0-0010<br>(C0160883 Spk K, 4 ppb Spike)    | 4                    | 41.8                                  | 44.2                               | *            |
| DF09-SS-0007-0-0010<br>(C0160883 Spk L, 400 ppb Spike)  | 400                  | 41.8                                  | 266                                | 56           |
| DF09-SS-0007-DB-0010<br>(C0160884 Spk C, 4 ppb Spike)   | 4                    | 34.8                                  | 38.7                               | *            |
| DF09-SS-0007-DB-0010<br>(C0160884 Spk D, 400 ppb Spike) | 400                  | 34.8                                  | 425                                | 98           |
| DF09-SS-0008-0-0000<br>(C0160885 Spk E, 4 ppb Spike)    | 4                    | 152                                   | 150                                | *            |
| DF09-SS-0008-0-0000<br>(C0160885 Spk F, 400 ppb Spike)  | 400                  | 152                                   | 518                                | 92           |
| DF09-SS-0008-0-0003<br>(C0160886 Spk G, 4 ppb Spike)    | 4                    | 131                                   | 120                                | *            |
| DF09-SS-0008-0-0003<br>(C0160886 Spk H, 400 ppb Spike)  | 400                  | 131                                   | 371                                | 60           |
| DF09-SS-0008-0-0010<br>(C0160887 Spk I, 4 ppb Spike)    | 4                    | 108                                   | 99.2                               | *            |
| DF09-SS-0008-0-0010<br>(C0160887 Spk J, 400 ppb Spike)  | 400                  | 108                                   | 426                                | 80           |
| DF09-SS-0009-0-0000<br>(C0160888 Spk K, 4 ppb Spike)    | 4                    | 8.88                                  | 14.2                               | 133          |
| DF09-SS-0009-0-0000<br>(C0160888 Spk L, 400 ppb Spike)  | 400                  | 8.88                                  | 435                                | 107          |
| DF09-SS-0009-0-0003<br>(C0160889 Spk C, 4 ppb Spike)    | 4                    | 10.8                                  | 15.8                               | 125          |
| DF09-SS-0009-0-0003<br>(C0160889 Spk D, 400 ppb Spike)  | 400                  | 10.8                                  | 486                                | 119          |
| DF09-SS-0009-DB-0003<br>(C0160890 Spk E, 4 ppb Spike)   | 4                    | 10.9                                  | 14.7                               | 95           |
| DF09-SS-0009-DB-0003<br>(C0160890 Spk F, 400 ppb Spike) | 400                  | 10.9                                  | 394                                | 96           |
| DF09-SS-0009-0-0010<br>(C0160891 Spk G, 4 ppb Spike)    | 4                    | 16.7                                  | 20.6                               | *            |
| DF09-SS-0009-0-0010<br>(C0160891 Spk H, 400 ppb Spike)  | 400                  | 16.7                                  | 422                                | 101          |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                      | Amount Spiked (ng/g) | Amt Found in Sample (ng/g) wet weight | C8 Acid PFOA                       |              |
|---------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                         |                      |                                       | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF09-SS-0010-0-0000<br>(C0160892 Spk I, 4 ppb Spike)    | 4                    | 638                                   | 706                                | *            |
| DF09-SS-0010-0-0000<br>(C0160892 Spk J, 400 ppb Spike)  | 400                  | 638                                   | 1070                               | 108          |
| DF09-SS-0010-0-0003<br>(C0160893 Spk K, 4 ppb Spike)    | 4                    | 386                                   | 367                                | *            |
| DF09-SS-0010-0-0003<br>(C0160893 Spk L, 400 ppb Spike)  | 400                  | 386                                   | 816                                | 108          |
| DF09-SS-0010-0-0010<br>(C0160894 Spk C, 4 ppb Spike)    | 4                    | 353                                   | 440                                | *            |
| DF09-SS-0010-0-0010<br>(C0160894 Spk D, 400 ppb Spike)  | 400                  | 353                                   | 683                                | 83           |
| DF09-SS-0011-0-0000<br>(C0160895 Spk E, 4 ppb Spike)    | 4                    | 222                                   | 234                                | *            |
| DF09-SS-0011-0-0000<br>(C0160895 Spk F, 400 ppb Spike)  | 400                  | 222                                   | 507                                | 71           |
| DF09-SS-0011-0-0003<br>(C0160896 Spk G, 4 ppb Spike)    | 4                    | 247                                   | 307                                | *            |
| DF09-SS-0011-0-0003<br>(C0160896 Spk H, 400 ppb Spike)  | 400                  | 247                                   | 550                                | 76           |
| DF09-SS-0011-0-0010<br>(C0160897 Spk I, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF09-SS-0011-0-0010<br>(C0160897 Spk J, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF09-SS-0012-0-0000<br>(C0160898 Spk K, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF09-SS-0012-0-0000<br>(C0160898 Spk L, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF09-SS-0012-DB-0000<br>(C0160899 Spk C, 4 ppb Spike)   | 4                    | 484                                   | 433                                | *            |
| DF09-SS-0012-DB-0000<br>(C0160899 Spk D, 400 ppb Spike) | 400                  | 484                                   | 764                                | 70           |
| DF09-SS-0012-0-0003<br>(C0160900 Spk E, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF09-SS-0012-0-0003<br>(C0160900 Spk F, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF09-SS-0012-0-0010<br>(C0160901 Spk G, 4 ppb Spike)    | 4                    | 418                                   | 373                                | *            |
| DF09-SS-0012-0-0010<br>(C0160901 Spk H, 400 ppb Spike)  | 400                  | 418                                   | 692                                | 69           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                     | C8 Acid PFOA         |                                       |                                    |              |
|--------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                        | Amount Spiked (ng/g) | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF09-SS-0013-0-0000<br>(C0160902 Spk I, 4 ppb Spike)   | 4                    | 18.5                                  | 23.8                               | *            |
| DF09-SS-0013-0-0000<br>(C0160902 Spk J, 400 ppb Spike) | 400                  | 18.5                                  | 342                                | 81           |
| DF09-SS-0013-0-0003<br>(C0160903 Spk K, 4 ppb Spike)   | 4                    | 22.2                                  | 23.7                               | *            |
| DF09-SS-0013-0-0003<br>(C0160903 Spk L, 400 ppb Spike) | 400                  | 22.2                                  | 377                                | 89           |
| DF09-SS-0013-0-0010<br>(C0160904 Spk C, 4 ppb Spike)   | 4                    | 43.3                                  | 46.7                               | *            |
| DF09-SS-0013-0-0010<br>(C0160904 Spk D, 400 ppb Spike) | 400                  | 43.3                                  | 289                                | 61           |
| DAL-SB-602R-0-0000<br>(C0164247 Spk K, 4 ppb Spike)    | 4                    | 4.75                                  | 7.58                               | 71           |
| DAL-SB-602R-0-0000<br>(C0164247 Spk L, 400 ppb Spike)  | 400                  | 4.75                                  | 305                                | 75           |
| DAL-SB-602R-0-0005<br>(C0164248 Spk C, 4 ppb Spike)    | 4                    | 5.69                                  | 8.80                               | 78           |
| DAL-SB-602R-0-0005<br>(C0164248 Spk D, 400 ppb Spike)  | 400                  | 5.69                                  | 327                                | 80           |
| DAL-SB-602R-0-0010<br>(C0164249 Spk E, 4 ppb Spike)    | 4                    | 3.39                                  | 6.38                               | 75           |
| DAL-SB-602R-0-0010<br>(C0164249 Spk F, 400 ppb Spike)  | 400                  | 3.39                                  | 415                                | 103          |
| DAL-SB-602R-0-0050<br>(C0164250 Spk G, 4 ppb Spike)    | 4                    | 0.630                                 | 3.41                               | 70           |
| DAL-SB-602R-0-0050<br>(C0164250 Spk H, 400 ppb Spike)  | 400                  | 0.630                                 | 304                                | 76           |
| DAL-SB-602R-0-0100<br>(C0164251 Spk I, 4 ppb Spike)    | 4                    | 0.555                                 | 3.00                               | 61           |
| DAL-SB-602R-0-0100<br>(C0164251 Spk J, 400 ppb Spike)  | 400                  | 0.555                                 | 277                                | 69           |
| DAL-SB-601R-0-0000<br>(C0164252 Spk K, 4 ppb Spike)    | 4                    | 1.57                                  | 4.51                               | 74           |
| DAL-SB-601R-0-0000<br>(C0164252 Spk L, 400 ppb Spike)  | 400                  | 1.57                                  | 383                                | 95           |
| DAL-SB-601R-DB-0000<br>(C0164253 Spk C, 4 ppb Spike)   | 4                    | 1.95                                  | 5.52                               | 89           |
| DAL-SB-601R-DB-0000<br>(C0164253 Spk D, 400 ppb Spike) | 400                  | 1.95                                  | 494                                | 123          |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table IV. Matrix Spike Recovery of PFOA in Soil Samples Continued**

| Sample Description                                     | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|--------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                        |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DAL-SB-601R-0-0005<br>(C0164254 Spk E, 4 ppb Spike)    | 4                    | 1.70                                  | 5.15                               | 86           |
| DAL-SB-601R-0-0005<br>(C0164254 Spk F, 400 ppb Spike)  | 400                  | 1.70                                  | 394                                | 98           |
| DAL-SB-601R-0-0010<br>(C0164255 Spk G, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DAL-SB-601R-0-0010<br>(C0164255 Spk H, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DAL-SB-601R-DB-0010<br>(C0164256 Spk I, 4 ppb Spike)   | 4                    | 1.90                                  | 7.43                               | 138          |
| DAL-SB-601R-DB-0010<br>(C0164256 Spk J, 400 ppb Spike) | 400                  | 1.90                                  | 490                                | 122          |
| DAL-SB-601R-0-0050<br>(C0164257 Spk K, 4 ppb Spike)    | 4                    | 0.728                                 | 3.80                               | 77           |
| DAL-SB-601R-0-0050<br>(C0164257 Spk L, 400 ppb Spike)  | 400                  | 0.728                                 | 270                                | 67           |
| DAL-SB-601R-0-0100<br>(C0164259 Spk C, 4 ppb Spike)    | 4                    | 0.414                                 | 3.76                               | 84           |
| DAL-SB-601R-0-0100<br>(C0164259 Spk D, 400 ppb Spike)  | 400                  | 0.414                                 | 366                                | 91           |
| DAL-SB-601R-DB-0100<br>(C0164260 Spk E, 4 ppb Spike)   | 4                    | 0.406                                 | 3.22                               | 70           |
| DAL-SB-601R-DB-0100<br>(C0164260 Spk F, 400 ppb Spike) | 400                  | 0.406                                 | 326                                | 81           |
| Average:                                               |                      |                                       |                                    | 80           |
| Standard Deviation:                                    |                      |                                       |                                    | 19           |

\*Sample residue exceeds the spiking level significantly (3x spiking level); therefore, an accurate recovery value cannot be calculated.

ND = Not detected at or above the Limit of Quantitation (LOQ) of 0.2 ng/g (wet weight).

NR = Not Reported due to Quality Control failures, see Table V for re-extract data.

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

**Table V. Matrix Spike Recovery of PFOA in Re-extracted Soil Samples**

| Sample Description                                        | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|-----------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                           |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF13-SS-SS03-0-0000<br>(C0156344 Spk E, 400 ppb Spike)    | 400                  | 344                                   | 585                                | 60           |
| DF13-SS-SS03-0-0003<br>(C0156345 Spk G, 400 ppb Spike)    | 400                  | 422                                   | 824                                | 101          |
| DF13-SS-SS05-0-0000<br>(C0156351 Spk M, 2000 ppb Spike)   | 2000                 | 880                                   | 2120                               | 62           |
| DF13-SS-SS05-0-0003<br>(C0156352 Spk E, 2000 ppb Spike)   | 2000                 | 864                                   | 3180                               | 116          |
| DF13-SS-SS05-DB-0003<br>(C0156353 Spk G, 2000 ppb Spike)  | 2000                 | 714                                   | 1750                               | 52           |
| DF10-SS-SS01-0-0000<br>(C0156354 Spk I, 400 ppb Spike)    | 400                  | NR                                    | NR                                 | NR           |
| DF11-SS-SS01-0-0003<br>(C0156367 Spk N, 400 ppb Spike)    | 400                  | 72.7                                  | 402                                | 82           |
| DF11-SS-SS02-0-0000<br>(C0156368 Spk E, 2000 ppb Spike)   | 2000                 | NR                                    | NR                                 | NR           |
| DF11-SS-SS02-0-0003<br>(C0156370 Spk G, 800 ppb Spike)    | 800                  | 579                                   | 1140                               | 70           |
| DF11-SS-SS03-0-0000<br>(C0156371 Spk I, 400 ppb Spike)    | 400                  | 81.6                                  | 357                                | 69           |
| DF11-SS-SS03-0-0003<br>(C0156373 Spk M, 400 ppb Spike)    | 400                  | NR                                    | NR                                 | NR           |
| DF05-SS-SS02-0-0003<br>(C0156381 Spk I, 400 ppb Spike)    | 400                  | 282                                   | 510                                | 57           |
| DF12-SS-SS01-0-0003<br>(C0156383 Spk M, 400 ppb Spike)    | 400                  | 160                                   | 481                                | 80           |
| DF12-SS-SS02-0-0000<br>(C0156384 Spk E, 2000 ppb Spike)   | 2000                 | 1470                                  | 2520                               | 53           |
| DF12-SS-SS02-0-0003<br>(C0156385 Spk G, 4000 ppb Spike)   | 4000                 | 2880                                  | 5400                               | 63           |
| DF12-SS-SS03-0-0000<br>(C0156386 Spk I, 40 ppb Spike)     | 40                   | 23.5                                  | 45.7                               | 56           |
| DF12-SS-SS04-0-0000<br>(C0156388 Spk M, 8000 ppb Spike)   | 8000                 | 3260                                  | 10600                              | 92           |
| DF12-SS-SS04-0-0003<br>(C0156389 Spk E, 20,000 ppb Spike) | 20000                | 9280                                  | 29800                              | 103          |
| DF13-SS-SS01-0-0003<br>(C0156393 Spk M, 400 ppb Spike)    | 400                  | 450                                   | 794                                | 86           |
| DF13-SS-SS02-0-0000<br>(C0156394 Spk E, 4000 ppb Spike)   | 4000                 | 2540                                  | 4760                               | 56           |

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 V. Matrix Spike Recovery of PFOA in Re-extracted Soil Samples Continued**

| Sample Description                                      | Amount Spiked (ng/g) | C8 Acid PFOA                          |                                    |              |
|---------------------------------------------------------|----------------------|---------------------------------------|------------------------------------|--------------|
|                                                         |                      | Amt Found in Sample (ng/g) wet weight | Amount Recovered (ng/g) wet weight | Recovery (%) |
| DF13-SS-SS02-0-0003<br>(C0156395 Spk G, 4000 ppb Spike) | 4000                 | 4760                                  | 7920                               | 79           |
| DAL-SB-603R-0-0000<br>(C0159446 Spk E, 4 ppb Spike)     | 4                    | NR                                    | NR                                 | NR           |
| DAL-SB-603R-0-0050<br>(C0159449 Spk M, 4 ppb Spike)     | 4                    | 3.07                                  | 6.50                               | 86           |
| DAL-SB-604R-0-0000<br>(C0159453 Spk I, 4 ppb Spike)     | 4                    | 2.05                                  | 4.29                               | 56           |
| DAL-SB-604R-0-0005<br>(C0159454 Spk K, 4 ppb Spike)     | 4                    | 3.08                                  | 6.26                               | 80           |
| DAL-SB-138R-0-0050<br>(C0159468 Spk G, 400 ppb Spike)   | 400                  | 85.6                                  | 498                                | 103          |
| DF05-SS-SS03-0-0000<br>(C0160871 Spk I, 400 ppb Spike)  | 400                  | 183                                   | 442                                | 65           |
| DF05-SS-SS03-0-0003<br>(C0160872 Spk K, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF05-SS-SS04-0-0000<br>(C0160873 Spk M, 4 ppb Spike)    | 4                    | NR                                    | NR                                 | NR           |
| DF09-SS-0011-0-0010<br>(C0160897 Spk K, 400 ppb Spike)  | 400                  | 205                                   | 455                                | 63           |
| DF09-SS-0012-0-0000<br>(C0160898 Spk M, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DF09-SS-0012-0-0003<br>(C0160900 Spk G, 400 ppb Spike)  | 400                  | NR                                    | NR                                 | NR           |
| DAL-SB-601R-0-0010<br>(C0164255 Spk I, 4 ppb Spike)     | 4                    | NR                                    | NR                                 | NR           |

Average: 74  
Standard Deviation: 18

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 VI. Matrix Spike Recovery of PFOA in Associated Trip Blanks**

| Sample Description                                         | C8 Acid PFOA         |                            |                         |              |
|------------------------------------------------------------|----------------------|----------------------------|-------------------------|--------------|
|                                                            | Amount Spiked (ng/L) | Amt Found in Sample (ng/L) | Amount Recovered (ng/L) | Recovery (%) |
| DF13-SS-TRIP1-LS-060221<br>(C0156349, 1.0 ppb Field Spike) | 1000                 | ND                         | 989                     | 99           |
| DF13-SS-TRIP1-HS-060221<br>(C0156350, 100 ppb Field Spike) | 100000               | ND                         | 96700                   | 97           |
| DAL-SB-TRIP1-LS-060302<br>(C0159444, 1.0 ppb Field Spike)  | 1000                 | ND                         | 862                     | 86           |
| DAL-SB-TRIP1-HS-060302<br>(C0159445, 100 ppb Field Spike)  | 100000               | ND                         | 81500                   | 82           |
| DF05-SS-TRIP2-LS-060308<br>(C0160868, 1.0 ppb Field Spike) | 1000                 | ND                         | 985                     | 99           |
| DF05-SS-TRIP2-HS-060308<br>(C0160869, 100 ppb Field Spike) | 100000               | ND                         | 96900                   | 97           |
| DAL-SB-TRIP-LS-060316<br>(C0164223, 1.0 ppb Field Spike)   | 1000                 | ND                         | 907                     | 91           |
| DAL-SB-TRIP-HS-060316<br>(C0164224, 10 ppb Field Spike)    | 10000                | ND                         | 8390                    | 84           |

Average: 92  
Standard Deviation: 7

ND = Not detected at or above the Limit of Quantitation (LOQ) of 25 ng/L.

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

**Table VII. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Soil Samples**

| Exygen ID      | Sample Description   | $^{13}\text{C}$ -PFOA |                                    |              |
|----------------|----------------------|-----------------------|------------------------------------|--------------|
|                |                      | Amount Spiked (ng/g)  | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0156344       | DF13-SS-SS03-0-0000  | 4                     | NR                                 | NR           |
| C0156344 Rep   | DF13-SS-SS03-0-0000  | 4                     | NR                                 | NR           |
| C0156344 Spk C | DF13-SS-SS03-0-0000  | 4                     | NR                                 | NR           |
| C0156344 Spk D | DF13-SS-SS03-0-0000  | 400                   | NR                                 | NR           |
| C0156345       | DF13-SS-SS03-0-0003  | 4                     | NR                                 | NR           |
| C0156345 Rep   | DF13-SS-SS03-0-0003  | 4                     | NR                                 | NR           |
| C0156345 Spk E | DF13-SS-SS03-0-0003  | 4                     | NR                                 | NR           |
| C0156345 Spk F | DF13-SS-SS03-0-0003  | 400                   | NR                                 | NR           |
| C0156346       | DF13-SS-SS04-0-0000  | 4                     | 4.87                               | 122          |
| C0156346 Rep   | DF13-SS-SS04-0-0000  | 4                     | 4.78                               | 120          |
| C0156346 Spk G | DF13-SS-SS04-0-0000  | 4                     | 2.28                               | 57           |
| C0156346 Spk H | DF13-SS-SS04-0-0000  | 400                   | 240                                | 60           |
| C0156347       | DF13-SS-SS04-0-0003  | 4                     | 4.18                               | 105          |
| C0156347 Rep   | DF13-SS-SS04-0-0003  | 4                     | 4.03                               | 101          |
| C0156347 Spk I | DF13-SS-SS04-0-0003  | 4                     | 2.69                               | 67           |
| C0156347 Spk J | DF13-SS-SS04-0-0003  | 400                   | 267                                | 67           |
| C0156351       | DF13-SS-SS05-0-0000  | 4                     | NR                                 | NR           |
| C0156351 Rep   | DF13-SS-SS05-0-0000  | 4                     | NR                                 | NR           |
| C0156351 Spk K | DF13-SS-SS05-0-0000  | 4                     | NR                                 | NR           |
| C0156351 Spk L | DF13-SS-SS05-0-0000  | 400                   | NR                                 | NR           |
| C0156352       | DF13-SS-SS05-0-0003  | 4                     | NR                                 | NR           |
| C0156352 Rep   | DF13-SS-SS05-0-0003  | 4                     | NR                                 | NR           |
| C0156352 Spk C | DF13-SS-SS05-0-0003  | 4                     | NR                                 | NR           |
| C0156352 Spk D | DF13-SS-SS05-0-0003  | 400                   | NR                                 | NR           |
| C0156353       | DF13-SS-SS05-DB-0003 | 4                     | NR                                 | NR           |
| C0156353 Rep   | DF13-SS-SS05-DB-0003 | 4                     | NR                                 | NR           |
| C0156353 Spk E | DF13-SS-SS05-DB-0003 | 4                     | NR                                 | NR           |
| C0156353 Spk F | DF13-SS-SS05-DB-0003 | 400                   | NR                                 | NR           |
| C0156354       | DF10-SS-SS01-0-0000  | 4                     | NR                                 | NR           |
| C0156354 Rep   | DF10-SS-SS01-0-0000  | 4                     | NR                                 | NR           |
| C0156354 Spk G | DF10-SS-SS01-0-0000  | 4                     | NR                                 | NR           |
| C0156354 Spk H | DF10-SS-SS01-0-0000  | 400                   | NR                                 | NR           |
| C0156355       | DF10-SS-SS01-0-0003  | 4                     | 3.98                               | 100          |
| C0156355 Rep   | DF10-SS-SS01-0-0003  | 4                     | 3.91                               | 98           |
| C0156355 Spk I | DF10-SS-SS01-0-0003  | 4                     | 1.90                               | 48           |
| C0156355 Spk J | DF10-SS-SS01-0-0003  | 400                   | 233                                | 58           |
| C0156356       | DF10-SS-SS02-0-0000  | 4                     | 4.49                               | 112          |
| C0156356 Rep   | DF10-SS-SS02-0-0000  | 4                     | 4.32                               | 108          |
| C0156356 Spk K | DF10-SS-SS02-0-0000  | 4                     | 2.10                               | 53           |
| C0156356 Spk L | DF10-SS-SS02-0-0000  | 400                   | 246                                | 62           |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
 NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Soil Samples  
Continued**

| Exygen ID      | Sample Description   | Amount Spiked (ng/g) | $^{13}\text{C}$ -PFOA              |              |
|----------------|----------------------|----------------------|------------------------------------|--------------|
|                |                      |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0156357       | DF10-SS-SS02-DB-0000 | 4                    | 4.90                               | 123          |
| C0156357 Rep   | DF10-SS-SS02-DB-0000 | 4                    | 4.95                               | 124          |
| C0156357 Spk C | DF10-SS-SS02-DB-0000 | 4                    | 2.43                               | 61           |
| C0156357 Spk D | DF10-SS-SS02-DB-0000 | 400                  | 250                                | 63           |
| C0156358       | DF10-SS-SS02-0-0003  | 4                    | 4.85                               | 121          |
| C0156358 Rep   | DF10-SS-SS02-0-0003  | 4                    | 4.98                               | 125          |
| C0156358 Spk E | DF10-SS-SS02-0-0003  | 4                    | 2.74                               | 69           |
| C0156358 Spk F | DF10-SS-SS02-0-0003  | 400                  | 247                                | 62           |
| C0156360       | DF10-SS-SS03-0-0000  | 4                    | 5.31                               | 133          |
| C0156360 Rep   | DF10-SS-SS03-0-0000  | 4                    | 5.25                               | 131          |
| C0156360 Spk G | DF10-SS-SS03-0-0000  | 4                    | 2.64                               | 66           |
| C0156360 Spk H | DF10-SS-SS03-0-0000  | 400                  | 235                                | 59           |
| C0156361       | DF10-SS-SS03-0-0003  | 4                    | 5.62                               | 141          |
| C0156361 Rep   | DF10-SS-SS03-0-0003  | 4                    | 5.46                               | 137          |
| C0156361 Spk I | DF10-SS-SS03-0-0003  | 4                    | 2.90                               | 73           |
| C0156361 Spk J | DF10-SS-SS03-0-0003  | 400                  | 270                                | 68           |
| C0156362       | DF10-SS-SS04-0-0000  | 4                    | 5.41                               | 135          |
| C0156362 Rep   | DF10-SS-SS04-0-0000  | 4                    | 5.17                               | 129          |
| C0156362 Spk K | DF10-SS-SS04-0-0000  | 4                    | 3.15                               | 79           |
| C0156362 Spk L | DF10-SS-SS04-0-0000  | 400                  | 253                                | 63           |
| C0156363       | DF10-SS-SS04-0-0003  | 4                    | 3.10                               | 78           |
| C0156363 Rep   | DF10-SS-SS04-0-0003  | 4                    | 2.78                               | 70           |
| C0156363 Spk C | DF10-SS-SS04-0-0003  | 4                    | 1.78                               | 45           |
| C0156363 Spk D | DF10-SS-SS04-0-0003  | 400                  | 258                                | 65           |
| C0156364       | DF10-SS-SS05-0-0000  | 4                    | 3.53                               | 88           |
| C0156364 Rep   | DF10-SS-SS05-0-0000  | 4                    | 3.28                               | 82           |
| C0156364 Spk E | DF10-SS-SS05-0-0000  | 4                    | 1.78                               | 45           |
| C0156364 Spk F | DF10-SS-SS05-0-0000  | 400                  | 243                                | 61           |
| C0156365       | DF10-SS-SS05-0-0003  | 4                    | 2.86                               | 72           |
| C0156365 Rep   | DF10-SS-SS05-0-0003  | 4                    | 2.78                               | 70           |
| C0156365 Spk G | DF10-SS-SS05-0-0003  | 4                    | 1.38                               | 35           |
| C0156365 Spk H | DF10-SS-SS05-0-0003  | 400                  | 235                                | 59           |
| C0156366       | DF11-SS-SS01-0-0000  | 4                    | 2.24                               | 56           |
| C0156366 Rep   | DF11-SS-SS01-0-0000  | 4                    | 2.90                               | 73           |
| C0156366 Spk I | DF11-SS-SS01-0-0000  | 4                    | 1.61                               | 40           |
| C0156366 Spk J | DF11-SS-SS01-0-0000  | 400                  | 250                                | 63           |
| C0156367       | DF11-SS-SS01-0-0003  | 4                    | NR                                 | NR           |
| C0156367 Rep   | DF11-SS-SS01-0-0003  | 4                    | NR                                 | NR           |
| C0156367 Spk K | DF11-SS-SS01-0-0003  | 4                    | NR                                 | NR           |
| C0156367 Spk L | DF11-SS-SS01-0-0003  | 400                  | NR                                 | NR           |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Soil Samples  
Continued**

| Exygen ID      | Sample Description   | Amount Spiked (ng/g) | $^{13}\text{C}$ -PFOA              |              |
|----------------|----------------------|----------------------|------------------------------------|--------------|
|                |                      |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0156368       | DF11-SS-SS02-0-0000  | 4                    | NR                                 | NR           |
| C0156368 Rep   | DF11-SS-SS02-0-0000  | 4                    | NR                                 | NR           |
| C0156368 Spk C | DF11-SS-SS02-0-0000  | 4                    | NR                                 | NR           |
| C0156368 Spk D | DF11-SS-SS02-0-0000  | 400                  | NR                                 | NR           |
| C0156370       | DF11-SS-SS02-0-0003  | 4                    | NR                                 | NR           |
| C0156370 Rep   | DF11-SS-SS02-0-0003  | 4                    | NR                                 | NR           |
| C0156370 Spk E | DF11-SS-SS02-0-0003  | 4                    | NR                                 | NR           |
| C0156370 Spk F | DF11-SS-SS02-0-0003  | 400                  | NR                                 | NR           |
| C0156371       | DF11-SS-SS03-0-0000  | 4                    | NR                                 | NR           |
| C0156371 Rep   | DF11-SS-SS03-0-0000  | 4                    | NR                                 | NR           |
| C0156371 Spk G | DF11-SS-SS03-0-0000  | 4                    | NR                                 | NR           |
| C0156371 Spk H | DF11-SS-SS03-0-0000  | 400                  | NR                                 | NR           |
| C0156372       | DF11-SS-SS03-DB-0000 | 4                    | 3.74                               | 94           |
| C0156372 Rep   | DF11-SS-SS03-DB-0000 | 4                    | 3.10                               | 78           |
| C0156372 Spk I | DF11-SS-SS03-DB-0000 | 4                    | 1.65                               | 41           |
| C0156372 Spk J | DF11-SS-SS03-DB-0000 | 400                  | 203                                | 51           |
| C0156373       | DF11-SS-SS03-0-0003  | 4                    | NR                                 | NR           |
| C0156373 Rep   | DF11-SS-SS03-0-0003  | 4                    | NR                                 | NR           |
| C0156373 Spk K | DF11-SS-SS03-0-0003  | 4                    | NR                                 | NR           |
| C0156373 Spk L | DF11-SS-SS03-0-0003  | 400                  | NR                                 | NR           |
| C0156374       | DF11-SS-SS04-0-0000  | 4                    | 4.80                               | 120          |
| C0156374 Rep   | DF11-SS-SS04-0-0000  | 4                    | 4.52                               | 113          |
| C0156374 Spk C | DF11-SS-SS04-0-0000  | 4                    | 2.29                               | 57           |
| C0156374 Spk D | DF11-SS-SS04-0-0000  | 400                  | 259                                | 65           |
| C0156375       | DF11-SS-SS04-0-0003  | 4                    | 4.15                               | 104          |
| C0156375 Rep   | DF11-SS-SS04-0-0003  | 4                    | 4.15                               | 104          |
| C0156375 Spk E | DF11-SS-SS04-0-0003  | 4                    | 1.77                               | 44           |
| C0156375 Spk F | DF11-SS-SS04-0-0003  | 400                  | 267                                | 67           |
| C0156376       | DF11-SS-SS05-0-0000  | 4                    | 4.66                               | 117          |
| C0156376 Rep   | DF11-SS-SS05-0-0000  | 4                    | 4.27                               | 107          |
| C0156376 Spk G | DF11-SS-SS05-0-0000  | 4                    | 2.55                               | 64           |
| C0156376 Spk H | DF11-SS-SS05-0-0000  | 400                  | 288                                | 72           |
| C0156377       | DF11-SS-SS05-0-0003  | 4                    | 4.38                               | 110          |
| C0156377 Rep   | DF11-SS-SS05-0-0003  | 4                    | 4.12                               | 103          |
| C0156377 Spk I | DF11-SS-SS05-0-0003  | 4                    | 2.22                               | 56           |
| C0156377 Spk J | DF11-SS-SS05-0-0003  | 400                  | 270                                | 68           |
| C0156378       | DF05-SS-SS01-0-0000  | 4                    | 3.74                               | 94           |
| C0156378 Rep   | DF05-SS-SS01-0-0000  | 4                    | 3.42                               | 86           |
| C0156378 Spk K | DF05-SS-SS01-0-0000  | 4                    | 1.86                               | 47           |
| C0156378 Spk L | DF05-SS-SS01-0-0000  | 400                  | 244                                | 61           |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Soil Samples  
Continued**

| Exygen ID      | Sample Description  | Amount Spiked (ng/g) | $^{13}\text{C}$ -PFOA              |              |
|----------------|---------------------|----------------------|------------------------------------|--------------|
|                |                     |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0156379       | DF05-SS-SS01-0-0003 | 4                    | 4.70                               | 118          |
| C0156379 Rep   | DF05-SS-SS01-0-0003 | 4                    | 4.59                               | 115          |
| C0156379 Spk C | DF05-SS-SS01-0-0003 | 4                    | 3.04                               | 76           |
| C0156379 Spk D | DF05-SS-SS01-0-0003 | 400                  | 278                                | 70           |
| C0156380       | DF05-SS-SS02-0-0000 | 4                    | 1.37                               | 34           |
| C0156380 Rep   | DF05-SS-SS02-0-0000 | 4                    | 1.16                               | 29           |
| C0156380 Spk E | DF05-SS-SS02-0-0000 | 4                    | 0.625                              | 16           |
| C0156380 Spk F | DF05-SS-SS02-0-0000 | 400                  | 248                                | 62           |
| C0156381       | DF05-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156381 Rep   | DF05-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156381 Spk G | DF05-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156381 Spk H | DF05-SS-SS02-0-0003 | 400                  | NR                                 | NR           |
| C0156382       | DF12-SS-SS01-0-0000 | 4                    | 3.87                               | 97           |
| C0156382 Rep   | DF12-SS-SS01-0-0000 | 4                    | 2.66                               | 67           |
| C0156382 Spk I | DF12-SS-SS01-0-0000 | 4                    | 1.90                               | 48           |
| C0156382 Spk J | DF12-SS-SS01-0-0000 | 400                  | 270                                | 68           |
| C0156383       | DF12-SS-SS01-0-0003 | 4                    | NR                                 | NR           |
| C0156383 Rep   | DF12-SS-SS01-0-0003 | 4                    | NR                                 | NR           |
| C0156383 Spk K | DF12-SS-SS01-0-0003 | 4                    | NR                                 | NR           |
| C0156383 Spk L | DF12-SS-SS01-0-0003 | 400                  | NR                                 | NR           |
| C0156384       | DF12-SS-SS02-0-0000 | 4                    | NR                                 | NR           |
| C0156384 Rep   | DF12-SS-SS02-0-0000 | 4                    | NR                                 | NR           |
| C0156384 Spk C | DF12-SS-SS02-0-0000 | 4                    | NR                                 | NR           |
| C0156384 Spk D | DF12-SS-SS02-0-0000 | 400                  | NR                                 | NR           |
| C0156385       | DF12-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156385 Rep   | DF12-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156385 Spk E | DF12-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156385 Spk F | DF12-SS-SS02-0-0003 | 400                  | NR                                 | NR           |
| C0156386       | DF12-SS-SS03-0-0000 | 4                    | NR                                 | NR           |
| C0156386 Rep   | DF12-SS-SS03-0-0000 | 4                    | NR                                 | NR           |
| C0156386 Spk G | DF12-SS-SS03-0-0000 | 4                    | NR                                 | NR           |
| C0156386 Spk H | DF12-SS-SS03-0-0000 | 400                  | NR                                 | NR           |
| C0156387       | DF12-SS-SS03-0-0003 | 4                    | 4.54                               | 114          |
| C0156387 Rep   | DF12-SS-SS03-0-0003 | 4                    | 4.29                               | 107          |
| C0156387 Spk I | DF12-SS-SS03-0-0003 | 4                    | 2.32                               | 58           |
| C0156387 Spk J | DF12-SS-SS03-0-0003 | 400                  | 2.14                               | 1            |
| C0156388       | DF12-SS-SS04-0-0000 | 4                    | NR                                 | NR           |
| C0156388 Rep   | DF12-SS-SS04-0-0000 | 4                    | NR                                 | NR           |
| C0156388 Spk K | DF12-SS-SS04-0-0000 | 4                    | NR                                 | NR           |
| C0156388 Spk L | DF12-SS-SS04-0-0000 | 400                  | NR                                 | NR           |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Soil Samples Continued**

| Exygen ID      | Sample Description  | Amount Spiked (ng/g) | $^{13}\text{C}$ -PFOA              |              |
|----------------|---------------------|----------------------|------------------------------------|--------------|
|                |                     |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0156389       | DF12-SS-SS04-0-0003 | 4                    | NR                                 | NR           |
| C0156389 Rep   | DF12-SS-SS04-0-0003 | 4                    | NR                                 | NR           |
| C0156389 Spk C | DF12-SS-SS04-0-0003 | 4                    | NR                                 | NR           |
| C0156389 Spk D | DF12-SS-SS04-0-0003 | 400                  | NR                                 | NR           |
| C0156390       | DF12-SS-SS05-0-0000 | 4                    | 2.34                               | 59           |
| C0156390 Rep   | DF12-SS-SS05-0-0000 | 4                    | 2.30                               | 58           |
| C0156390 Spk E | DF12-SS-SS05-0-0000 | 4                    | 1.57                               | 39           |
| C0156390 Spk F | DF12-SS-SS05-0-0000 | 400                  | 335                                | 84           |
| C0156391       | DF12-SS-SS05-0-0003 | 4                    | 1.71                               | 43           |
| C0156391 Rep   | DF12-SS-SS05-0-0003 | 4                    | 1.70                               | 43           |
| C0156391 Spk G | DF12-SS-SS05-0-0003 | 4                    | 0.944                              | 24           |
| C0156391 Spk H | DF12-SS-SS05-0-0003 | 400                  | 281                                | 70           |
| C0156392       | DF13-SS-SS01-0-0000 | 4                    | 0.880                              | 22           |
| C0156392 Rep   | DF13-SS-SS01-0-0000 | 4                    | 1.37                               | 34           |
| C0156392 Spk I | DF13-SS-SS01-0-0000 | 4                    | 0.547                              | 14           |
| C0156392 Spk J | DF13-SS-SS01-0-0000 | 400                  | 216                                | 54           |
| C0156393       | DF13-SS-SS01-0-0003 | 4                    | NR                                 | NR           |
| C0156393 Rep   | DF13-SS-SS01-0-0003 | 4                    | NR                                 | NR           |
| C0156393 Spk K | DF13-SS-SS01-0-0003 | 4                    | NR                                 | NR           |
| C0156393 Spk L | DF13-SS-SS01-0-0003 | 400                  | NR                                 | NR           |
| C0156394       | DF13-SS-SS02-0-0000 | 4                    | NR                                 | NR           |
| C0156394 Rep   | DF13-SS-SS02-0-0000 | 4                    | NR                                 | NR           |
| C0156394 Spk C | DF13-SS-SS02-0-0000 | 4                    | NR                                 | NR           |
| C0156394 Spk D | DF13-SS-SS02-0-0000 | 400                  | NR                                 | NR           |
| C0156395       | DF13-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156395 Rep   | DF13-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156395 Spk E | DF13-SS-SS02-0-0003 | 4                    | NR                                 | NR           |
| C0156395 Spk F | DF13-SS-SS02-0-0003 | 400                  | NR                                 | NR           |
| C0159446       | DAL-SB-603R-0-0000  | 4                    | NR                                 | NR           |
| C0159446 Rep   | DAL-SB-603R-0-0000  | 4                    | NR                                 | NR           |
| C0159446 Spk E | DAL-SB-603R-0-0000  | 4                    | NR                                 | NR           |
| C0159446 Spk F | DAL-SB-603R-0-0000  | 400                  | NR                                 | NR           |
| C0159447       | DAL-SB-603R-0-0005  | 4                    | 4.62                               | 116          |
| C0159447 Rep   | DAL-SB-603R-0-0005  | 4                    | 4.32                               | 108          |
| C0159447 Spk G | DAL-SB-603R-0-0005  | 4                    | 2.53                               | 63           |
| C0159447 Spk H | DAL-SB-603R-0-0005  | 400                  | 265                                | 66           |
| C0159448       | DAL-SB-603R-0-0010  | 4                    | 4.47                               | 112          |
| C0159448 Rep   | DAL-SB-603R-0-0010  | 4                    | 4.86                               | 122          |
| C0159448 Spk I | DAL-SB-603R-0-0010  | 4                    | 2.82                               | 71           |
| C0159448 Spk J | DAL-SB-603R-0-0010  | 400                  | 285                                | 71           |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
 NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of <sup>13</sup>C PFOA in Soil Samples  
Continued**

| Exygen<br>ID   | Sample<br>Description | Amount<br>Spiked<br>(ng/g) | <sup>13</sup> C-PFOA                        |                 |
|----------------|-----------------------|----------------------------|---------------------------------------------|-----------------|
|                |                       |                            | Amount<br>Recovered<br>Wet Weight<br>(ng/g) | Recovery<br>(%) |
| C0159449       | DAL-SB-603R-0-0050    | 4                          | NR                                          | NR              |
| C0159449 Rep   | DAL-SB-603R-0-0050    | 4                          | NR                                          | NR              |
| C0159449 Spk K | DAL-SB-603R-0-0050    | 4                          | NR                                          | NR              |
| C0159449 Spk L | DAL-SB-603R-0-0050    | 400                        | NR                                          | NR              |
| C0159450       | DAL-SB-603R-0-0100    | 4                          | 5.24                                        | 131             |
| C0159450 Rep   | DAL-SB-603R-0-0100    | 4                          | 5.04                                        | 126             |
| C0159450 Spk C | DAL-SB-603R-0-0100    | 4                          | 2.56                                        | 64              |
| C0159450 Spk D | DAL-SB-603R-0-0100    | 400                        | 296                                         | 74              |
| C0159451       | DAL-SB-603R-DB-0100   | 4                          | 4.79                                        | 120             |
| C0159451 Rep   | DAL-SB-603R-DB-0100   | 4                          | 4.85                                        | 121             |
| C0159451 Spk E | DAL-SB-603R-DB-0100   | 4                          | 2.94                                        | 74              |
| C0159451 Spk F | DAL-SB-603R-DB-0100   | 400                        | 287                                         | 72              |
| C0159453       | DAL-SB-604R-0-0000    | 4                          | NR                                          | NR              |
| C0159453 Rep   | DAL-SB-604R-0-0000    | 4                          | NR                                          | NR              |
| C0159453 Spk G | DAL-SB-604R-0-0000    | 4                          | NR                                          | NR              |
| C0159453 Spk H | DAL-SB-604R-0-0000    | 400                        | NR                                          | NR              |
| C0159454       | DAL-SB-604R-0-0005    | 4                          | NR                                          | NR              |
| C0159454 Rep   | DAL-SB-604R-0-0005    | 4                          | NR                                          | NR              |
| C0159454 Spk I | DAL-SB-604R-0-0005    | 4                          | NR                                          | NR              |
| C0159454 Spk J | DAL-SB-604R-0-0005    | 400                        | NR                                          | NR              |
| C0159455       | DAL-SB-604R-0-0010    | 4                          | 5.26                                        | 132             |
| C0159455 Rep   | DAL-SB-604R-0-0010    | 4                          | 5.32                                        | 133             |
| C0159455 Spk K | DAL-SB-604R-0-0010    | 4                          | 3.08                                        | 77              |
| C0159455 Spk L | DAL-SB-604R-0-0010    | 400                        | 348                                         | 87              |
| C0159456       | DAL-SB-604R-0-0050    | 4                          | 5.16                                        | 129             |
| C0159456 Rep   | DAL-SB-604R-0-0050    | 4                          | 5.29                                        | 132             |
| C0159456 Spk C | DAL-SB-604R-0-0050    | 4                          | 3.34                                        | 84              |
| C0159456 Spk D | DAL-SB-604R-0-0050    | 400                        | 305                                         | 76              |
| C0159457       | DAL-SB-604R-0-0100    | 4                          | 5.69                                        | 142             |
| C0159457 Rep   | DAL-SB-604R-0-0100    | 4                          | 5.14                                        | 129             |
| C0159457 Spk E | DAL-SB-604R-0-0100    | 4                          | 3.63                                        | 91              |
| C0159457 Spk F | DAL-SB-604R-0-0100    | 400                        | 314                                         | 79              |
| C0159458       | DAL-SB-605R-0-0000    | 4                          | 5.18                                        | 130             |
| C0159458 Rep   | DAL-SB-605R-0-0000    | 4                          | 5.15                                        | 129             |
| C0159458 Spk G | DAL-SB-605R-0-0000    | 4                          | 3.14                                        | 79              |
| C0159458 Spk H | DAL-SB-605R-0-0000    | 400                        | 351                                         | 88              |
| C0159459       | DAL-SB-605R-DB-0000   | 4                          | 4.76                                        | 119             |
| C0159459 Rep   | DAL-SB-605R-DB-0000   | 4                          | 5.22                                        | 131             |
| C0159459 Spk I | DAL-SB-605R-DB-0000   | 4                          | 2.95                                        | 74              |
| C0159459 Spk J | DAL-SB-605R-DB-0000   | 400                        | 362                                         | 91              |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
 NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of <sup>13</sup>C PFOA in Soil Samples  
Continued**

| Exygen ID      | Sample Description  | Amount Spiked (ng/g) | <sup>13</sup> C-PFOA               |              |
|----------------|---------------------|----------------------|------------------------------------|--------------|
|                |                     |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0159460       | DAL-SB-605R-0-0005  | 4                    | 5.60                               | 140          |
| C0159460 Rep   | DAL-SB-605R-0-0005  | 4                    | 5.37                               | 134          |
| C0159460 Spk K | DAL-SB-605R-0-0005  | 4                    | 2.59                               | 65           |
| C0159460 Spk L | DAL-SB-605R-0-0005  | 400                  | 323                                | 81           |
| C0159461       | DAL-SB-605R-0-0010  | 4                    | 5.94                               | 149          |
| C0159461 Rep   | DAL-SB-605R-0-0010  | 4                    | 5.89                               | 147          |
| C0159461 Spk C | DAL-SB-605R-0-0010  | 4                    | 2.71                               | 68           |
| C0159461 Spk D | DAL-SB-605R-0-0010  | 400                  | 278                                | 70           |
| C0159462       | DAL-SB-605R-0-0050  | 4                    | 5.54                               | 139          |
| C0159462 Rep   | DAL-SB-605R-0-0050  | 4                    | 5.53                               | 138          |
| C0159462 Spk E | DAL-SB-605R-0-0050  | 4                    | 2.98                               | 75           |
| C0159462 Spk F | DAL-SB-605R-0-0050  | 400                  | 357                                | 89           |
| C0159463       | DAL-SB-605R-0-0100  | 4                    | 5.46                               | 137          |
| C0159463 Rep   | DAL-SB-605R-0-0100  | 4                    | 5.06                               | 127          |
| C0159463 Spk G | DAL-SB-605R-0-0100  | 4                    | 2.98                               | 75           |
| C0159463 Spk H | DAL-SB-605R-0-0100  | 400                  | 276                                | 69           |
| C0159464       | DAL-SB-138R-0-0000  | 4                    | 5.45                               | 136          |
| C0159464 Rep   | DAL-SB-138R-0-0000  | 4                    | 5.52                               | 138          |
| C0159464 Spk I | DAL-SB-138R-0-0000  | 4                    | 2.93                               | 73           |
| C0159464 Spk J | DAL-SB-138R-0-0000  | 400                  | 329                                | 82           |
| C0159466       | DAL-SB-138R-0-0005  | 4                    | 4.82                               | 121          |
| C0159466 Rep   | DAL-SB-138R-0-0005  | 4                    | 4.85                               | 121          |
| C0159466 Spk K | DAL-SB-138R-0-0005  | 4                    | 2.67                               | 67           |
| C0159466 Spk L | DAL-SB-138R-0-0005  | 400                  | 359                                | 90           |
| C0159467       | DAL-SB-138R-0-0010  | 4                    | 5.58                               | 140          |
| C0159467 Rep   | DAL-SB-138R-0-0010  | 4                    | 5.35                               | 134          |
| C0159467 Spk C | DAL-SB-138R-0-0010  | 4                    | 3.03                               | 76           |
| C0159467 Spk D | DAL-SB-138R-0-0010  | 400                  | 291                                | 73           |
| C0159468       | DAL-SB-138R-0-0050  | 4                    | NR                                 | NR           |
| C0159468 Rep   | DAL-SB-138R-0-0050  | 4                    | NR                                 | NR           |
| C0159468 Spk E | DAL-SB-138R-0-0050  | 4                    | NR                                 | NR           |
| C0159468 Spk F | DAL-SB-138R-0-0050  | 400                  | NR                                 | NR           |
| C0159470       | DAL-SB-138R-0-0100  | 4                    | 4.54                               | 114          |
| C0159470 Rep   | DAL-SB-138R-0-0100  | 4                    | 5.06                               | 127          |
| C0159470 Spk G | DAL-SB-138R-0-0100  | 4                    | 2.78                               | 70           |
| C0159470 Spk H | DAL-SB-138R-0-0100  | 400                  | 274                                | 69           |
| C0159471       | DAL-SB-138R-DB-0100 | 4                    | 4.40                               | 110          |
| C0159471 Rep   | DAL-SB-138R-DB-0100 | 4                    | 4.19                               | 105          |
| C0159471 Spk I | DAL-SB-138R-DB-0100 | 4                    | 2.41                               | 60           |
| C0159471 Spk J | DAL-SB-138R-DB-0100 | 400                  | 328                                | 82           |
| C0160871       | DF05-SS-SS03-0-0000 | 4                    | NR                                 | NR           |
| C0160871 Rep   | DF05-SS-SS03-0-0000 | 4                    | NR                                 | NR           |
| C0160871 Spk G | DF05-SS-SS03-0-0000 | 4                    | NR                                 | NR           |
| C0160871 Spk H | DF05-SS-SS03-0-0000 | 400                  | NR                                 | NR           |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of <sup>13</sup>C PFOA in Soil Samples  
Continued**

| Exygen ID      | Sample Description   | Amount Spiked (ng/g) | <sup>13</sup> C-PFOA               |              |
|----------------|----------------------|----------------------|------------------------------------|--------------|
|                |                      |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0160872       | DF05-SS-SS03-0-0003  | 4                    | NR                                 | NR           |
| C0160872 Rep   | DF05-SS-SS03-0-0003  | 4                    | NR                                 | NR           |
| C0160872 Spk I | DF05-SS-SS03-0-0003  | 4                    | NR                                 | NR           |
| C0160872 Spk J | DF05-SS-SS03-0-0003  | 400                  | NR                                 | NR           |
| C0160873       | DF05-SS-SS04-0-0000  | 4                    | NR                                 | NR           |
| C0160873 Rep   | DF05-SS-SS04-0-0000  | 4                    | NR                                 | NR           |
| C0160873 Spk K | DF05-SS-SS04-0-0000  | 4                    | NR                                 | NR           |
| C0160873 Spk L | DF05-SS-SS04-0-0000  | 400                  | NR                                 | NR           |
| C0160874       | DF05-SS-SS04-0-0003  | 4                    | 5.19                               | 130          |
| C0160874 Rep   | DF05-SS-SS04-0-0003  | 4                    | 5.19                               | 130          |
| C0160874 Spk C | DF05-SS-SS04-0-0003  | 4                    | 3.00                               | 75           |
| C0160874 Spk D | DF05-SS-SS04-0-0003  | 400                  | 306                                | 77           |
| C0160875       | DF05-SS-SS05-0-0000  | 4                    | 5.61                               | 140          |
| C0160875 Rep   | DF05-SS-SS05-0-0000  | 4                    | 5.34                               | 134          |
| C0160875 Spk E | DF05-SS-SS05-0-0000  | 4                    | 2.85                               | 71           |
| C0160875 Spk F | DF05-SS-SS05-0-0000  | 400                  | 294                                | 74           |
| C0160876       | DF05-SS-SS05-0-0003  | 4                    | 5.39                               | 135          |
| C0160876 Rep   | DF05-SS-SS05-0-0003  | 4                    | 5.14                               | 129          |
| C0160876 Spk G | DF05-SS-SS05-0-0003  | 4                    | 3.94                               | 99           |
| C0160876 Spk H | DF05-SS-SS05-0-0003  | 400                  | 314                                | 79           |
| C0160877       | DF05-SS-SS05-DB-0003 | 4                    | 5.38                               | 135          |
| C0160877 Rep   | DF05-SS-SS05-DB-0003 | 4                    | 4.94                               | 124          |
| C0160877 Spk I | DF05-SS-SS05-DB-0003 | 4                    | 3.09                               | 77           |
| C0160877 Spk J | DF05-SS-SS05-DB-0003 | 400                  | 291                                | 73           |
| C0160878       | DF09-SS-0006-0-0000  | 4                    | 5.23                               | 131          |
| C0160878 Rep   | DF09-SS-0006-0-0000  | 4                    | 5.21                               | 130          |
| C0160878 Spk K | DF09-SS-0006-0-0000  | 4                    | 4.02                               | 101          |
| C0160878 Spk L | DF09-SS-0006-0-0000  | 400                  | 390                                | 98           |
| C0160879       | DF09-SS-0006-0-0003  | 4                    | 6.04                               | 151          |
| C0160879 Rep   | DF09-SS-0006-0-0003  | 4                    | 5.62                               | 141          |
| C0160879 Spk I | DF09-SS-0006-0-0003  | 4                    | 3.83                               | 96           |
| C0160879 Spk J | DF09-SS-0006-0-0003  | 400                  | 348                                | 87           |
| C0160880       | DF09-SS-0006-0-0010  | 4                    | 5.81                               | 145          |
| C0160880 Rep   | DF09-SS-0006-0-0010  | 4                    | 5.58                               | 140          |
| C0160880 Spk K | DF09-SS-0006-0-0010  | 4                    | 3.00                               | 75           |
| C0160880 Spk L | DF09-SS-0006-0-0010  | 400                  | 348                                | 87           |
| C0160881       | DF09-SS-0007-0-0000  | 4                    | 2.86                               | 72           |
| C0160881 Rep   | DF09-SS-0007-0-0000  | 4                    | 2.55                               | 64           |
| C0160881 Spk C | DF09-SS-0007-0-0000  | 4                    | 1.74                               | 44           |
| C0160881 Spk D | DF09-SS-0007-0-0000  | 400                  | 261                                | 65           |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
 NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Soil Samples  
Continued**

| Exygen<br>ID   | Sample<br>Description | Amount<br>Spiked<br>(ng/g) | $^{13}\text{C}$ -PFOA                       |                 |
|----------------|-----------------------|----------------------------|---------------------------------------------|-----------------|
|                |                       |                            | Amount<br>Recovered<br>Wet Weight<br>(ng/g) | Recovery<br>(%) |
| C0160882       | DF09-SS-0007-0-0003   | 4                          | 3.07                                        | 77              |
| C0160882 Rep   | DF09-SS-0007-0-0003   | 4                          | 3.00                                        | 75              |
| C0160882 Spk E | DF09-SS-0007-0-0003   | 4                          | 1.70                                        | 43              |
| C0160882 Spk F | DF09-SS-0007-0-0003   | 400                        | 240                                         | 60              |
| C0160883       | DF09-SS-0007-0-0010   | 4                          | 4.26                                        | 107             |
| C0160883 Rep   | DF09-SS-0007-0-0010   | 4                          | 4.05                                        | 101             |
| C0160883 Spk G | DF09-SS-0007-0-0010   | 4                          | 2.24                                        | 56              |
| C0160883 Spk H | DF09-SS-0007-0-0010   | 400                        | 251                                         | 63              |
| C0160884       | DF09-SS-0007-DB-0010  | 4                          | 4.88                                        | 122             |
| C0160884 Rep   | DF09-SS-0007-DB-0010  | 4                          | 4.93                                        | 123             |
| C0160884 Spk I | DF09-SS-0007-DB-0010  | 4                          | 3.50                                        | 88              |
| C0160884 Spk J | DF09-SS-0007-DB-0010  | 400                        | 395                                         | 99              |
| C0160885       | DF09-SS-0008-0-0000   | 4                          | 2.68                                        | 67              |
| C0160885 Rep   | DF09-SS-0008-0-0000   | 4                          | 2.61                                        | 65              |
| C0160885 Spk K | DF09-SS-0008-0-0000   | 4                          | 2.03                                        | 51              |
| C0160885 Spk L | DF09-SS-0008-0-0000   | 400                        | 369                                         | 92              |
| C0160886       | DF09-SS-0008-0-0003   | 4                          | 2.10                                        | 53              |
| C0160886 Rep   | DF09-SS-0008-0-0003   | 4                          | 2.35                                        | 59              |
| C0160886 Spk C | DF09-SS-0008-0-0003   | 4                          | 1.39                                        | 35              |
| C0160886 Spk D | DF09-SS-0008-0-0003   | 400                        | 278                                         | 70              |
| C0160887       | DF09-SS-0008-0-0010   | 4                          | 2.79                                        | 70              |
| C0160887 Rep   | DF09-SS-0008-0-0010   | 4                          | 3.04                                        | 76              |
| C0160887 Spk E | DF09-SS-0008-0-0010   | 4                          | 1.84                                        | 46              |
| C0160887 Spk F | DF09-SS-0008-0-0010   | 400                        | 330                                         | 83              |
| C0160888       | DF09-SS-0009-0-0000   | 4                          | 5.10                                        | 128             |
| C0160888 Rep   | DF09-SS-0009-0-0000   | 4                          | 5.22                                        | 131             |
| C0160888 Spk G | DF09-SS-0009-0-0000   | 4                          | 3.70                                        | 93              |
| C0160888 Spk H | DF09-SS-0009-0-0000   | 400                        | 429                                         | 107             |
| C0160889       | DF09-SS-0009-0-0003   | 4                          | 5.11                                        | 128             |
| C0160889 Rep   | DF09-SS-0009-0-0003   | 4                          | 5.11                                        | 128             |
| C0160889 Spk C | DF09-SS-0009-0-0003   | 4                          | 3.46                                        | 87              |
| C0160889 Spk D | DF09-SS-0009-0-0003   | 400                        | 480                                         | 120             |
| C0160890       | DF09-SS-0009-DB-0003  | 4                          | 4.94                                        | 124             |
| C0160890 Rep   | DF09-SS-0009-DB-0003  | 4                          | 5.00                                        | 125             |
| C0160890 Spk E | DF09-SS-0009-DB-0003  | 4                          | 3.42                                        | 86              |
| C0160890 Spk F | DF09-SS-0009-DB-0003  | 400                        | 391                                         | 98              |
| C0160891       | DF09-SS-0009-0-0010   | 4                          | 5.10                                        | 128             |
| C0160891 Rep   | DF09-SS-0009-0-0010   | 4                          | 4.82                                        | 121             |
| C0160891 Spk G | DF09-SS-0009-0-0010   | 4                          | 2.92                                        | 73              |
| C0160891 Spk H | DF09-SS-0009-0-0010   | 400                        | 413                                         | 103             |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
 NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of <sup>13</sup>C PFOA in Soil Samples  
Continued**

| Exygen<br>ID   | Sample<br>Description | Amount<br>Spiked<br>(ng/g) | <sup>13</sup> C-PFOA                        |                 |
|----------------|-----------------------|----------------------------|---------------------------------------------|-----------------|
|                |                       |                            | Amount<br>Recovered<br>Wet Weight<br>(ng/g) | Recovery<br>(%) |
| C0160892       | DF09-SS-0010-0-0000   | 4                          | 3.26                                        | 82              |
| C0160892 Rep   | DF09-SS-0010-0-0000   | 4                          | 3.26                                        | 82              |
| C0160892 Spk I | DF09-SS-0010-0-0000   | 4                          | 2.14                                        | 54              |
| C0160892 Spk J | DF09-SS-0010-0-0000   | 400                        | 370                                         | 93              |
| C0160893       | DF09-SS-0010-0-0003   | 4                          | 4.02                                        | 101             |
| C0160893 Rep   | DF09-SS-0010-0-0003   | 4                          | 3.34                                        | 84              |
| C0160893 Spk K | DF09-SS-0010-0-0003   | 4                          | 2.40                                        | 60              |
| C0160893 Spk L | DF09-SS-0010-0-0003   | 400                        | 330                                         | 83              |
| C0160894       | DF09-SS-0010-0-0010   | 4                          | 3.10                                        | 78              |
| C0160894 Rep   | DF09-SS-0010-0-0010   | 4                          | 4.30                                        | 108             |
| C0160894 Spk I | DF09-SS-0010-0-0010   | 4                          | 2.44                                        | 61              |
| C0160894 Spk J | DF09-SS-0010-0-0010   | 400                        | 349                                         | 87              |
| C0160895       | DF09-SS-0011-0-0000   | 4                          | 3.73                                        | 93              |
| C0160895 Rep   | DF09-SS-0011-0-0000   | 4                          | 3.35                                        | 84              |
| C0160895 Spk K | DF09-SS-0011-0-0000   | 4                          | 2.13                                        | 53              |
| C0160895 Spk L | DF09-SS-0011-0-0000   | 400                        | 298                                         | 75              |
| C0160896       | DF09-SS-0011-0-0003   | 4                          | 3.45                                        | 86              |
| C0160896 Rep   | DF09-SS-0011-0-0003   | 4                          | 3.29                                        | 82              |
| C0160896 Spk C | DF09-SS-0011-0-0003   | 4                          | 1.64                                        | 41              |
| C0160896 Spk D | DF09-SS-0011-0-0003   | 400                        | 275                                         | 69              |
| C0160897       | DF09-SS-0011-0-0010   | 4                          | NR                                          | NR              |
| C0160897 Rep   | DF09-SS-0011-0-0010   | 4                          | NR                                          | NR              |
| C0160897 Spk E | DF09-SS-0011-0-0010   | 4                          | NR                                          | NR              |
| C0160897 Spk F | DF09-SS-0011-0-0010   | 400                        | NR                                          | NR              |
| C0160898       | DF09-SS-0012-0-0000   | 4                          | NR                                          | NR              |
| C0160898 Rep   | DF09-SS-0012-0-0000   | 4                          | NR                                          | NR              |
| C0160898 Spk G | DF09-SS-0012-0-0000   | 4                          | NR                                          | NR              |
| C0160898 Spk H | DF09-SS-0012-0-0000   | 400                        | NR                                          | NR              |
| C0160899       | DF09-SS-0012-DB-0000  | 4                          | 3.95                                        | 99              |
| C0160899 Rep   | DF09-SS-0012-DB-0000  | 4                          | 4.10                                        | 103             |
| C0160899 Spk C | DF09-SS-0012-DB-0000  | 4                          | 2.49                                        | 62              |
| C0160899 Spk D | DF09-SS-0012-DB-0000  | 400                        | 365                                         | 91              |
| C0160900       | DF09-SS-0012-0-0003   | 4                          | NR                                          | NR              |
| C0160900 Rep   | DF09-SS-0012-0-0003   | 4                          | NR                                          | NR              |
| C0160900 Spk E | DF09-SS-0012-0-0003   | 4                          | NR                                          | NR              |
| C0160900 Spk F | DF09-SS-0012-0-0003   | 400                        | NR                                          | NR              |
| C0160901       | DF09-SS-0012-0-0010   | 4                          | 4.02                                        | 101             |
| C0160901 Rep   | DF09-SS-0012-0-0010   | 4                          | 4.22                                        | 106             |
| C0160901 Spk G | DF09-SS-0012-0-0010   | 4                          | 2.15                                        | 54              |
| C0160901 Spk H | DF09-SS-0012-0-0010   | 400                        | 326                                         | 82              |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of <sup>13</sup>C PFOA in Soil Samples  
Continued**

| Exygen<br>ID   | Sample<br>Description | Amount<br>Spiked<br>(ng/g) | <sup>13</sup> C-PFOA                        |                 |
|----------------|-----------------------|----------------------------|---------------------------------------------|-----------------|
|                |                       |                            | Amount<br>Recovered<br>Wet Weight<br>(ng/g) | Recovery<br>(%) |
| C0160902       | DF09-SS-0013-0-0000   | 4                          | 4.50                                        | 113             |
| C0160902 Rep   | DF09-SS-0013-0-0000   | 4                          | 4.82                                        | 121             |
| C0160902 Spk I | DF09-SS-0013-0-0000   | 4                          | 2.89                                        | 72              |
| C0160902 Spk J | DF09-SS-0013-0-0000   | 400                        | 326                                         | 82              |
| C0160903       | DF09-SS-0013-0-0003   | 4                          | 5.14                                        | 129             |
| C0160903 Rep   | DF09-SS-0013-0-0003   | 4                          | 5.32                                        | 133             |
| C0160903 Spk K | DF09-SS-0013-0-0003   | 4                          | 2.37                                        | 59              |
| C0160903 Spk L | DF09-SS-0013-0-0003   | 400                        | 357                                         | 89              |
| C0160904       | DF09-SS-0013-0-0010   | 4                          | 4.43                                        | 111             |
| C0160904 Rep   | DF09-SS-0013-0-0010   | 4                          | 4.38                                        | 110             |
| C0160904 Spk C | DF09-SS-0013-0-0010   | 4                          | 2.22                                        | 56              |
| C0160904 Spk D | DF09-SS-0013-0-0010   | 400                        | 245                                         | 61              |
| C0164247       | DAL-SB-602R-0-0000    | 4                          | 5.40                                        | 135             |
| C0164247 Rep   | DAL-SB-602R-0-0000    | 4                          | 5.17                                        | 129             |
| C0164247 Spk K | DAL-SB-602R-0-0000    | 4                          | 2.90                                        | 73              |
| C0164247 Spk L | DAL-SB-602R-0-0000    | 400                        | 299                                         | 75              |
| C0164248       | DAL-SB-602R-0-0005    | 4                          | 5.34                                        | 134             |
| C0164248 Rep   | DAL-SB-602R-0-0005    | 4                          | 5.22                                        | 131             |
| C0164248 Spk C | DAL-SB-602R-0-0005    | 4                          | 2.86                                        | 72              |
| C0164248 Spk D | DAL-SB-602R-0-0005    | 400                        | 309                                         | 77              |
| C0164249       | DAL-SB-602R-0-0010    | 4                          | 5.89                                        | 147             |
| C0164249 Rep   | DAL-SB-602R-0-0010    | 4                          | 5.78                                        | 145             |
| C0164249 Spk E | DAL-SB-602R-0-0010    | 4                          | 4.30                                        | 108             |
| C0164249 Spk F | DAL-SB-602R-0-0010    | 400                        | 408                                         | 102             |
| C0164250       | DAL-SB-602R-0-0050    | 4                          | 5.40                                        | 135             |
| C0164250 Rep   | DAL-SB-602R-0-0050    | 4                          | 4.87                                        | 122             |
| C0164250 Spk G | DAL-SB-602R-0-0050    | 4                          | 2.69                                        | 67              |
| C0164250 Spk H | DAL-SB-602R-0-0050    | 400                        | 295                                         | 74              |
| C0164251       | DAL-SB-602R-0-0100    | 4                          | 3.97                                        | 99              |
| C0164251 Rep   | DAL-SB-602R-0-0100    | 4                          | 4.68                                        | 117             |
| C0164251 Spk I | DAL-SB-602R-0-0100    | 4                          | 2.58                                        | 65              |
| C0164251 Spk J | DAL-SB-602R-0-0100    | 400                        | 276                                         | 69              |
| C0164252       | DAL-SB-601R-0-0000    | 4                          | 5.36                                        | 134             |
| C0164252 Rep   | DAL-SB-601R-0-0000    | 4                          | 5.26                                        | 132             |
| C0164252 Spk K | DAL-SB-601R-0-0000    | 4                          | 2.72                                        | 68              |
| C0164252 Spk L | DAL-SB-601R-0-0000    | 400                        | 390                                         | 98              |
| C0164253       | DAL-SB-601R-DB-0000   | 4                          | 7.01                                        | 175             |
| C0164253 Rep   | DAL-SB-601R-DB-0000   | 4                          | 5.75                                        | 144             |
| C0164253 Spk C | DAL-SB-601R-DB-0000   | 4                          | 4.02                                        | 101             |
| C0164253 Spk D | DAL-SB-601R-DB-0000   | 400                        | 473                                         | 118             |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VII. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Soil Samples  
Continued**

| Exygen ID                  | Sample Description  | Amount Spiked (ng/g) | $^{13}\text{C}$ -PFOA              |              |
|----------------------------|---------------------|----------------------|------------------------------------|--------------|
|                            |                     |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0164254                   | DAL-SB-601R-0-0005  | 4                    | 5.88                               | 147          |
| C0164254 Rep               | DAL-SB-601R-0-0005  | 4                    | 5.60                               | 140          |
| C0164254 Spk E             | DAL-SB-601R-0-0005  | 4                    | 3.16                               | 79           |
| C0164254 Spk F             | DAL-SB-601R-0-0005  | 400                  | 397                                | 99           |
| C0164255                   | DAL-SB-601R-0-0010  | 4                    | NR                                 | NR           |
| C0164255 Rep               | DAL-SB-601R-0-0010  | 4                    | NR                                 | NR           |
| C0164255 Spk G             | DAL-SB-601R-0-0010  | 4                    | NR                                 | NR           |
| C0164255 Spk H             | DAL-SB-601R-0-0010  | 400                  | NR                                 | NR           |
| C0164256                   | DAL-SB-601R-DB-0010 | 4                    | 5.89                               | 147          |
| C0164256 Rep               | DAL-SB-601R-DB-0010 | 4                    | 5.54                               | 139          |
| C0164256 Spk I             | DAL-SB-601R-DB-0010 | 4                    | 3.70                               | 93           |
| C0164256 Spk J             | DAL-SB-601R-DB-0010 | 400                  | 499                                | 125          |
| C0164257                   | DAL-SB-601R-0-0050  | 4                    | 5.10                               | 128          |
| C0164257 Rep               | DAL-SB-601R-0-0050  | 4                    | 4.93                               | 123          |
| C0164257 Spk K             | DAL-SB-601R-0-0050  | 4                    | 2.79                               | 70           |
| C0164257 Spk L             | DAL-SB-601R-0-0050  | 400                  | 269                                | 67           |
| C0164259                   | DAL-SB-601R-0-0100  | 4                    | 5.96                               | 149          |
| C0164259 Rep               | DAL-SB-601R-0-0100  | 4                    | 5.91                               | 148          |
| C0164259 Spk C             | DAL-SB-601R-0-0100  | 4                    | 3.29                               | 82           |
| C0164259 Spk D             | DAL-SB-601R-0-0100  | 400                  | 352                                | 88           |
| C0164260                   | DAL-SB-601R-DB-0100 | 4                    | 5.78                               | 145          |
| C0164260 Rep               | DAL-SB-601R-DB-0100 | 4                    | 5.41                               | 135          |
| C0164260 Spk E             | DAL-SB-601R-DB-0100 | 4                    | 2.81                               | 70           |
| C0164260 Spk F             | DAL-SB-601R-DB-0100 | 400                  | 322                                | 81           |
| <b>Average:</b>            |                     |                      |                                    | <b>91</b>    |
| <b>Standard Deviation:</b> |                     |                      |                                    | <b>32</b>    |

Note: Since this summary table shows rounded results, recovery values may vary slightly from the values in the raw data.  
 NR = Not Reported due to Quality Control failures, see Table VIII for re-extract data.

**Table VIII. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Re-extracted Soil Samples**

| Exygen ID      | Sample Description   | Amount Spiked (ng/g) | $^{13}\text{C}$ -PFOA              |              |
|----------------|----------------------|----------------------|------------------------------------|--------------|
|                |                      |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0156344       | DF13-SS-SS03-0-0000  | 4                    | 0.639                              | 16           |
| C0156344 Spk E | DF13-SS-SS03-0-0000  | 400                  | 113                                | 28           |
| C0156345       | DF13-SS-SS03-0-0003  | 4                    | 0.268                              | 7            |
| C0156345 Spk G | DF13-SS-SS03-0-0003  | 400                  | 87.2                               | 22           |
| C0156351       | DF13-SS-SS05-0-0000  | 4                    | 0.468                              | 12           |
| C0156351 Spk M | DF13-SS-SS05-0-0000  | 2000                 | 832                                | 42           |
| C0156352       | DF13-SS-SS05-0-0003  | 4                    | 0.550                              | 14           |
| C0156352 Spk E | DF13-SS-SS05-0-0003  | 2000                 | 2170                               | 109          |
| C0156353       | DF13-SS-SS05-DB-0003 | 4                    | 0.438                              | 11           |
| C0156353 Spk G | DF13-SS-SS05-DB-0003 | 2000                 | 792                                | 40           |
| C0156354       | DF10-SS-SS01-0-0000  | 4                    | NR                                 | NR           |
| C0156354 Spk I | DF10-SS-SS01-0-0000  | 400                  | NR                                 | NR           |
| C0156367       | DF11-SS-SS01-0-0003  | 4                    | 0.928                              | 23           |
| C0156367 Spk N | DF11-SS-SS01-0-0003  | 400                  | 153                                | 38           |
| C0156368       | DF11-SS-SS02-0-0000  | 4                    | NR                                 | NR           |
| C0156368 Spk E | DF11-SS-SS02-0-0000  | 2000                 | NR                                 | NR           |
| C0156370       | DF11-SS-SS02-0-0003  | 4                    | 0.594                              | 15           |
| C0156370 Spk G | DF11-SS-SS02-0-0003  | 800                  | 425                                | 53           |
| C0156371       | DF11-SS-SS03-0-0000  | 4                    | 1.50                               | 38           |
| C0156371 Spk I | DF11-SS-SS03-0-0000  | 400                  | 215                                | 54           |
| C0156373       | DF11-SS-SS03-0-0003  | 4                    | NR                                 | NR           |
| C0156373 Spk M | DF11-SS-SS03-0-0003  | 400                  | NR                                 | NR           |
| C0156381       | DF05-SS-SS02-0-0003  | 4                    | 1.80                               | 45           |
| C0156381 Spk I | DF05-SS-SS02-0-0003  | 400                  | 654                                | 164          |
| C0156383       | DF12-SS-SS01-0-0003  | 4                    | 1.34                               | 34           |
| C0156383 Spk M | DF12-SS-SS01-0-0003  | 400                  | 214                                | 54           |
| C0156384       | DF12-SS-SS02-0-0000  | 4                    | 1.04                               | 26           |
| C0156384 Spk E | DF12-SS-SS02-0-0000  | 2000                 | 1010                               | 51           |
| C0156385       | DF12-SS-SS02-0-0003  | 4                    | 0.928                              | 23           |
| C0156385 Spk G | DF12-SS-SS02-0-0003  | 4000                 | 1810                               | 45           |
| C0156386       | DF12-SS-SS03-0-0000  | 4                    | 4.05                               | 101          |
| C0156386 Spk I | DF12-SS-SS03-0-0000  | 40                   | 21.4                               | 54           |
| C0156388       | DF12-SS-SS04-0-0000  | 4                    | 1.13                               | 28           |
| C0156388 Spk M | DF12-SS-SS04-0-0000  | 8000                 | 6380                               | 80           |
| C0156389       | DF12-SS-SS04-0-0003  | 4                    | 1.12                               | 28           |
| C0156389 Spk E | DF12-SS-SS04-0-0003  | 20000                | 23000                              | 115          |
| C0156393       | DF13-SS-SS01-0-0003  | 4                    | 1.34                               | 34           |
| C0156393 Spk M | DF13-SS-SS01-0-0003  | 400                  | 263                                | 66           |

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. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Re-extracted Soil Samples Continued**

| Exygen ID      | Sample Description  | Amount Spiked (ng/g) | $^{13}\text{C}$ -PFOA              |              |
|----------------|---------------------|----------------------|------------------------------------|--------------|
|                |                     |                      | Amount Recovered Wet Weight (ng/g) | Recovery (%) |
| C0156394       | DF13-SS-SS02-0-0000 | 4                    | 1.08                               | 27           |
| C0156394 Spk E | DF13-SS-SS02-0-0000 | 4000                 | 2160                               | 54           |
| C0156395       | DF13-SS-SS02-0-0003 | 4                    | 0.888                              | 22           |
| C0156395 Spk G | DF13-SS-SS02-0-0003 | 4000                 | 2680                               | 67           |
| C0159446       | DAL-SB-603R-0-0000  | 4                    | NR                                 | NR           |
| C0159446 Spk E | DAL-SB-603R-0-0000  | 4                    | NR                                 | NR           |
| C0159449       | DAL-SB-603R-0-0050  | 4                    | 3.42                               | 86           |
| C0159449 Spk M | DAL-SB-603R-0-0050  | 4                    | 1.79                               | 45           |
| C0159453       | DAL-SB-604R-0-0000  | 4                    | 3.06                               | 77           |
| C0159453 Spk I | DAL-SB-604R-0-0000  | 4                    | 1.54                               | 39           |
| C0159454       | DAL-SB-604R-0-0005  | 4                    | 3.18                               | 80           |
| C0159454 Spk K | DAL-SB-604R-0-0005  | 4                    | 2.46                               | 62           |
| C0159468       | DAL-SB-138R-0-0050  | 4                    | 2.40                               | 60           |
| C0159468 Spk G | DAL-SB-138R-0-0050  | 400                  | 218                                | 55           |
| C0160871       | DF05-SS-SS03-0-0000 | 4                    | 1.33                               | 33           |
| C0160871 Spk I | DF05-SS-SS03-0-0000 | 400                  | 162                                | 41           |
| C0160872       | DF05-SS-SS03-0-0003 | 4                    | NR                                 | NR           |
| C0160872 Spk K | DF05-SS-SS03-0-0003 | 400                  | NR                                 | NR           |
| C0160873       | DF05-SS-SS04-0-0000 | 4                    | NR                                 | NR           |
| C0160873 Spk M | DF05-SS-SS04-0-0000 | 4                    | NR                                 | NR           |
| C0160897       | DF09-SS-0011-0-0010 | 4                    | 2.10                               | 53           |
| C0160897 Spk K | DF09-SS-0011-0-0010 | 400                  | 154                                | 39           |
| C0160898       | DF09-SS-0012-0-0000 | 4                    | NR                                 | NR           |
| C0160898 Spk M | DF09-SS-0012-0-0000 | 400                  | NR                                 | NR           |
| C0160900       | DF09-SS-0012-0-0003 | 4                    | NR                                 | NR           |
| C0160900 Spk G | DF09-SS-0012-0-0003 | 400                  | NR                                 | NR           |
| C0164255       | DAL-SB-601R-0-0010  | 4                    | NR                                 | NR           |
| C0164255 Spk I | DAL-SB-601R-0-0010  | 4                    | NR                                 | NR           |

Average: 48  
Standard Deviation: 30

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 IX. Surrogate Spike Recovery of  $^{13}\text{C}$  PFOA in Associated Rinse Blanks and Trip Blanks**

| Exygen ID | Sample Description      | $^{13}\text{C}$ -PFOA |                         |              |
|-----------|-------------------------|-----------------------|-------------------------|--------------|
|           |                         | Amount Spiked (ng/L)  | Amount Recovered (ng/L) | Recovery (%) |
| C0156348  | DF13-SS-TRIP1-0-060221  | 500                   | 541                     | 108          |
| C0156349  | DF13-SS-TRIP1-LS-060221 | 1000                  | 875                     | 88           |
| C0156350  | DF13-SS-TRIP1-HS-060221 | 100000                | 87200                   | 87           |
| C0156359  | DF10-SS-SS03-RB-0000    | 500                   | 566                     | 113          |
| C0156369  | DF11-SS-SS02-RB-0003    | 500                   | 530                     | 106          |
| C0159443  | DAL-SB-TRIP1-0-060302   | 500                   | 595                     | 119          |
| C0159444  | DAL-SB-TRIP1-LS-060302  | 1000                  | 1010                    | 101          |
| C0159445  | DAL-SB-TRIP1-HS-060302  | 100000                | 99800                   | 100          |
| C0159452  | DAL-SB-603R-RB-0100     | 500                   | 619                     | 124          |
| C0159465  | DAL-SB-138R-RB-0000     | 500                   | 573                     | 115          |
| C0159469  | DAL-SB-138R-RB-0100     | 500                   | 601                     | 120          |
| C0160867  | DF05-SS-TRIP2-0-060308  | 500                   | 602                     | 120          |
| C0160868  | DF05-SS-TRIP2-LS-060308 | 1000                  | 883                     | 88           |
| C0160869  | DF05-SS-TRIP2-HS-060308 | 100000                | 86500                   | 87           |
| C0160870  | DF05-SS-SS03-RB-0000    | 500                   | 555                     | 111          |
| C0164222  | DAL-SB-TRIP-0-060316    | 500                   | 666                     | 133          |
| C0164223  | DAL-SB-TRIP-LS-060316   | 1000                  | 998                     | 100          |
| C0164224  | DAL-SB-TRIP-HS-060316   | 10000                 | 9150                    | 92           |
| C0164258  | DAL-SB-601R-RB-0100     | 500                   | 594                     | 119          |

Average: 107  
Standard Deviation: 14

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

**Table X. Total Percent Solids in Soil Samples**

| <b>Exygen ID</b> | <b>Sample Description</b> | <b>Total Solids (%)</b> |
|------------------|---------------------------|-------------------------|
| C0156344         | DF13-SS-SS03-0-0000       | 65.54                   |
| C0156345         | DF13-SS-SS03-0-0003       | 76.73                   |
| C0156346         | DF13-SS-SS04-0-0000       | 83.41                   |
| C0156347         | DF13-SS-SS04-0-0003       | 87.27                   |
| C0156351         | DF13-SS-SS05-0-0000       | 78.17                   |
| C0156352         | DF13-SS-SS05-0-0003       | 84.70                   |
| C0156353         | DF13-SS-SS05-DB-0003      | 83.33                   |
| C0156354         | DF10-SS-SS01-0-0000       | 84.27                   |
| C0156355         | DF10-SS-SS01-0-0003       | 86.73                   |
| C0156356         | DF10-SS-SS02-0-0000       | 86.29                   |
| C0156357         | DF10-SS-SS02-DB-0000      | 86.48                   |
| C0156358         | DF10-SS-SS02-0-0003       | 86.56                   |
| C0156360         | DF10-SS-SS03-0-0000       | 81.74                   |
| C0156361         | DF10-SS-SS03-0-0003       | 84.16                   |
| C0156362         | DF10-SS-SS04-0-0000       | 71.57                   |
| C0156363         | DF10-SS-SS04-0-0003       | 75.62                   |
| C0156364         | DF10-SS-SS05-0-0000       | 82.18                   |
| C0156365         | DF10-SS-SS05-0-0003       | 82.54                   |
| C0156366         | DF11-SS-SS01-0-0000       | 81.11                   |
| C0156367         | DF11-SS-SS01-0-0003       | 83.77                   |
| C0156368         | DF11-SS-SS02-0-0000       | 67.83                   |
| C0156370         | DF11-SS-SS02-0-0003       | 70.80                   |
| C0156371         | DF11-SS-SS03-0-0000       | 79.72                   |
| C0156372         | DF11-SS-SS03-DB-0000      | 79.82                   |
| C0156373         | DF11-SS-SS03-0-0003       | 85.23                   |
| C0156374         | DF11-SS-SS04-0-0000       | 86.67                   |
| C0156375         | DF11-SS-SS04-0-0003       | 89.38                   |
| C0156376         | DF11-SS-SS05-0-0000       | 83.65                   |
| C0156377         | DF11-SS-SS05-0-0003       | 85.17                   |

**Table X. Total Percent Solids in Soil Samples Continued**

| <b>Exygen ID</b> | <b>Sample Description</b> | <b>Total Solids (%)</b> |
|------------------|---------------------------|-------------------------|
| C0156378         | DF05-SS-SS01-0-0000       | 81.57                   |
| C0156379         | DF05-SS-SS01-0-0003       | 84.09                   |
| C0156380         | DF05-SS-SS02-0-0000       | 82.35                   |
| C0156381         | DF05-SS-SS02-0-0003       | 84.38                   |
| C0156382         | DF12-SS-SS01-0-0000       | 82.65                   |
| C0156383         | DF12-SS-SS01-0-0003       | 81.26                   |
| C0156384         | DF12-SS-SS02-0-0000       | 68.74                   |
| C0156385         | DF12-SS-SS02-0-0003       | 73.68                   |
| C0156386         | DF12-SS-SS03-0-0000       | 82.19                   |
| C0156387         | DF12-SS-SS03-0-0003       | 87.57                   |
| C0156388         | DF12-SS-SS04-0-0000       | 74.29                   |
| C0156389         | DF12-SS-SS04-0-0003       | 78.13                   |
| C0156390         | DF12-SS-SS05-0-0000       | 78.20                   |
| C0156391         | DF12-SS-SS05-0-0003       | 81.22                   |
| C0156392         | DF13-SS-SS01-0-0000       | 77.44                   |
| C0156393         | DF13-SS-SS01-0-0003       | 80.09                   |
| C0156394         | DF13-SS-SS02-0-0000       | 75.65                   |
| C0156395         | DF13-SS-SS02-0-0003       | 80.31                   |
| C0159446         | DAL-SB-603R-0-0000        | 90.63                   |
| C0159447         | DAL-SB-603R-0-0005        | 85.84                   |
| C0159448         | DAL-SB-603R-0-0010        | 81.62                   |
| C0159449         | DAL-SB-603R-0-0050        | 82.85                   |
| C0159450         | DAL-SB-603R-0-0100        | 83.40                   |
| C0159451         | DAL-SB-603R-DB-0100       | 78.33                   |
| C0159453         | DAL-SB-604R-0-0000        | 89.54                   |
| C0159454         | DAL-SB-604R-0-0005        | 82.13                   |
| C0159455         | DAL-SB-604R-0-0010        | 81.93                   |
| C0159456         | DAL-SB-604R-0-0050        | 88.21                   |

**Table X. Total Percent Solids in Soil Samples Continued**

| <b>Exygen<br/>ID</b> | <b>Sample<br/>Description</b> | <b>Total Solids (%)</b> |
|----------------------|-------------------------------|-------------------------|
| C0159457             | DAL-SB-604R-0-0100            | 77.17                   |
| C0159458             | DAL-SB-605R-0-0000            | 81.42                   |
| C0159459             | DAL-SB-605R-DB-0000           | 81.76                   |
| C0159460             | DAL-SB-605R-0-0005            | 84.78                   |
| C0159461             | DAL-SB-605R-0-0010            | 82.01                   |
| C0159462             | DAL-SB-605R-0-0050            | 79.83                   |
| C0159463             | DAL-SB-605R-0-0100            | 73.86                   |
| C0159464             | DAL-SB-138R-0-0000            | 86.23                   |
| C0159466             | DAL-SB-138R-0-0005            | 87.94                   |
| C0159467             | DAL-SB-138R-0-0010            | 86.15                   |
| C0159468             | DAL-SB-138R-0-0050            | 81.93                   |
| C0159470             | DAL-SB-138R-0-0100            | 78.61                   |
| C0159471             | DAL-SB-138R-DB-0100           | 80.36                   |
| C0160871             | DF05-SS-SS03-0-0000           | 80.74                   |
| C0160872             | DF05-SS-SS03-0-0003           | 83.20                   |
| C0160873             | DF05-SS-SS04-0-0000           | 84.22                   |
| C0160874             | DF05-SS-SS04-0-0003           | 87.24                   |
| C0160875             | DF05-SS-SS05-0-0000           | 86.54                   |
| C0160876             | DF05-SS-SS05-0-0003           | 86.68                   |
| C0160877             | DF05-SS-SS05-DB-0003          | 86.89                   |
| C0160878             | DF09-SS-0006-0-0000           | 89.09                   |
| C0160879             | DF09-SS-0006-0-0003           | 87.28                   |
| C0160880             | DF09-SS-0006-0-0010           | 86.20                   |
| C0160881             | DF09-SS-0007-0-0000           | 86.39                   |
| C0160882             | DF09-SS-0007-0-0003           | 89.57                   |
| C0160883             | DF09-SS-0007-0-0010           | 89.36                   |
| C0160884             | DF09-SS-0007-DB-0010          | 89.39                   |
| C0160885             | DF09-SS-0008-0-0000           | 86.47                   |

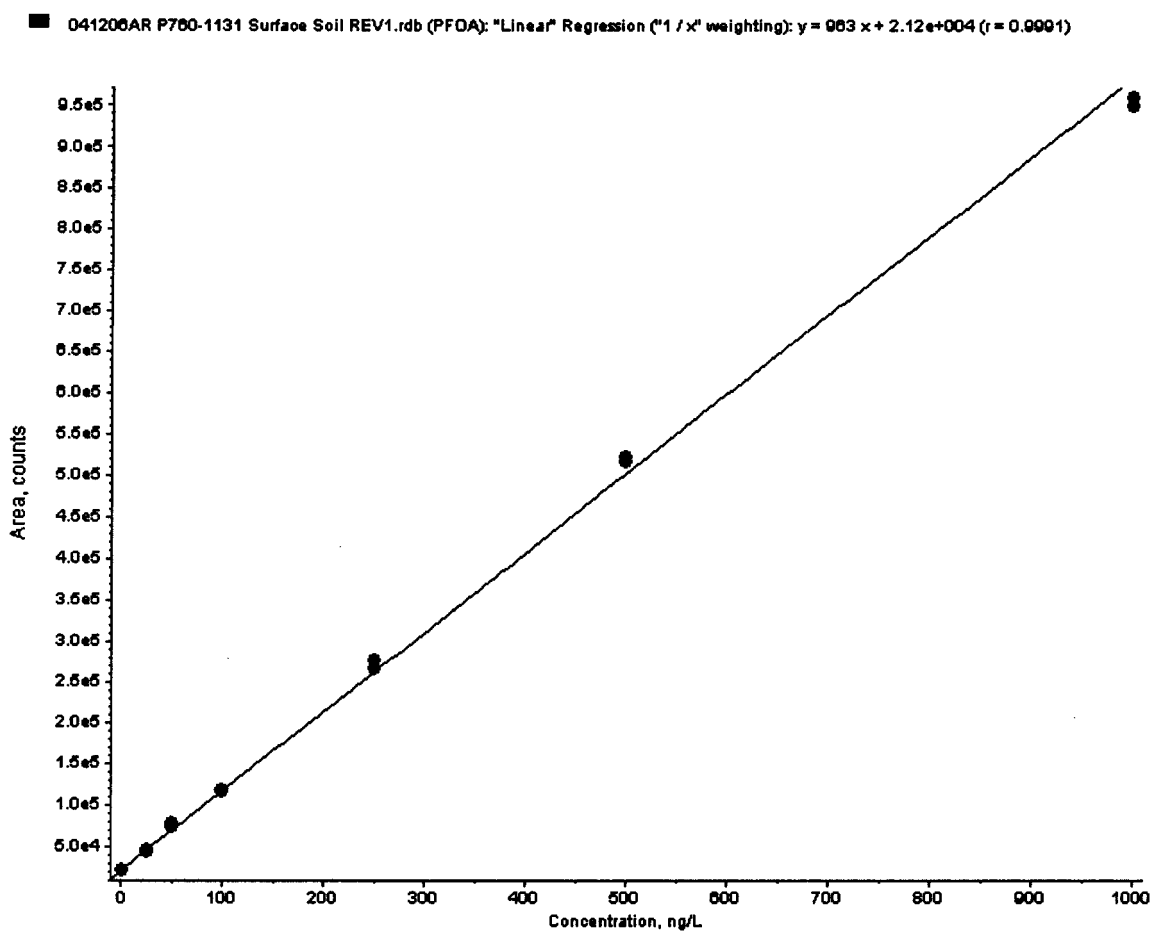
**Table X. Total Percent Solids in Soil Samples Continued**

| <b>Exygen<br/>ID</b> | <b>Sample<br/>Description</b> | <b>Total Solids (%)</b> |
|----------------------|-------------------------------|-------------------------|
| C0160886             | DF09-SS-0008-0-0003           | 88.27                   |
| C0160887             | DF09-SS-008-0-0010            | 88.54                   |
| C0160888             | DF09-SS-0009-0-0000           | 87.96                   |
| C0160889             | DF09-SS-0009-0-0003           | 88.35                   |
| C0160890             | DF09-SS-0009-DB-0003          | 88.75                   |
| C0160891             | DF09-SS-0009-0-0010           | 87.79                   |
| C0160892             | DF09-SS-0010-0-0000           | 72.09                   |
| C0160893             | DF09-SS-0010-0-0003           | 75.60                   |
| C0160894             | DF09-SS-0010-0-0010           | 76.57                   |
| C0160895             | DF09-SS-0011-0-0000           | 87.53                   |
| C0160896             | DF09-SS-0011-0-0003           | 86.92                   |
| C0160897             | DF09-SS-0011-0-0010           | 86.19                   |
| C0160898             | DF09-SS-0012-0-0000           | 90.45                   |
| C0160899             | DF09-SS-0012-DB-0000          | 90.43                   |
| C0160900             | DF09-SS-0012-0-0003           | 88.07                   |
| C0160901             | DF09-SS-0012-0-0010           | 87.02                   |
| C0160902             | DF09-SS-0013-0-0000           | 87.52                   |
| C0160903             | DF09-SS-0013-0-0003           | 87.52                   |
| C0160904             | DF09-SS-0013-0-0010           | 85.76                   |
| C0164247             | DAL-SB-602R-0-0000            | 77.82                   |
| C0164248             | DAL-SB-602R-0-0005            | 82.30                   |
| C0164249             | DAL-SB-602R-0-0010            | 84.24                   |
| C0164250             | DAL-SB-602R-0-0050            | 82.18                   |
| C0164251             | DAL-SB-602R-0-0100            | 83.52                   |
| C0164252             | DAL-SB-601R-0-0000            | 89.43                   |
| C0164253             | DAL-SB-601R-DB-0000           | 87.98                   |
| C0164254             | DAL-SB-601R-0-0005            | 88.75                   |
| C0164255             | DAL-SB-601R-0-0010            | 88.87                   |

**Table X. Total Percent Solids in Soil Samples Continued**

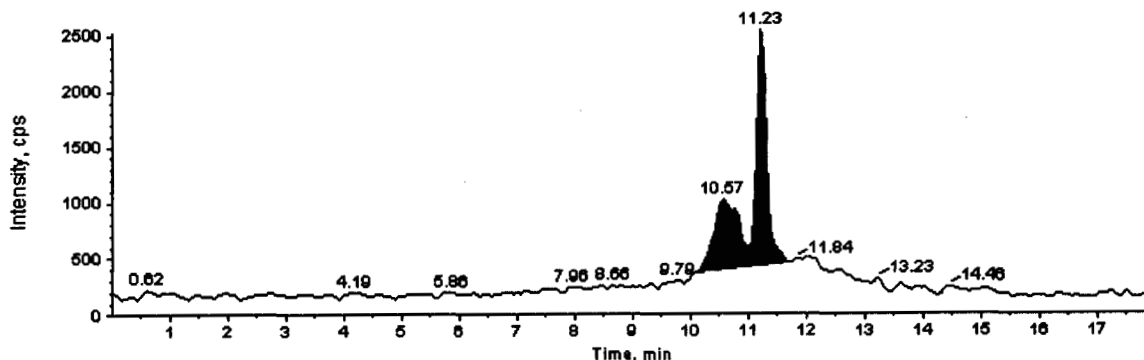
| <b>Exygen<br/>ID</b> | <b>Sample<br/>Description</b> | <b>Total Solids (%)</b> |
|----------------------|-------------------------------|-------------------------|
| C0164256             | DAL-SB-601R-DB-0010           | 87.99                   |
| C0164257             | DAL-SB-601R-0-0050            | 81.36                   |
| C0164259             | DAL-SB-601R-0-0100            | 86.85                   |
| C0164260             | DAL-SB-601R-DB-0100           | 83.56                   |

# FIGURES

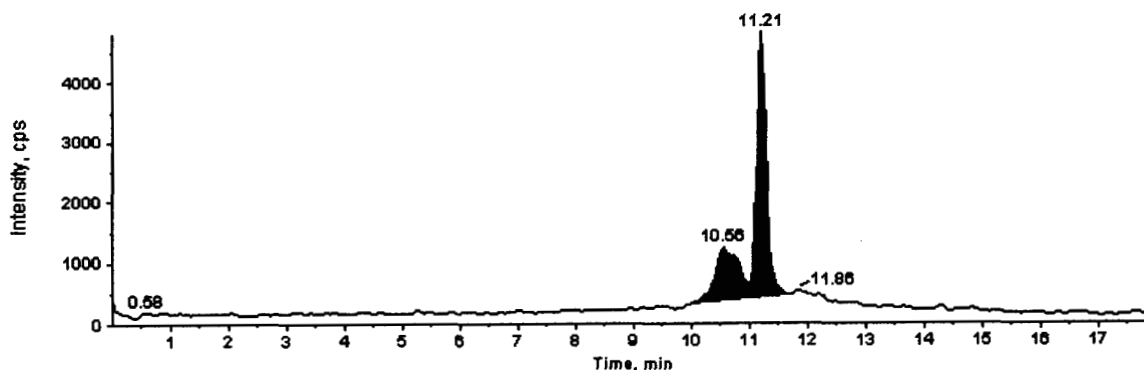
**Figure 1. Typical Calibration Curve for PFOA in Methanol**

**Figure 2. Extracted Standards of PFOA in Methanol, 25 ng/mL and 50 ng/mL, Respectively**

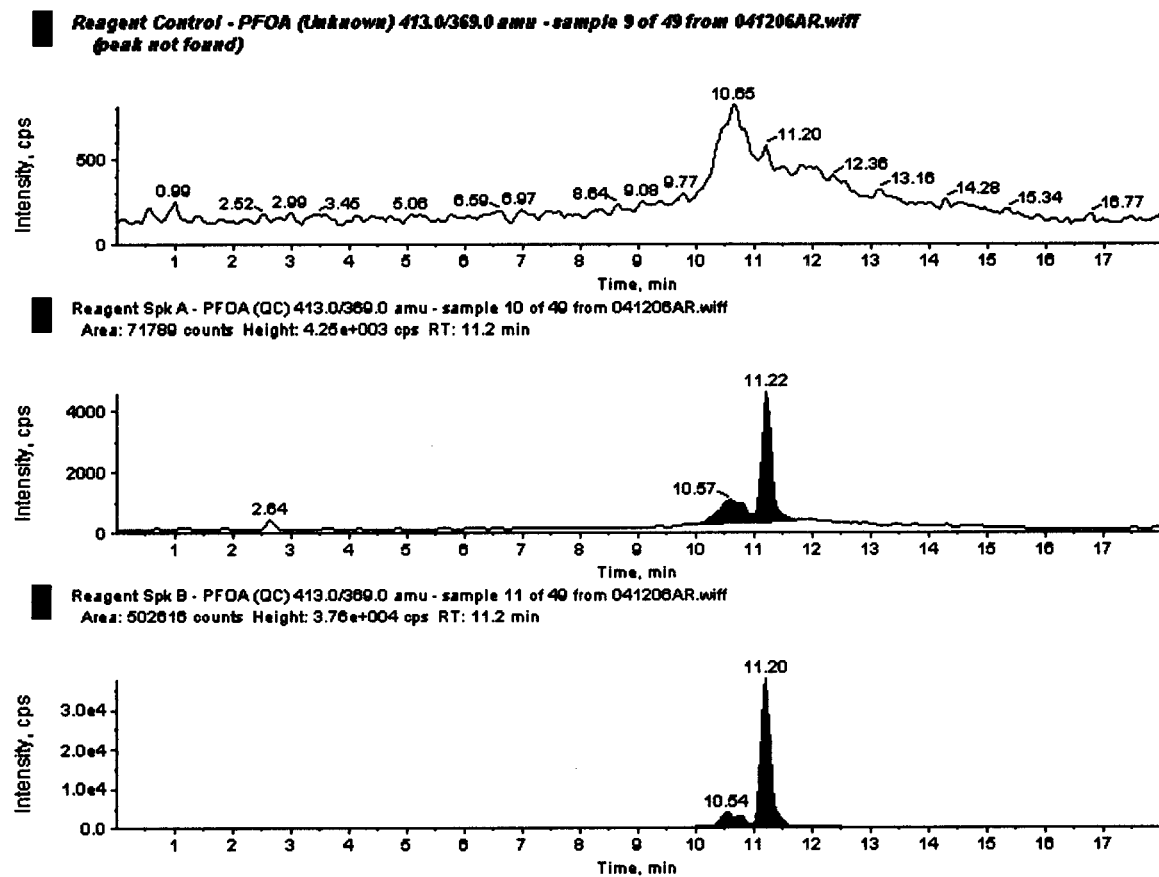
■ XC041306-1 - PFOA (Standard) 413.0/369.0 amu - sample 2 of 49 from 041206AR.wiff  
Area: 44523 counts Height: 2.15e+003 cps RT: 11.2 min



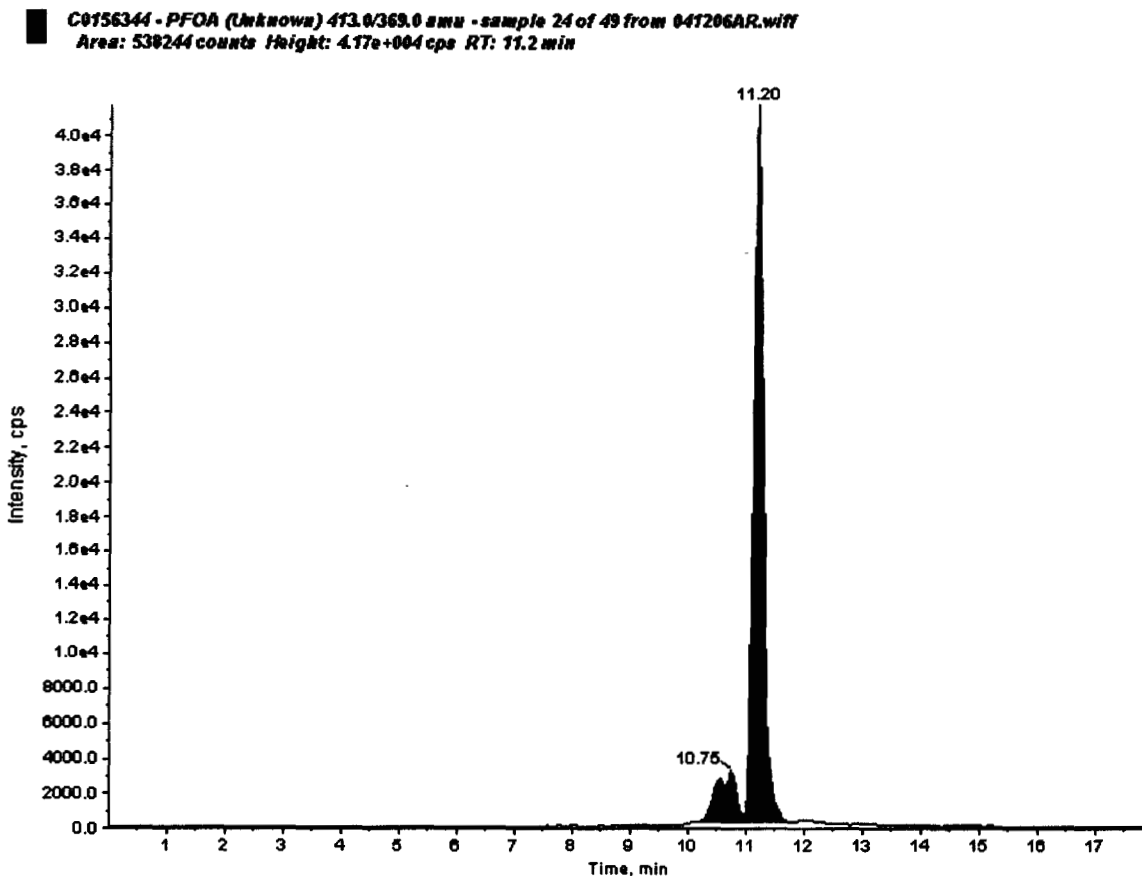
■ XC041306-2 - PFOA (Standard) 413.0/369.0 amu - sample 3 of 49 from 041206AR.wiff  
Area: 74142 counts Height: 4.40e+003 cps RT: 11.2 min

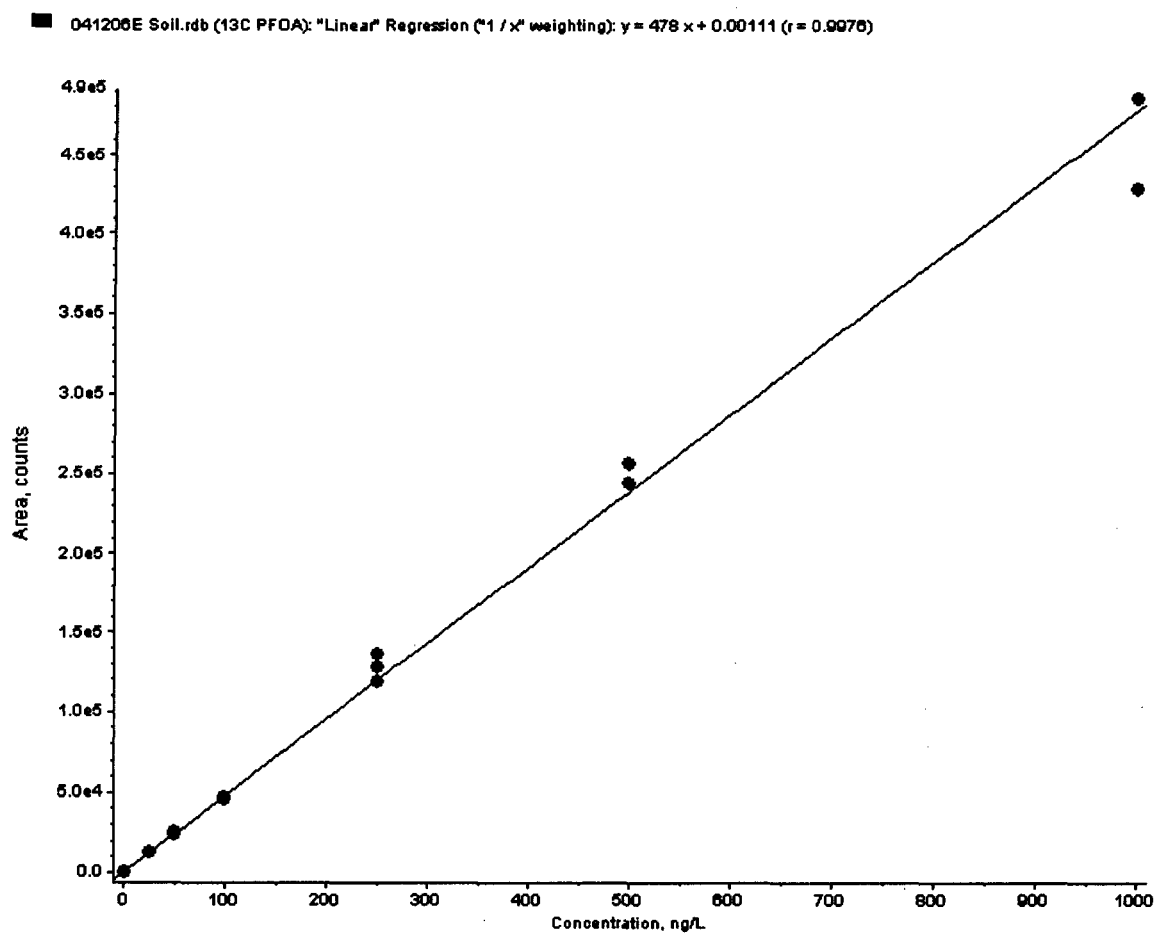


**Figure 3. PFOA in Methanol Reagent Blank, 50 ng/L Fortified Reagent Spk A, and 500 ng/L Fortified Reagent Spk B, Respectively**

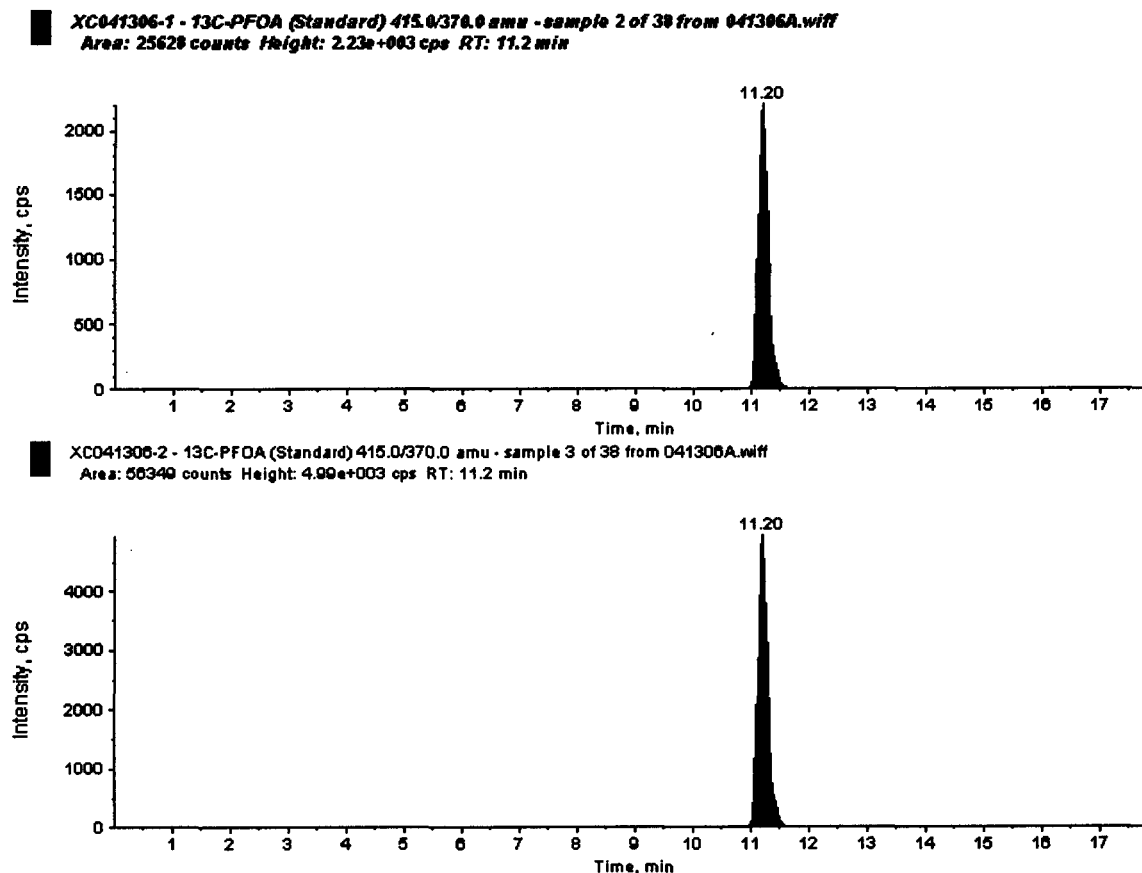


**Figure 4. Chromatogram Representing a Soil Sample Analyzed for PFOA (Exygen ID: C0156344, Data Set: 041206AR)**

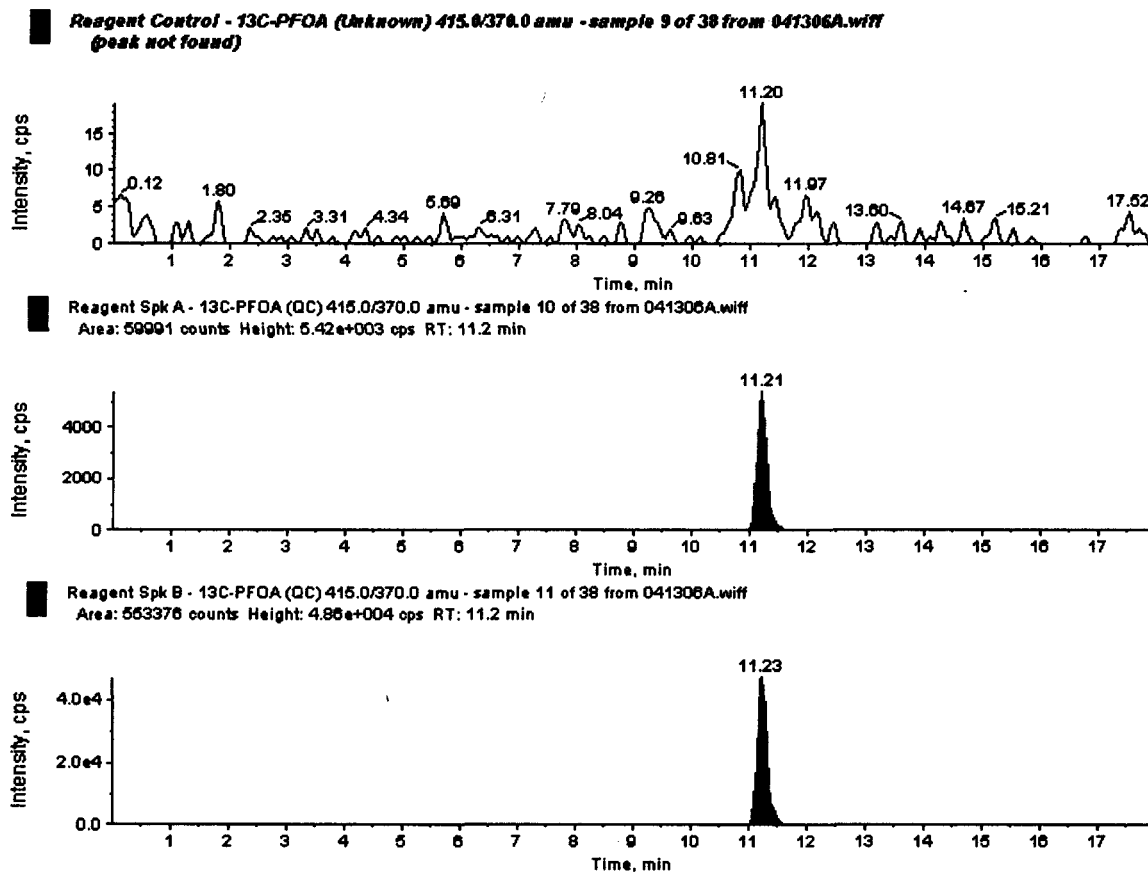


**Figure 5. Typical Calibration Curve for  $^{13}\text{C}$  PFOA in Methanol**

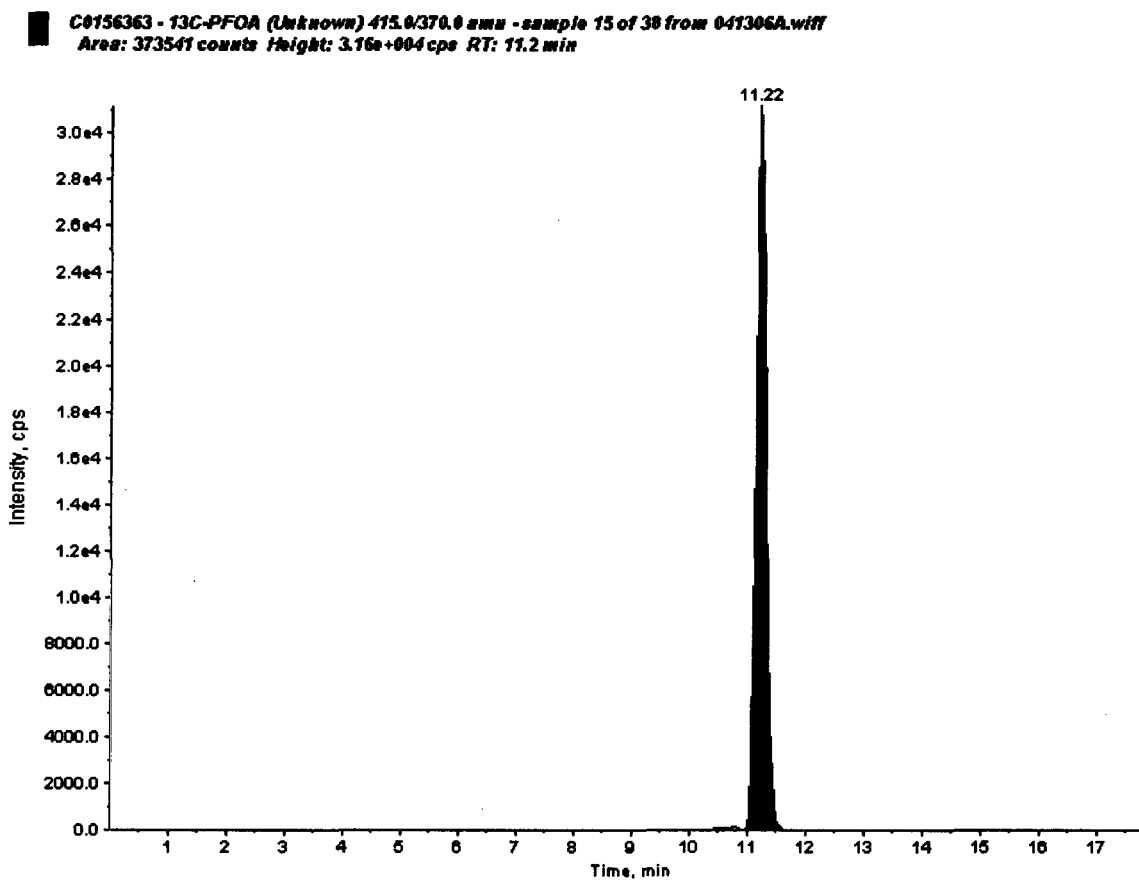
**Figure 6. Extracted Standards of  $^{13}\text{C}$  PFOA in Methanol, 25 ng/mL and 50 ng/mL, Respectively**



**Figure 7.  $^{13}\text{C}$  PFOA in Methanol Reagent Blank, 50 ng/L Fortified Reagent Spk A, and 500 ng/L Fortified Reagent Spk B, Respectively**



**Figure 8. Chromatogram Representing a Soil Sample Analyzed for  $^{13}\text{C}$  PFOA (Exygen ID: C0156363, Data Set: 041306A)**



# **APPENDIX A**

## **Study Protocol P0000760 (Exygen Study No. P0000760) with Analytical Methods and Protocol Amendments**

Exygen Protocol Number: P0000760

## STUDY PROTOCOL

### Study Title:

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

**Exygen Protocol Number: P0000760**

**Performing Laboratory:**  
Exygen Research  
3058 Research Drive  
State College, PA 16801  
Phone: (814) 272-1039

**Sponsor Representative:**  
Michael A. Santoro  
Director of Regulatory Affairs  
3M Building 0236-01-B-10  
St. Paul, MN 55144  
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Page 1 of 65

Exygen Protocol Number: P0000760

**DISTRIBUTION:**

- 1) Jaisimha Kesari, Study Director, Weston Solutions
- 2) John M. Flaherty, Principal Investigator, Exygen Research
- 3) Michael A. Santoro, Sponsor Representative, 3M Company
- 4) Exygen Research Quality Assurance Unit

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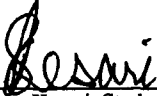
Exygen Protocol Number: P0000760

**PROTOCOL APPROVAL**

**Study Title:** Analysis of Perfluorooctanoic Acid (PFOA) in Water, Soil, Sediment, Fish, Clams, Vegetation, Small Mammal Livers and Small Mammal Serum Using LC/MS/MS for the 3M Decatur Monitoring Program

Exygen Protocol Number: P0000760

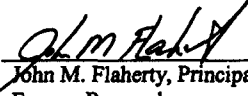
**APPROVALS**

  
\_\_\_\_\_  
Jaisimha Kesari, Study Director  
Weston Solutions

11/5/04  
\_\_\_\_\_  
Date

  
\_\_\_\_\_  
Michael A. Santoro, Sponsor Representative  
3M Company

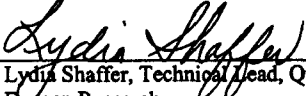
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\_\_\_\_\_  
Date

  
\_\_\_\_\_  
John M. Flaherty, Principal Investigator  
Exygen Research

10/29/04  
\_\_\_\_\_  
Date

  
\_\_\_\_\_  
Richard A. Grazzini, President, Facility Management  
Exygen Research

29-Oct-2004  
\_\_\_\_\_  
Date

  
\_\_\_\_\_  
Lydia Shaffer, Technical Lead, Quality Assurance Unit  
Exygen Research

10/29/04  
\_\_\_\_\_  
Date

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Exygen Protocol Number: P0000760

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Exygen Protocol Number: P0000760

## INTRODUCTION

The purpose of this study is to perform analysis for perfluorooctanoic acid (PFOA) in water, soil, sediment, fish, clams, vegetation, small mammal livers and small mammal serum using LC/MS/MS for the 3M Decatur Monitoring Program.

The study will be audited for compliance with EPA TSCA Good Laboratory Practice Standards 40 CFR 792 by the Quality Assurance Unit of Exygen Research.

## TEST MATERIAL

The test material is perfluorooctanoic acid (PFOA) and was purchased from Sigma-Aldrich.

### PFOA

Chemical Name: Perfluorooctanoic acid

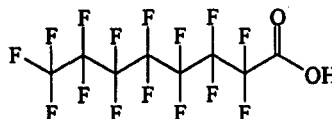
Molecular Weight: 414

Lot Number: 23116HB

Purity: 97.64%

Transitions Monitored: 413 → 369 (for quantification) and  
413 → 219 (for confirmation)

Structure:



## SURROGATE FIELD SPIKE COMPOUND

The surrogate field spiking compound is  $^{13}\text{C}$  labeled perfluorooctanoic acid ( $^{13}\text{C}$  PFOA) and was purchased by 3M from Perkin-Elmer Life and Analytical Sciences.

### $^{13}\text{C}$ PFOA

Chemical Name: 1,2- $^{13}\text{C}$  perfluorooctanoic acid

Molecular Weight: 416

Lot Number: 3507-195

Purity: 97%

Transition Monitored: 415 → 370

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## OBJECTIVE

The purpose of this study is to perform analysis for perfluorooctanoic acid (PFOA) in water, soil, sediment, fish, clams, vegetation, small mammal livers and small mammal serum for the 3M Decatur Monitoring Program using the current versions of the following Exygen analytical methods:

- V0001780: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS"
- V0001781: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS"
- V0001782: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS"
- V0001783: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS"
- V0001784: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS"
- V0001785: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS"
- V0001786: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS"

## TESTING FACILITY

Exygen Research  
3058 Research Drive  
State College, PA 16801  
Phone: (814) 272-1039

## STUDY DIRECTOR

Jaisimha Kesari P.E., DEE  
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**SPONSOR REPRESENTATIVE**

Michael A. Santoro  
3M Company  
Director of Regulatory Affairs  
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**PRINCIPAL INVESTIGATOR**

John M. Flaherty  
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**PROPOSED EXPERIMENTAL START AND TERMINATION DATES**

It is proposed that the analytical portion of this study be conducted from October 01, 2004 to December 31, 2005. The actual experimental start and termination dates will be included in the final report.

**IDENTIFICATION AND JUSTIFICATION OF THE TEST SYSTEM**

The following are the test systems for this study:

- Water (groundwater and surface water)
- Soil
- Sediment
- Fish
- Clams
- Vegetation
- Small Mammal Liver
- Small Mammal Serum

The samples will be collected by Weston Solutions. The control samples will be purchased and prepared by the testing facility. Purchase and processing

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details for the control samples will be included in the final report associated with this study.

The test systems were chosen to assess the environmental impact of PFOA in the Decatur, Alabama area.

### **SAMPLE PROCUREMENT, RECEIPT AND RETENTION**

Water, soil, sediment, fish, clam, vegetation, small mammal liver and small mammal serum samples will be received at Exygen directly from Weston Solutions. The details of sample procurement for this study are outlined in the 3M work plan entitled "Phase 2 Work Plan for Sampling Environmental Media." The number and types of samples collected will vary depending on availability in the field. The total number of samples received and analyzed for each matrix will be documented in the final report associated with this study.

Water, soil, and sediment samples will be used as received without further processing at Exygen. These samples will be stored refrigerated at 2°C-8°C. Fish, clam, vegetation and small mammal liver samples will be processed according to the appropriate analytical method (see Appendix D). These samples will be stored frozen at  $\leq -10^{\circ}\text{C}$ . Small mammal whole blood samples will be centrifuged in the field at the time of collection and the serum fraction will be used for the study. Small mammal serum will be stored frozen at  $\leq -10^{\circ}\text{C}$ .

The receipt and processing of the samples will be documented in the final report and raw data associated with the study.

### **SAMPLE IDENTIFICATION**

Prior to analysis, each sample will be assigned a laboratory sample reference number. The reference number will be unique and will distinguish each laboratory sample that is processed throughout the analytical procedure. Chromatographic data will be identified by the laboratory sample reference number.

Sample storage conditions and locations will be documented throughout the study.

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## ANALYTICAL PROCEDURE SUMMARY

### References:

- V0001780: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS"  
V0001781: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS"  
V0001782: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS"  
V0001783: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS"  
V0001784: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS"  
V0001785: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS"  
V0001786: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS"

The above methods use analytical conditions capable of separating the isomers of PFOA. The final report will include the isomers summed into total PFOA found.

## VERIFICATION OF ANALYTICAL PROCEDURE

A laboratory control sample will be used for the preparation of fortified control samples. The test substance will be made into solutions as per the method, and added to the matrices via a micropipette.

For water sampling, Exygen will supply one bottle per sample collected. The bottles will be 500 mL precleaned Sci/Spec Premier wide mouth HDPE bottles. These bottles have been routinely used for PFOA sample collection at the testing facility and have been shown to be free of PFOA. All containers used for water sample collection will be shipped to the sample location containing 100 ng of  $^{13}\text{C}$  PFOA (equivalent to 500 ng/L in the final sample). Samples will be added to each container to a volumetric fill line at 200 mL. A field duplicate, a low field spike and a high field spike of each sample will be collected. The low and high field spikes will contain PFOA as well as perfluorobutanesulfonate (PFBS), perfluorohexanesulfonate (PFHS), and perfluorooctanesulfonate (PFOS). These compounds are included in the solutions used to spike the samples. The results for PFBS, PFHS and PFOS will not be reported in this study. Exygen will supply one field blank (control water) and two field blank spikes (control water fortified with PFOA at a low

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and high level) for every twenty samples collected. At the testing facility, each water sample (excluding field duplicates and field spikes) will be extracted in duplicate and will also be fortified at a low and high concentration with PFOA and processed through the described procedure to determine method accuracy and to check for bias.

For soil, sediment, clams, and vegetation, Exygen will supply one 500 mL precleaned Sci/Spec Premier wide mouth HDPE bottle per sample collected or a zip-seal bag. All containers/bags used for sample collection will be shipped to the sample location. Samples will be added to each container or bag in the field. At the testing facility, each sample will be extracted in duplicate and will also be fortified at a known concentration with PFOA at both a low and high level and processed through the described procedure to determine method accuracy and to check for bias.  $^{13}\text{C}$ -PFOA will also be added to each sample in the laboratory prior to extraction at the level specified below.

For small mammal liver, Exygen will supply a 50 mL polypropylene centrifuge tube. For small mammal serum, Exygen will supply a collection kit for each sample containing serum separator tubes (red top), vacutainers, needle holders and needles, transfer pipettes, and polypropylene tubes. At the testing facility, each liver and serum sample will be extracted in duplicate and will also be fortified at a known concentration with PFOA at both a low and high level and processed through the described procedure to determine method accuracy and to check for bias.  $^{13}\text{C}$ -PFOA will also be added to each sample in the laboratory prior to extraction at the level specified below.

Low and high spiking levels for each matrix are defined below:

| Matrix             | Low PFOA Spiking Level | High PFOA Spiking Level | $^{13}\text{C}$ -PFOA Spiking Level |
|--------------------|------------------------|-------------------------|-------------------------------------|
| Water              | 500 ng/L               | 5000 ng/L               | 500 ng/L                            |
| Soil               | 4 ng/g                 | 40 ng/g                 | 40 ng/g                             |
| Sediment           | 4 ng/g                 | 40 ng/g                 | 40 ng/g                             |
| Fish               | 10 ng/g                | 100 ng/g                | 100 ng/g                            |
| Clams              | 10 ng/g                | 100 ng/g                | 100 ng/g                            |
| Vegetation         | 10 ng/g                | 100 ng/g                | 100 ng/g                            |
| Small Mammal Liver | 10 ng/g                | 100 ng/g                | 100 ng/g                            |
| Small Mammal Serum | 10 ng/g                | 100 ng/mL               | 100 ng/mL                           |

Recoveries are anticipated to be between 70% and 130% of the fortified levels; however, the exact precision and accuracy will be determined by the analysis of the quality control samples described above. A statement of accuracy will be included in the final report.

Prior to sample analysis, a laboratory control spike study will be conducted in water to justify the use of  $^{13}\text{C}$  PFOA results to establish precision and accuracy of PFOA samples. The following samples are to be prepared:

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- Two control water blanks
- Two control water samples fortified at 250 ng/L with <sup>13</sup>C PFOA
- Two control water samples fortified at 250 ng/L with <sup>13</sup>C PFOA and at 250 ng/L with PFOA.
- Two control water samples fortified at 5000 ng/L with <sup>13</sup>C PFOA and at 5000 ng/L with PFOA.

The above samples are to be extracted and analyzed according to method V0001780 entitled "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS."

### **METHOD FOR CONTROL OF BIAS**

Control of bias will be addressed by taking representative sub-samples from a homogeneous mixture of each matrix from untreated control samples, and by analyzing at least two levels of fortifications.

### **STATISTICAL METHODS**

Statistics will be limited to those specified in the subject methods and to the calculation of average recoveries, as applicable.

### **GLP STATEMENT**

All aspects of this study shall be performed and reported in compliance with EPA TSCA Good Laboratory Practice Standards 40 CFR 792. The final report or data package (supplied to the Sponsor) shall contain a statement that the study was conducted in compliance with current and applicable GLP standards and will outline any deviations in the study from those standards. This statement will be signed by the Study Director and Sponsor Representative.

### **REPORT**

A final report will be prepared by the principal investigator or their designee at the conclusion of the study. The report will include, but will not be limited to, the following:

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- The name and address of the Study Director, Sponsor Representative, and of the testing facility.
- A statement of GLP compliance (any related documentation, such as chain-of-custody records, must be in the study records).
- The signed and dated statement by the Exygen Research Quality Assurance Unit regarding dates of study inspections and dates findings were reported to the Study Director and Management.
- A description of the exact analytical conditions employed in the study. If the subject method was followed exactly, it is necessary to include only a copy of the analytical method. Any modifications to this method will be incorporated into the report. If the method is photo-reduced, the project number and page number must be included on each page.
- Description of the instrumentation used and operating conditions.
- All results from all sets analyzed. Control and fortified samples will be identified and the data table will include sample number and fortification level.
- Representative chromatograms for each analyte in each matrix, including chromatograms of a standard and a control sample, and a chromatogram at a fortification level. The location of the analyte peaks will be clearly identified in all chromatograms.
- All circumstances that may have affected the quality or integrity of the data will be documented in the report.
- Locations where raw data and the final report are to be archived.
- Additions or corrections to the final report shall be in the form of an amendment signed by the Study Director. The amendment shall clearly identify that part of the report that is being altered and the reasons for the alterations. The amendment will be signed and dated by the Study Director and the Sponsor Representative.

#### **SAFETY AND HEALTH**

- Laboratory personnel will practice good sanitation and health habits.
- Every reasonable precaution shall be taken to prevent inadvertent exposure of personnel and the environment to the test or reference substance(s).

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## AMENDMENTS TO PROTOCOL

All significant changes to the analytical protocol outlined here will be expressed in writing, signed and dated by the Study Director and Sponsor Representative. Amendments usually will be issued prior to initiation of study plan change. However, when a change is required without sufficient time for the issue of a written amendment, that change may be effected verbally with supporting documentation signed and dated by the Study Director and followed with a written amendment as soon as possible. In this case, the effective date of the written amendment will be the date of the documented change. Copies of the signed amendments will be appended to all distributed study plan copies. The original amendment will be maintained with the original study plan. Any deviations from the study plan or from the analytical method as provided will be documented and reported promptly to the Sponsor Representative.

## DATA RECORD KEEPING

Records to be maintained include the following (as appropriate):

- Sample tracking sheet(s)
- Sample receipt records, storage history, and chains of custody
- History and preparation of standards (stock, fortification, calibration)
- Description of any modifications to the method
- Instrument run sheets, bench-sheets or logs
- Analytical data tables
- All chromatographic and instrumental conditions
- Sample extraction and analysis dates
- A complete listing of study personnel, signatures and initials
- Chronological presentation of all study correspondence
- Any other documentation necessary for the reconstruction of the study

**Chromatograms-** All chromatograms will contain the following:

- Sample identification, injection date, arrow or other indication of the area of interest, and injection number corresponding to the run.
- Additionally, fortifications will include the amount of analyte added and the sample number of the sample that was fortified.
- Analytical standard chromatograms will additionally include the concentration (e.g.,  $\mu\text{g/mL}$ ).

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- As part of the documentation the following sheets will be included in each analytical set: a run sheet listing the samples to be run in the set, and an instrument conditions sheet describing the instrument type and operating conditions.
- All applicable requirements for reporting of study results as per 40 CFR 792.185.

## **QUALITY ASSURANCE**

The QA Unit of Exygen Research will inspect the study at intervals adequate to assure compliance with GLP's, and will report the findings of audits to the Study Director, Exygen Management, and the Sponsor Representative.

## **RETENTION OF DATA AND ARCHIVING**

All hard copy raw data, including, but not limited to, the original chromatograms, worksheets, correspondence, and results shall be included with the data package submitted to the Study Director. These will be archived with the original study plan, amendments, final report, and all pertinent information from the Sponsor.

The testing facility shall keep all electronic raw data and any instrument, equipment, and storage logs for the period of time specified in 40 CFR 792.195. An exact copy of the materials submitted to the study director will also be kept at Exygen Research.

Exygen will obtain permission from the study director before discarding or returning samples.

Exygen Protocol Number: P0000760

## APPENDIX I

### ANALYTICAL METHODS

- V0001780: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS"
- V0001781: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS"
- V0001782: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS"
- V0001783: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS"
- V0001784: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS"
- V0001785: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS"
- V0001786: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS"

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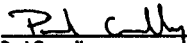
**ANALYTICAL METHOD**  
Method Number: V0001780

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**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water  
by LC/MS/MS**

Analytical Testing Facility: Exygen Research  
3058 Research Drive  
State College, PA 16801

Approved By:

  
Paul Connolly  
Technical Leader, LC-MS, Exygen Research

10/24/04  
Date

  
John Flaherty  
Vice President, Operations, Exygen Research

10/24/04  
Date

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Total Pages: 7

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Exygen Research

Method Number V0001780

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS****1.0 Scope**

This method is to be employed for the isolation and quantitation of perfluorooctanoic acid by High Performance Liquid Chromatography coupled to a tandem Mass Spectrometric Detector (LC/MS/MS) in water.

**2.0 Safety**

- 2.1 Always observe safe laboratory practices.
- 2.2 Consult the appropriate MSDS before handling any chemical for proper safety precautions.

**3.0 Sample Requirement**

- 3.1 At least 40 mL of test sample for extraction.
- 3.2 No sample processing is needed for water samples.
- 3.3 Samples stored refrigerated should be allowed to equilibrate to room temperature.
- 3.4 All samples must be thoroughly mixed before being sampled for extraction.
- 3.5 Any samples containing particles should be centrifuged at ~3000 rpm for ~5 minutes and the supernatant used for the extraction.
- 3.6 Sample collection procedures will be specified in the sampling plan for this project.

**4.0 Reagents and Standards**

- 4.1 Water - HPLC grade
- 4.2 Methanol - HPLC grade
- 4.3 Ammonium Acetate - A.C.S. Reagent Grade
- 4.4 Perfluorooctanoic Acid - Sigma-Aldrich

**5.0 Instrument and Equipment**

- 5.1 A high performance liquid chromatograph capable of pumping up to 2 solvents equipped with a variable volume injector capable of injecting 5-200  $\mu$ L connected to a tandem Mass Spectrometer (LC/MS/MS).
- 5.2 A device to collect raw data for peak integration and quantitation.
- 5.3 Analytical balance capable of reading to 0.00001 g.
- 5.4 50 mL disposable polypropylene centrifuge tubes.
- 5.5 15 mL disposable polypropylene centrifuge tubes.
- 5.6 Disposable micropipets (50-100 $\mu$ L, 100-200 $\mu$ L).
- 5.7 125-mL LDPE narrow-mouth bottles.
- 5.8 2 mL clear HPLC vial kit.
- 5.9 Disposable pipettes.
- 5.10 Autopipettes (100-1000  $\mu$ L and 10-100  $\mu$ L), with disposable tips.
- 5.11 Waters Sep Pak Vac 6 cc (1g)  $\mu$ C18 SPE cartridges.

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Exygen Research

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS

- 5.12 SPE vacuum manifold.  
5.13 Centrifuge capable of spinning 50 mL polypropylene tubes at 3000 rpm.

**6.0 Chromatographic System**

- 6.1 Analytical Column: Fluophase RP (Keystone Scientific), 2.1 mm x 50 mm, 5µ (P/N: 82505-052130)  
6.2 Temperature: 30°C  
6.3 Mobile Phase (A): 2 mM Ammonium Acetate in Water  
6.4 Mobile Phase (B): Methanol  
6.5 Gradient Program:

| Time (min) | % A | % B | Flow Rate (mL/min) |
|------------|-----|-----|--------------------|
| 0.0        | 65  | 35  | 0.3                |
| 1.0        | 65  | 35  | 0.3                |
| 8.0        | 25  | 75  | 0.3                |
| 20.0       | 25  | 75  | 0.3                |
| 22.5       | 65  | 35  | 0.3                |

- 6.6 Injection Volume: 15 µL (can be increased to as much as 50 µL).  
6.7 Quantitation: Peak Area - external standard calibration curve.  
6.8 Run Time: ~ 23 minutes.

The above conditions are intended as a guide and may be changed in order to optimize the HPLC system.

**7.0 MS/MS System**

- 7.1 Mode: Electrospray Negative MRM mode, monitoring 413 → 369 m/z.

The above conditions are intended as a guide and may be changed in order to optimize the MS/MS system.

**8.0 Preparation of Solutions****8.1 Mobile Phase**

- 8.1.1 2 mM ammonium acetate in water is prepared by adding 0.154 g of ammonium acetate to 1000 mL of water.

Alternate volumes may be prepared.

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Exygen Research

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS

**9.0 Standard Preparation****9.1 Standard Stock/Fortification Solution**

- 9.1.1 Prepare a stock solution of ~100 µg/mL of PFOA by weighing 10 mg of analytical standard (corrected for purity) and dilute to 100 mL with methanol in a 125-mL LDPE bottle.
- 9.1.2 A 10 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 100 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.3 A 1.0 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 10 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.4 A 0.1 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 1.0 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.5 A 0.01 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 0.1 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.6 The stock and fortification solutions are to be stored in a refrigerator at approximately 4°C and are stable for a maximum period of 6 months from the date of preparation.

**9.2 Standard Calibration Solutions**

- 9.2.1 LC/MS/MS calibration standards are prepared in HPLC water. The calibration standards are processed through the extraction procedure, identical to samples.
- 9.2.2 The following is a typical example: additional concentrations may be prepared as needed.

| Concentration of Fortification Solution (ppb) | Fortification Volume (µL) | Volume of Fortified Control Sample (mL) | Final Concentration of Calibration Standard (ppt)* | Calibration Standard ID (example) |
|-----------------------------------------------|---------------------------|-----------------------------------------|----------------------------------------------------|-----------------------------------|
| 0                                             | 0                         | 40                                      | 0                                                  | XCmmddy-0                         |
| 10                                            | 100                       | 40                                      | 25                                                 | XCmmddy-1                         |
| 10                                            | 200                       | 40                                      | 50                                                 | XCmmddy-2                         |
| 10                                            | 400                       | 40                                      | 100                                                | XCmmddy-3                         |
| 100                                           | 100                       | 40                                      | 250                                                | XCmmddy-4                         |
| 100                                           | 200                       | 40                                      | 500                                                | XCmmddy-5                         |
| 100                                           | 400                       | 40                                      | 1000                                               | XCmmddy-6                         |

\* The extracted concentration of the calibration standard is equal to 8x its initial concentration, due to the concentration of the standard during the extraction (SPE).  
XC = extracted calibration standard.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS

- 9.2.3 A zero standard solution (reagent blank) must be prepared with each set of standards extracted.
- 9.2.4 Store all extracted calibration standards in 15-mL polypropylene tubes at 2°C to 6°C, up to two weeks.
- 9.2.5 Alternate volumes and concentrations of standards may be prepared as needed.
- 10.0 Batch Set Up
- 10.1 Each batch of samples extracted (typically 20 or less) must include at least one reagent control (method blank using HPLC water) and two reagent controls fortified at known concentrations (lab control spike) to verify procedural recovery for the batch.
- 10.2 Requirements for field and laboratory duplicates and spikes will be specified in the quality assurance plan for this project.
- 11.0 Sample Extraction
- 11.1 Measure 40 mL of sample or a portion of sample diluted to 40 mL with water into 50 mL polypropylene centrifuge tubes (fortify as needed, replace lid and mix well).
- 11.2 Condition the C<sub>18</sub> SPE cartridges (1 g, 6 mL) by passing 10 mL methanol followed by 5 mL of HPLC water (~2 drop/sec). Do not let column run dry.
- 11.3 Load sample on conditioned C<sub>18</sub> SPE cartridge. Discard eluate.
- 11.4 Elute with ~5 mL 100% methanol. Collect 5 mL of eluate into graduated 15 mL polypropylene centrifuge tubes (final volume = 5 mL).
- 11.5 Analyze samples using electrospray LC/MS/MS.
- 12.0 Chromatography
- 12.1 Inject the same amount of each standard, sample and fortified sample into the LC/MS/MS system. A calibration standard must precede and follow all analyzed samples.
- 12.2 Standards of PFOA corresponding to at least five or more concentration levels must be included in an analytical set.
- 12.3 An entire set of extracted calibration standards must be included at the beginning and at the end of a sample set. Extracted standards must be interspersed between every 5-10 samples. As an alternative, an entire set of extracted calibration standards may be injected at the beginning of a set followed by extracted calibration standards interspersed every 5-10 samples (to account for a second set of extracted standards). In either case, extracted calibration standards must be the first and last injection in a sample set.
- 12.4 Use linear standard curves for quantitation. Linear standard curves are generated for the analyte by linear regression using 1/x weighting of peak area

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Exygen Research

Method Number V0001780

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS**

versus calibration standard concentration using MassLynx 3.3 (or equivalent) software system.

- 12.5 Sample response should not exceed standard responses. Any samples that exceed standard responses should be further diluted and reanalyzed.

**13.0 Acceptance Criteria**

- 13.1 Chromatogram must show a peak of a daughter ion at 369 amu from a parent of 413 amu. The 413 amu parent corresponds to the PFOA anion, while the daughter ion (369 amu) represents the loss of carbon dioxide.
- 13.2 Method blanks must not contain PFOA at levels greater than the LOQ. If a blank contains PFOA at levels greater than 50 ng/L, then a new blank sample must be obtained and the entire set must be re-extracted.
- 13.3 Recoveries of control spikes and matrix spikes must be between 70-130% of their known values. If a control spike falls outside the acceptable limits, the entire set of samples should be re-extracted. Any matrix spike outside 70-130% should be evaluated by the analyst to determine if re-extraction is warranted.
- 13.4 Any calibration standard found to be a statistical outlier by using the Huge Error Test, may be excluded from the calculation of the calibration curve. However, the total number of extracted calibration standards that could be excluded must not exceed 20% of the total number of extracted standards injected.
- 13.5 The correlation coefficient (R) for calibration curves generated must be  $\geq 0.992$  ( $R^2 \geq 0.985$ ). If calibration results fall outside these limits, then appropriate steps must be taken to adjust instrument operation, and the standards or the relevant set of samples should be reanalyzed.
- 13.6 Retention times between standards and samples must not drift more than  $\pm 4\%$  within an analytical run. If retention time drift exceeds this limit within an analytical run then the set must be reanalyzed.

**14.0 Calculations**

- 14.1 Use the following equation to calculate the amount of PFOA found (in ng/L, based on peak area) using the standard curve (linear regression parameters) generated by the Mass Lynx software program:

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

DF = factor by which the final volume was diluted, if necessary.

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Exygen Research

Method Number V0001780

**ANALYTICAL METHOD**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by  
LC/MS/MS

- 14.2 For samples fortified with known amounts of PFOA prior to extraction, use the following equation to calculate the percent recovery.

Recovery (%) =

$$\frac{[\text{total analyte found (ng/L)} - \text{analyte found in control (ng/L)}]}{\text{analyte added (ng/L)}} \times 100$$

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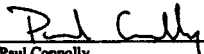
**ANALYTICAL METHOD**  
Method Number: V0001781

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**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS**

Analytical Testing Facility: Exygen Research  
3058 Research Drive  
State College, PA 16801

Approved By:

  
Paul Connolly  
Technical Leader, LC-MS, Exygen Research

10/24/04  
Date

  
John Flaherty  
Vice President, Operations, Exygen Research

10/24/04  
Date

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001781

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS****1.0 Scope**

This method is to be employed for the isolation and quantitation of perfluorooctanoic acid by High Performance Liquid Chromatography coupled to a tandem Mass Spectrometric Detector (LC/MS/MS) in soil.

**2.0 Safety**

- 2.1 Always observe safe laboratory practices.
- 2.2 Consult the appropriate MSDS before handling any chemical for proper safety precautions.

**3.0 Sample Requirement**

- 3.1 At least 15 g of test sample for extraction.
- 3.2 No sample processing is needed for soil samples.
- 3.3 Samples stored refrigerated should be allowed to equilibrate to room temperature.
- 3.4 All samples must be thoroughly mixed before being sampled for extraction.
- 3.5 Sample collection procedures will be specified in the sampling plan for this project.

**4.0 Reagents and Standards**

- 4.1 Water – HPLC grade
- 4.2 Methanol – HPLC grade
- 4.3 Ammonium Acetate – A.C.S. Reagent Grade
- 4.4 Perfluorooctanoic Acid – Sigma-Aldrich

**5.0 Instrument and Equipment**

- 5.1 A high performance liquid chromatograph capable of pumping up to 2 solvents equipped with a variable volume injector capable of injecting 5-200  $\mu$ L connected to a tandem Mass Spectrometer (LC/MS/MS).
- 5.2 A device to collect raw data for peak integration and quantitation.
- 5.3 Analytical balance capable of reading to 0.00001 g.
- 5.4 50 mL disposable polypropylene centrifuge tubes.
- 5.5 15 mL disposable polypropylene centrifuge tubes.
- 5.6 Disposable micropipets (50-100 $\mu$ L, 100-200 $\mu$ L).
- 5.7 125-mL LDPE narrow-mouth bottles.
- 5.8 2 mL clear HPLC vial kit.
- 5.9 Disposable pipettes.
- 5.10 Autopipettes (100-1000  $\mu$ L and 10-100  $\mu$ L), with disposable tips.
- 5.11 Waters Sep Pak Vac 6 cc (1g) tC18 SPE cartridges.
- 5.12 SPE vacuum manifold.
- 5.13 Ultrasonic bath.

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**ANALYTICAL METHOD**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by  
LC/MS/MS

- 5.14 Wrist-action shaker.  
5.15 Centrifuge capable of spinning 50 mL polypropylene tubes at 5000 rpm.

**6.0 Chromatographic System**

- 6.1 Analytical Column: Fluophase RP (Keystone Scientific), 2.1 mm x 50 mm, 5µ (P/N: 82505-052130)  
6.2 Temperature: 30°C  
6.3 Mobile Phase (A): 2 mM Ammonium Acetate in Water  
6.4 Mobile Phase (B): Methanol  
6.5 Gradient Program:

| Time (min) | % A | % B | Flow Rate (mL/min) |
|------------|-----|-----|--------------------|
| 0.0        | 65  | 35  | 0.3                |
| 1.0        | 65  | 35  | 0.3                |
| 8.0        | 25  | 75  | 0.3                |
| 20.0       | 25  | 75  | 0.3                |
| 22.5       | 65  | 35  | 0.3                |

- 6.6 Injection Volume: 15 µL (can be increased to as much as 50 µL).  
6.7 Quantitation: Peak Area - external standard calibration curve.  
6.8 Run Time: ~ 23 minutes.

The above conditions are intended as a guide and may be changed in order to optimize the HPLC system.

**7.0 MS/MS System**

- 7.1 Mode: Electrospray Negative MRM mode, monitoring 413 → 369 m/z for PFOA.

The above conditions are intended as a guide and may be changed in order to optimize the MSMS system.

**8.0 Preparation of Solutions****8.1 Mobile Phase**

- 8.1.1 2 mM ammonium acetate in water is prepared by adding 0.154 g of ammonium acetate to 1000 mL of water.

Alternate volumes may be prepared.

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Exygen Protocol Number: P0000760

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**ANALYTICAL METHOD**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by  
LC/MS/MS**9.0 Standard Preparation****9.1 Standard Stock/fortification Solution**

- 9.1.1 Prepare a stock solution of ~100 µg/mL of PFOA by weighing 10 mg of analytical standard (corrected for purity) and dilute to 100 mL with methanol in a 125-mL LDPE bottle.
- 9.1.2 A 10 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 100 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.3 A 1.0 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 10 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.4 A 0.1 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 1.0 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.5 A 0.01 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 0.1 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.6 The stock and fortification solutions are to be stored in a refrigerator at approximately 4°C and are stable for a maximum period of 6 months from the date of preparation.

**9.2 Standard Calibration Solutions**

- 9.2.1 LC/MS/MS calibration standards are prepared in HPLC water. The calibration standards are processed through the extraction procedure, identical to samples.
- 9.2.2 The following is a typical example: additional concentrations may be prepared as needed.

| Concentration of Fortification Solution (ppb) | Fortification Volume (µL) | Volume of Fortified Control Sample (mL) | Final Concentration of Calibration Standard (ppt)* | Calibration Standard ID (example) |
|-----------------------------------------------|---------------------------|-----------------------------------------|----------------------------------------------------|-----------------------------------|
| 0                                             | 0                         | 40                                      | 0                                                  | XCmmdyyy-0                        |
| 10                                            | 100                       | 40                                      | 25                                                 | XCmmdyyy-1                        |
| 10                                            | 200                       | 40                                      | 50                                                 | XCmmdyyy-2                        |
| 10                                            | 400                       | 40                                      | 100                                                | XCmmdyyy-3                        |
| 100                                           | 100                       | 40                                      | 250                                                | XCmmdyyy-4                        |
| 100                                           | 200                       | 40                                      | 500                                                | XCmmdyyy-5                        |
| 100                                           | 400                       | 40                                      | 1000                                               | XCmmdyyy-6                        |

\* The extracted concentration of the calibration standard is equal to 8x its initial concentration, due to the concentration of the standard during the extraction (SPE).  
XC = extracted calibration standard.

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Method Number V0001781

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS**

- 9.2.3 A zero standard solution (reagent blank) must be prepared with each set of standards extracted.
- 9.2.4 Store all extracted calibration standards in 15-mL polypropylene tubes at 2°C to 6°C, up to two weeks.
- 9.2.5 Alternate volumes and concentrations of standards may be prepared as needed.
- 10.0 Batch Set Up
- 10.1 Each batch of samples extracted (typically 20 or less) must include at least one reagent control (method blank using 5 mL of methanol) and two reagent controls fortified at known concentrations (lab control spike) to verify procedural recovery for the batch.
- 10.2 Requirements for field and laboratory duplicates and spikes will be specified in the quality assurance plan for this project.
- 11.0 Sample Extraction
- 11.1 Weigh 5 g of sample into 50 mL polypropylene centrifuge tubes (fortify as needed, replace lid and mix well).
- 11.2 Add 5 mL of methanol and shake on a wrist action shaker for ~15 minutes.
- 11.3 Transfer the tubes to an ultrasonic bath and sonicate for ~15 minutes.
- 11.4 Bring the volume up to 40 mL with water in the 50 mL polypropylene centrifuge tube.
- 11.5 Centrifuge for ~10 minutes at ~3000 rpm.
- 11.6 Condition the C<sub>18</sub> SPE cartridges (1 g, 6 mL) by passing 10 mL methanol followed by 5 mL of HPLC water (~2 drop/sec). Do not let column run dry.
- 11.7 Load (decant) the sample on the conditioned C<sub>18</sub> SPE cartridge. Discard eluate.
- 11.8 Elute with ~5 mL 100% methanol. Collect 5 mL of eluate into graduated 15 mL polypropylene centrifuge tubes (final volume = 5 mL).
- 11.9 Analyze samples using electrospray LC/MS/MS.
- 12.0 Chromatography
- 12.1 Inject the same amount of each standard, sample and fortified sample into the LC/MS/MS system. A calibration standard must precede and follow all analyzed samples.
- 12.2 Standards of PFOA corresponding to at least five or more concentration levels must be included in an analytical set.
- 12.3 An entire set of extracted calibration standards must be included at the beginning and at the end of a sample set. Extracted standards must be interspersed between every 5-10 samples. As an alternative, an entire set of

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Exygen Research

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**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by LC/MS/MS**

extracted calibration standards may be injected at the beginning of a set followed by extracted calibration standards interspersed every 5-10 samples (to account for a second set of extracted standards). In either case, extracted calibration standards must be the first and last injection in a sample set.

- 12.4 Use linear standard curves for quantitation. Linear standard curves are generated for the analyte by linear regression using 1/x weighting of peak area versus calibration standard concentration using MassLynx 3.3 (or equivalent) software system.
- 12.5 Sample response should not exceed standard responses. Any samples that exceed standard responses should be further diluted and reanalyzed.

**13.0 Acceptance Criteria**

- 13.1 Chromatogram must show a peak of a daughter ion at 369 amu from a parent of 413 amu. The 413 amu parent corresponds to the PFOA anion, while the daughter ion (369 amu) represents the loss of carbon dioxide.
- 13.2 Method blanks must not contain PFOA at levels greater than the LOQ. If a blank contains PFOA at levels greater than 50 ng/L, then a new blank sample must be obtained and the entire set must be re-extracted.
- 13.3 Recoveries of control spikes and matrix spikes must be between 70-130% of their known values. If a control spike falls outside the acceptable limits, the entire set of samples should be re-extracted. Any matrix spike outside 70-130% should be evaluated by the analyst to determine if re-extraction is warranted.
- 13.4 Any calibration standard found to be a statistical outlier by using the Huge Error Test, may be excluded from the calculation of the calibration curve. However, the total number of extracted calibration standards that could be excluded must not exceed 20% of the total number of extracted standards injected.
- 13.5 The correlation coefficient (R) for calibration curves generated must be  $\geq 0.992$  ( $R^2 \geq 0.985$ ). If calibration results fall outside these limits, then appropriate steps must be taken to adjust instrument operation, and the standards or the relevant set of samples should be reanalyzed.
- 13.6 Retention times between standards and samples must not drift more than  $\pm 4\%$  within an analytical run. If retention time drift exceeds this limit within an analytical run then the set must be reanalyzed.

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Exygen Research

Method Number V0001781

**ANALYTICAL METHOD**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Soil by  
LC/MS/MS**14.0 Calculations**

- 14.1 Use the following equation to calculate the amount of PFOA found (in ng/L, based on peak area) using the standard curve (linear regression parameters) generated by the Mass Lynx software program:

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

DF = factor by which the final volume was diluted, if necessary.

- 14.2 For samples fortified with known amounts of PFOA prior to extraction, use the following equation to calculate the percent recovery.

Recovery (%) =

$$\frac{[\text{total analyte found (ng/L)} - \text{analyte found in control (ng/L)}]}{\text{analyte added (ng/L)}} \times 100$$

- 14.3 Use the following equation to convert the amount of PFOA found in ng/L to ng/g (ppb).

$$\text{PFOA found (ppb)} = \frac{[\text{PFOA found (ng/L)} \times \text{volume extracted (0.04L)}]}{\text{sample weight (5 g)}}$$

- 14.4 Use the following equation to calculate the amount of PFOA found in ppb based on dry weight.

$$\text{PFOA found (ppb) dry weight} = \text{PFOA found (ppb)} \times [100\% / \text{total solids}(\%)]$$

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**ANALYTICAL METHOD**  
Method Number: V0001782

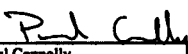
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Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in  
Sediment by LC/MS/MS

Analytical Testing Facility:

Exygen Research  
3058 Research Drive  
State College, PA 16801

Approved By:

  
Paul Connolly  
Technical Leader, LC-MS, Exygen Research

10/26/04  
Date

  
John Flaherty  
Vice President, Operations, Exygen Research

10/26/04  
Date

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Total Pages: 7

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001782

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS

**1.0 Scope**

This method is to be employed for the isolation and quantitation of perfluorooctanoic acid by High Performance Liquid Chromatography coupled to a tandem Mass Spectrometric Detector (LC/MS/MS) in sediment.

**2.0 Safety**

- 2.1 Always observe safe laboratory practices.
- 2.2 Consult the appropriate MSDS before handling any chemical for proper safety precautions.

**3.0 Sample Requirement**

- 3.1 At least 30 g of test sample for extraction.
- 3.2 No sample processing is needed for sediment samples.
- 3.3 Samples stored refrigerated should be allowed to equilibrate to room temperature.
- 3.4 All samples must be thoroughly mixed before being sampled for extraction.
- 3.5 Sample collection procedures will be specified in the sampling plan for this project.

**4.0 Reagents and Standards**

- 4.1 Water – HPLC grade
- 4.2 Methanol – HPLC grade
- 4.3 Acetic Acid – Reagent grade
- 4.4 Ammonium Acetate – A.C.S. Reagent Grade
- 4.5 Perfluorooctanoic Acid – Sigma-Aldrich

**5.0 Instrument and Equipment**

- 5.1 A high performance liquid chromatograph capable of pumping up to 2 solvents equipped with a variable volume injector capable of injecting 5-200  $\mu$ L connected to a tandem Mass Spectrometer (LC/MS/MS).
- 5.2 A device to collect raw data for peak integration and quantitation.
- 5.3 Analytical balance capable of reading to 0.00001 g.
- 5.4 50 mL disposable polypropylene centrifuge tubes.
- 5.5 15 mL disposable polypropylene centrifuge tubes.
- 5.6 Disposable micropipets (30-100 $\mu$ L, 100-200 $\mu$ L).
- 5.7 125-mL LDPE narrow-mouth bottles.
- 5.8 2 mL clear HPLC vial kit.
- 5.9 Disposable pipettes.
- 5.10 Autopipettes (100-1000  $\mu$ L and 10-100  $\mu$ L), with disposable tips.
- 5.11 Waters Sep Pak Vac 6 cc (1g)  $\text{C}_{18}$  SPE cartridges.
- 5.12 SPE vacuum manifold.

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001782

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS

- 5.13 Vortexer.
- 5.14 Wrist-action shaker.
- 5.15 Centrifuge capable of spinning 50 mL polypropylene tubes at 3000 rpm.

**6.0 Chromatographic System**

- 6.1 Analytical Column: Fluophase RP (Keystone Scientific), 2.1 mm x 50 mm, 5µ (P/N: 82505-052130)
- 6.2 Temperature: 30°C
- 6.3 Mobile Phase (A): 2 mM Ammonium Acetate in Water
- 6.4 Mobile Phase (B): Methanol
- 6.5 Gradient Program:

| Time (min) | % A | % B | Flow Rate (mL/min) |
|------------|-----|-----|--------------------|
| 0.0        | 65  | 35  | 0.3                |
| 1.0        | 65  | 35  | 0.3                |
| 8.0        | 25  | 75  | 0.3                |
| 20.0       | 25  | 75  | 0.3                |
| 22.5       | 65  | 35  | 0.3                |

- 6.6 Injection Volume: 15 µL (can be increased to as much as 50 µL).
- 6.7 Quantitation: Peak Area - external standard calibration curve.
- 6.8 Run Time: ~ 23 minutes.

The above conditions are intended as a guide and may be changed in order to optimize the HPLC system.

**7.0 MS/MS System**

- 7.1 Mode: Electrospray Negative MRM mode, monitoring 413 → 369 m/z for PFOA.

The above conditions are intended as a guide and may be changed in order to optimize the MS/MS system.

**8.0 Preparation of Solutions****8.1 Mobile Phase**

- 8.1.1 2 mM ammonium acetate in water is prepared by adding 0.154 g of ammonium acetate to 1000 mL of water.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS

**8.2 Extraction Solutions**

- 8.2.1 1% acetic acid in water is prepared by adding 10 mL of acetic acid to 1000 mL of water.

Alternate volumes may be prepared.

**9.0 Standard Preparation****9.1 Standard Stock/Fortification Solution**

- 9.1.1 Prepare a stock solution of ~100 µg/mL of PFOA by weighing 10 mg of analytical standard (corrected for purity) and dilute to 100 mL with methanol in a 125-mL LDPE bottle.
- 9.1.2 A 10 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 100 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.3 A 1.0 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 10 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.4 A 0.1 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 1.0 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.5 A 0.01 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 0.1 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.6 The stock and fortification solutions are to be stored in a refrigerator at approximately 4°C and are stable for a maximum period of 6 months from the date of preparation.

**9.2 Standard Calibration Solutions**

- 9.2.1 LC/MS/MS calibration standards are prepared in methanol via dilution of the 0.1 µg/mL fortification solution.
- 9.2.2 The following is a typical example: additional concentrations may be prepared as needed.

| Concentration of Fortification Solution (ng/mL) | Volume (mL) | Diluted to (mL) | Final Concentration (ng/mL) |
|-------------------------------------------------|-------------|-----------------|-----------------------------|
| 100                                             | 10          | 100             | 10.0                        |
| 100                                             | 5           | 100             | 5.0                         |
| 100                                             | 2           | 100             | 2.0                         |
| 10                                              | 10          | 100             | 1.0                         |
| 5                                               | 10          | 100             | 0.5                         |
| 2                                               | 10          | 100             | 0.2                         |

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**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS**

- 9.2.3 Store all calibration standards in 125-mL LDPE narrow-mouth bottles at 2°C to 6°C, up to six months.
- 9.2.4 Alternate volumes and concentrations of standards may be prepared as needed.
- 10.0 Batch Set Up
- 10.1 Each batch of samples extracted (typically 20 or less) must include at least one untreated control and two untreated controls fortified at known concentrations (lab control spike) to verify procedural recovery for the batch.
- 10.2 Requirements for field and laboratory duplicates and spikes will be specified in the quality assurance plan for this project.
- 11.0 Sample Extraction
- 11.1 Weigh 5 g of sample into 50 mL polypropylene centrifuge tubes (fortify as needed, replace lid and mix well).
- 11.2 Add 35 mL of 1% acetic acid, cap, vortex and shake on a wrist action shaker for ~60 minutes.
- 11.3 Centrifuge the tubes at ~3000 rpm for ~20 minutes.
- 11.4 Condition the C<sub>18</sub> SPE cartridges (1 g, 6 mL) by passing 10 mL methanol followed by 20 mL of HPLC water (~2 drop/sec). Do not let column run dry.
- 11.5 Load (decant) the sample on the conditioned C<sub>18</sub> SPE cartridge. Discard eluate.
- 11.6 Add 20 mL of methanol to the sediment left in the bottom of the 50 mL centrifuge tube. Cap, vortex and shake on a wrist action shaker for ~30 minutes.
- 11.7 Centrifuge the tubes at ~3000 rpm for ~20 minutes.
- 11.8 Decant the methanol onto the same SPE cartridge. Collect the eluate.
- 11.9 Wash the column with 4 mL of methanol. Collect the eluate and add it to the eluate collected in step 11.8.
- 11.10 Condition a second C<sub>18</sub> SPE cartridge (1 g, 6 mL) by passing 10 mL methanol followed by 20 mL of HPLC water (~2 drop/sec). Do not let column run dry.
- 11.11 Add the methanol to ~200 mL of water and load on the second conditioned SPE cartridge.
- 11.12 Elute with ~5 mL 100% methanol. Collect 5 mL of eluate into graduated 15 mL polypropylene centrifuge tubes (final volume = 5 mL).
- 11.13 Analyze samples using electrospray LC/MS/MS.

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Method Number V0001782

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS

**12.0 Chromatography**

- 12.1 Inject the same amount of each standard, sample and fortified sample into the LC/MS/MS system. A calibration standard must precede and follow all analyzed samples.
- 12.2 Standards of PFOA corresponding to at least five or more concentration levels must be included in an analytical set.
- 12.3 An entire set of extracted calibration standards must be included at the beginning and at the end of a sample set. Standards must be interspersed between every 5-10 samples. As an alternative, an entire set of calibration standards may be injected at the beginning of a set followed by calibration standards interspersed every 5-10 samples (to account for a second set of standards). In either case, calibration standards must be the first and last injection in a sample set.
- 12.4 Use linear standard curves for quantitation. Linear standard curves are generated for the analyte by linear regression using 1/x weighting of peak area versus calibration standard concentration using MassLynx 3.3 (or equivalent) software system.
- 12.5 Sample response should not exceed standard responses. Any samples that exceed standard responses should be further diluted and reanalyzed.

**13.0 Acceptance Criteria**

- 13.1 Chromatogram must show a peak of a daughter ion at 369 amu from a parent of 413 amu. The 413 amu parent corresponds to the PFOA anion, while the daughter ion (369 amu) represents the loss of carbon dioxide.
- 13.2 Method blanks must not contain PFOA at levels greater than the LOQ. If a blank contains PFOA at levels greater than 0.2 ng/mL, then a new blank sample must be obtained and the entire set must be re-extracted.
- 13.3 Recoveries of control spikes and matrix spikes must be between 70-130% of their known values. If a control spike falls outside the acceptable limits, the entire set of samples should be re-extracted. Any matrix spike outside 70-130% should be evaluated by the analyst to determine if re-extraction is warranted.
- 13.4 Any calibration standard found to be a statistical outlier by using the Huge Error Test, may be excluded from the calculation of the calibration curve. However, the total number of extracted calibration standards that could be excluded must not exceed 20% of the total number of extracted standards injected.
- 13.5 The correlation coefficient (R) for calibration curves generated must be  $\geq 0.992$  ( $R^2 \geq 0.985$ ). If calibration results fall outside these limits, then appropriate steps must be taken to adjust instrument operation, and the standards or the relevant set of samples should be reanalyzed.

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Exygen Research

Method Number V0001782

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Sediment by LC/MS/MS

- 13.6 Retention times between standards and samples must not drift more than  $\pm 4\%$  within an analytical run. If retention time drift exceeds this limit within an analytical run then the set must be reanalyzed.

**14.0 Calculations**

- 14.1 Use the following equation to calculate the amount of PFOA found (in ng/mL, based on peak area) using the standard curve (linear regression parameters) generated by the Mass Lynx software program:

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

DF = factor by which the final volume was diluted, if necessary.

- 14.2 For samples fortified with known amounts of PFOA prior to extraction, use the following equation to calculate the percent recovery.

Recovery (%) =

$$\frac{[\text{total analyte found (ng/mL)} - \text{analyte found in control (ng/mL)}]}{\text{analyte added (ng/mL)}} \times 100$$

- 14.3 Use the following equation to convert the amount of PFOA found in ng/mL to ng/g (ppb).

$$\text{PFOA found (ppb)} = \frac{[\text{PFOA found (ng/mL)} \times \text{final volume (5 mL)}]}{\text{sample weight (5 g)}}$$

- 14.4 Use the following equation (if necessary) to calculate the amount of PFOA found in ppb based on dry weight.

$$\text{PFOA found (ppb) dry weight} = \text{PFOA found (ppb)} \times [100\% / \text{total solids}(\%)]$$

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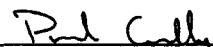
**ANALYTICAL METHOD**  
Method Number: V0001783

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**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS**

Analytical Testing Facility: Exygen Research  
3058 Research Drive  
State College, PA 16801

Approved By:

  
Paul Connolly  
Technical Leader, LC-MS, Exygen Research

10/25/04  
Date

  
John Flaherty  
Vice President, Operations, Exygen Research

10/26/04  
Date

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001783

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS****1.0 Scope**

This method is to be employed for the isolation and quantitation of perfluorooctanoic acid by High Performance Liquid Chromatography coupled to a tandem Mass Spectrometric Detector (LC/MS/MS) in fish and clams.

**2.0 Safety**

- 2.1 Always observe safe laboratory practices.
- 2.2 Consult the appropriate MSDS before handling any chemical for proper safety precautions.

**3.0 Sample Requirement**

- 3.1 At least 20 g of test sample for extraction.
- 3.2 Samples should be processed before extraction. Place the frozen sample in a food processor and homogenize with dry ice. Place the samples in containers and leave open in frozen storage overnight to allow for carbon dioxide sublimation. Seal and place the samples in frozen storage until time of analysis.
- 3.3 Sample collection procedures will be specified in the sampling plan for this project.

**4.0 Reagents and Standards**

- 4.1 Water - HPLC grade
- 4.2 Acetonitrile - HPLC grade
- 4.3 Carbon (120-400 mesh) - Reagent grade
- 4.4 Methanol - HPLC grade
- 4.5 Silica gel (60-200 mesh) - Reagent grade
- 4.6 Florisil (60-100 mesh) - Reagent grade
- 4.7 Superclean LC-NH<sub>2</sub> - Reagent grade
- 4.8 1-Octanol - HPLC grade
- 4.9 L-Ascorbic acid - Reagent grade
- 4.10 Dimethyldichlorosilane - Reagent grade
- 4.11 Toluene - Reagent grade
- 4.12 Ammonium Acetate - A.C.S. Reagent Grade
- 4.13 Perfluorooctanoic Acid - Sigma-Aldrich

**5.0 Instrument and Equipment**

- 5.1 A high performance liquid chromatograph capable of pumping up to 2 solvents equipped with a variable volume injector capable of injecting 5-200 µL connected to a tandem Mass Spectrometer (LC/MS/MS).
- 5.2 A device to collect raw data for peak integration and quantitation.
- 5.3 Analytical balance capable of reading to 0.00001 g.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS

- 5.4 Rotary evaporator.
- 5.5 Tissue grinder.
- 5.6 125 mL pear-shaped flasks.
- 5.7 50 mL disposable polypropylene centrifuge tubes.
- 5.8 15 mL disposable polypropylene centrifuge tubes.
- 5.9 Disposable micropipets (50-100 $\mu$ L, 100-200 $\mu$ L).
- 5.10 125-mL LDPE narrow-mouth bottles.
- 5.11 2 mL clear HPLC vial kit.
- 5.12 Disposable pipettes.
- 5.13 Autopipettes (100-1000  $\mu$ L and 10-100  $\mu$ L), with disposable tips.
- 5.14 SPE tubes (20mL) (Supelco cat. no. N057177).
- 5.15 Wrist action shaker.
- 5.16 Centrifuge capable of spinning 50 mL polypropylene tubes at 2000 rpm.

**6.0 Chromatographic System**

- 6.1 Analytical Column: Fluorophase RP (Keystone Scientific), 2.1 mm x 50 mm, 5 $\mu$  (P/N: 82505-052130)
- 6.2 Temperature: 30°C
- 6.3 Mobile Phase (A) : 2 mM Ammonium Acetate in Water
- 6.4 Mobile Phase (B) : Methanol
- 6.5 Gradient Program:

| Time (min) | % A | % B | Flow Rate (mL/min) |
|------------|-----|-----|--------------------|
| 0.0        | 65  | 35  | 0.3                |
| 1.0        | 65  | 35  | 0.3                |
| 8.0        | 25  | 75  | 0.3                |
| 20.0       | 25  | 75  | 0.3                |
| 22.5       | 65  | 35  | 0.3                |

- 6.6 Injection Volume: 15  $\mu$ L (can be increased to as much as 50  $\mu$ L).
- 6.7 Quantitation: Peak Area - external standard calibration curve.
- 6.8 Run Time: ~ 23 minutes.

The above conditions are intended as a guide and may be changed in order to optimize the HPLC system.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS

**7.0 MS/MS System**

- 7.1 Mode: Electrospray Negative MRM mode, monitoring 413 → 369 m/z for PFOA.

The above conditions are intended as a guide and may be changed in order to optimize the MS/MS system.

**8.0 Preparation of Solutions****8.1 Mobile Phase**

- 8.1.1 2 mM ammonium acetate in water is prepared by adding 0.154 g of ammonium acetate to 1000 mL of water.

**8.2 Extraction Solutions**

- 8.2.1 2% ascorbic acid in methanol is prepared by dissolving 2 g of ascorbic acid in 100 mL of methanol.
- 8.2.2 30% Dimethyldichlorosilane in toluene is prepared by bringing 3 mL of dimethyldichlorosilane to a final volume of 10 mL with toluene.

Alternate volumes may be prepared.

**9.0 Standard Preparation****9.1 Standard Stock/Fortification Solution**

- 9.1.1 Prepare a stock solution of ~100 µg/mL of PFOA by weighing 10 mg of analytical standard (corrected for purity) and dilute to 100 mL with methanol in a 125-mL LDPE bottle.
- 9.1.2 A 1.0 µg/mL fortification solution of PFOA is prepared by bringing 1 mL of the 100 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.3 A 0.1 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 1.0 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.4 A 0.01 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 0.1 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.5 The stock and fortification solutions are to be stored in a refrigerator at approximately 4°C and are stable for a maximum period of 6 months from the date of preparation.

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**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS****9.2 Standard Calibration Solutions**

9.2.1 LC/MS/MS calibration standards are prepared in methanol via dilution of the 1.0 µg/mL fortification solution.

9.2.2 The following is a typical example; additional concentrations may be prepared as needed.

| Concentration of Fortification Solution (µg/mL) | Volume (mL) | Diluted to (mL) | Final Concentration (µg/mL) |
|-------------------------------------------------|-------------|-----------------|-----------------------------|
| 1.0                                             | 5.0         | 100             | 0.05                        |
| 1.0                                             | 2.5         | 100             | 0.025                       |
| 1.0                                             | 1.0         | 100             | 0.01                        |
| 0.05                                            | 10          | 100             | 0.005                       |
| 0.025                                           | 10          | 100             | 0.0025                      |
| 0.1                                             | 10          | 100             | 0.001                       |
| 0.005                                           | 10          | 100             | 0.0005                      |

9.2.3 Store all calibration standards in 125-mL LDPE narrow-mouth bottles at 2°C to 6°C, up to six months.

9.2.4 Alternate volumes and concentrations of standards may be prepared as needed.

**10.0 Batch Set Up**

10.1 Each batch of samples extracted (typically 20 or less) must include at least one untreated control and two untreated controls fortified at known concentrations (lab control spike) to verify procedural recovery for the batch.

10.2 Requirements for field and laboratory duplicates and spikes will be specified in the quality assurance plan for this project.

**11.0 Sample Extraction**

11.1 Weigh 5 g of frozen sample into 50 mL polypropylene centrifuge tubes (fortify as needed, replace lid and mix well).

11.2 Add 30 mL of acetonitrile and shake on a wrist action shaker for ~15 minutes.

11.3 Place the tubes in a freezer for ~1 hour.

11.4 Pack and condition the SPE tubes and silanize the pear-shaped flasks.

11.5 Pack the 20 mL SPE tubes in sequence with 2 g florisil, 2 g silica gel, 2 g carbon, and 1 g LC-NH<sub>2</sub>. Condition the columns with 20 mL of methanol, then 20 mL of acetonitrile. Discard all washes. Do not allow the column to dry.

11.6 Silanize the 125 mL pear-shaped flasks by rinsing with the 30% dimethyldichlorosilane in toluene solution. Rinse the flask with toluene once, followed by methanol (three times). Dry the flasks completely before use, either by air-drying or with a stream of nitrogen.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS

- 11.7 Centrifuge the 50 mL polypropylene tubes containing sample at ~2000 rpm for ~10 minutes.
- 11.8 Decant the extract on to a conditioned SPE column fitted inside the mouth of the pear-shaped flask. Collect the eluate in the 125 mL silanized pear-shape flask.
- 11.9 Add 10 mL of acetonitrile to the sample in the 50 mL centrifuge tube. Homogenize the frozen fat phase using a tissumizer for ~30 seconds and rinse the tissumizer with ~10 mL of acetonitrile into the tube.
- 11.10 Shake the sample again for ~10 minutes on a wrist-action shaker.
- 11.11 Place the tubes in a freezer for ~1 hour more.
- 11.12 Centrifuge the 50 mL polypropylene tubes containing sample at ~2000 rpm for ~10 minutes.
- 11.13 Decant the extract onto the same SPE column. Collect the eluate into the same pear-shaped flask and combine with the eluent from the initial extraction.
- 11.14 Pass 20 mL of acetonitrile through the SPE column and combine the eluate in the same pear-shaped flask.
- 11.15 Add 3-4 drops of 1-octanol to the extract in the pear-shaped flask and evaporate at reduced pressure using a rotary evaporator (at < 40°C).
- 11.16 Make the final volume, by adding 2 mL of 2% ascorbic acid in methanol to the pear-shaped flask and swirl to mix/dissolve.
- 11.17 Transfer the extracts to HPLC vials using disposable pipets.
- 11.18 Analyze samples using electrospray LC/MS/MS.

**12.0 Chromatography**

- 12.1 Inject the same amount of each standard, sample and fortified sample into the LC/MS/MS system. A calibration standard must precede and follow all analyzed samples.
- 12.2 Standards of PFOA corresponding to at least five or more concentration levels must be included in an analytical set.
- 12.3 An entire set of calibration standards must be included at the beginning and at the end of a sample set. Standards must be interspersed between every 5-10 samples. As an alternative, an entire set of calibration standards may be injected at the beginning of a set followed by calibration standards interspersed every 5-10 samples (to account for a second set of standards). In either case, calibration standards must be the first and last injection in a sample set.
- 12.4 Use linear standard curves for quantitation. Linear standard curves are generated for the analyte by linear regression using 1/x weighting of peak area versus calibration standard concentration using MassLynx 3.3 (or equivalent) software system.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS

- 12.5 Sample response should not exceed standard responses. Any samples that exceed standard responses should be further diluted and reanalyzed.

**13.0 Acceptance Criteria**

- 13.1 Chromatogram must show a peak of a daughter ion at 369 amu from a parent of 413 amu. The 413 amu parent corresponds to the PFOA anion, while the daughter ion (369 amu) represents the loss of carbon dioxide.
- 13.2 Method blanks must not contain PFOA at levels greater than the LOQ. If a blank contains PFOA at levels greater than 0.5 ppb, then a new blank sample must be obtained and the entire set must be re-extracted.
- 13.3 Recoveries of control spikes and matrix spikes must be between 70-130% of their known values. If a control spike falls outside the acceptable limits, the entire set of samples should be re-extracted.
- 13.4 Any calibration standard found to be a statistical outlier by using the Huge Error Test, may be excluded from the calculation of the calibration curve. However, the total number of calibration standards that could be excluded must not exceed 20% of the total number of standards injected.
- 13.5 The correlation coefficient (R) for calibration curves generated must be  $\geq 0.992$  ( $R^2 \geq 0.983$ ). If calibration results fall outside these limits, then appropriate steps must be taken to adjust instrument operation, and the standards or the relevant set of samples should be reanalyzed.
- 13.6 Retention times between standards and samples must not drift more than  $\pm 4\%$  within an analytical run. If retention time drift exceeds this limit within an analytical run then the set must be reanalyzed.

**14.0 Calculations**

- 14.1 Use the following equation to calculate the amount of PFOA found (in ng/mL, based on peak area) using the standard curve (linear regression parameters) generated by the Mass Lynx software program:

$$\text{PFOA found (ng/mL)} = \frac{(\text{Peak area} - \text{intercept})}{\text{slope}}$$

- 14.2 Use the following equation to convert the amount of PFOA found in ng/mL to ng/g (ppb).

$$\text{PFOA found (ppb)} = \frac{(\text{PFOA found (ng/mL)} \times \text{final volume (mL)}) \times \text{DF}}{\text{sample weight (g)}}$$

DF = factor by which the final volume was diluted, if necessary.

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Method Number V0001783

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Fish and Clams by LC/MS/MS

- 14.3 For samples fortified with known amounts of PFOA prior to extraction, use the following equation to calculate the percent recovery.

Recovery (%) =

$$\frac{[\text{total analyte found (ng/g)} - \text{analyte found in control (ng/g)}]}{\text{analyte added (ng/g)}} \times 100$$

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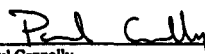
**ANALYTICAL METHOD**  
Method Number: V0001784

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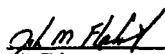
**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in  
Vegetation by LC/MS/MS**

Analytical Testing Facility: Exygen Research  
3058 Research Drive  
State College, PA 16801

Approved By:

  
Paul Connolly  
Technical Leader, LC-MS, Exygen Research

10/26/04  
Date

  
John Flaherty  
Vice President, Operations, Exygen Research

11/16/04  
Date

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Total Pages: 7

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Exygen Research

Method Number V0001784

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS

**1.0 Scope**

This method is to be employed for the isolation and quantitation of perfluorooctanoic acid by High Performance Liquid Chromatography coupled to a tandem Mass Spectrometric Detector (LC/MS/MS) in vegetation.

**2.0 Safety**

- 2.1 Always observe safe laboratory practices.
- 2.2 Consult the appropriate MSDS before handling any chemical for proper safety precautions.

**3.0 Sample Requirement**

- 3.1 At least 20 g of test sample for extraction.
- 3.2 Samples should be processed before extraction. Place the frozen sample in a food processor and homogenize with dry ice. Place the samples in containers and leave open in frozen storage overnight to allow for carbon dioxide sublimation. Seal and place the samples in frozen storage until time of analysis.
- 3.3 Sample collection procedures will be specified in the sampling plan for this project.

**4.0 Reagents and Standards**

- 4.1 Water - HPLC grade
- 4.2 Acetonitrile - HPLC grade
- 4.3 Carbon (120-400 mesh) - Reagent grade
- 4.4 Methanol - HPLC grade
- 4.5 Silica gel (60-200 mesh) - Reagent grade
- 4.6 Florisil (60-100 mesh) - Reagent grade
- 4.7 Superclean LC-NH<sub>2</sub> - Reagent grade
- 4.8 1-Octanol - HPLC grade
- 4.9 L-Ascorbic acid - Reagent grade
- 4.10 Dimethyldichlorosilane - Reagent grade
- 4.11 Toluene - Reagent grade
- 4.12 Ammonium Acetate - A.C.S. Reagent Grade
- 4.13 Perfluorooctanoic Acid - Sigma-Aldrich

**5.0 Instrument and Equipment**

- 5.1 A high performance liquid chromatograph capable of pumping up to 2 solvents equipped with a variable volume injector capable of injecting 5-200 µL connected to a tandem Mass Spectrometer (LC/MS/MS).
- 5.2 A device to collect raw data for peak integration and quantitation.
- 5.3 Analytical balance capable of reading to 0.00001 g.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS

- 5.4 Rotary evaporator.
- 5.5 125 mL pear-shaped flasks.
- 5.6 50 mL disposable polypropylene centrifuge tubes.
- 5.7 15 mL disposable polypropylene centrifuge tubes.
- 5.8 Disposable micropipets (50-100 $\mu$ L, 100-200 $\mu$ L).
- 5.9 125-mL LDPE narrow-mouth bottles.
- 5.10 2 mL clear HPLC vial kit.
- 5.11 Disposable pipettes.
- 5.12 Autopipettes (100-1000  $\mu$ L and 10-100  $\mu$ L), with disposable tips.
- 5.13 SPE tubes (20mL) (Supelco cat. no. N057177).
- 5.14 Wrist action shaker.
- 5.15 Centrifuge capable of spinning 50 mL polypropylene tubes at 2000 rpm.

**6.0 Chromatographic System**

- 6.1 Analytical Column: Fluophase RP (Keystone Scientific), 2.1 mm x 50 mm, 5 $\mu$  (P/N: 82505-052130)
- 6.2 Temperature: 30°C
- 6.3 Mobile Phase (A): 2 mM Ammonium Acetate in Water
- 6.4 Mobile Phase (B): Methanol
- 6.5 Gradient Program:

| Time (min) | % A | % B | Flow Rate (mL/min) |
|------------|-----|-----|--------------------|
| 0.0        | 65  | 35  | 0.3                |
| 1.0        | 65  | 35  | 0.3                |
| 8.0        | 25  | 75  | 0.3                |
| 20.0       | 25  | 75  | 0.3                |
| 22.5       | 65  | 35  | 0.3                |

- 6.6 Injection Volume: 15  $\mu$ L (can be increased to as much as 50  $\mu$ L).
- 6.7 Quantitation: Peak Area - external standard calibration curve.
- 6.8 Run Time: ~ 23 minutes.

The above conditions are intended as a guide and may be changed in order to optimize the HPLC system.

**7.0 MS/MS System**

- 7.1 Mode: Electrospray Negative MRM mode, monitoring 413  $\rightarrow$  369 m/z for PFOA.

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**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS**

The above conditions are intended as a guide and may be changed in order to optimize the MS/MS system.

**8.0 Preparation of Solutions****8.1 Mobile Phase**

8.1.1 2 mM ammonium acetate in water is prepared by adding 0.154 g of ammonium acetate to 1000 mL of water.

**8.2 Extraction Solutions**

8.2.1 2% ascorbic acid in methanol is prepared by dissolving 2 g of ascorbic acid in 100 mL of methanol.

8.2.2 30% Dimethyldichlorosilane in toluene is prepared by bringing 3 mL of dimethyldichlorosilane to a final volume of 10 mL with toluene.

Alternate volumes may be prepared.

**9.0 Standard Preparation****9.1 Standard Stock/fortification Solution**

9.1.1 Prepare a stock solution of ~100 µg/mL of PFOA by weighing 10 mg of analytical standard (corrected for purity) and dilute to 100 mL with methanol in a 125-mL LDPE bottle.

9.1.2 A 1.0 µg/mL fortification solution of PFOA is prepared by bringing 1 mL of the 100 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.

9.1.3 A 0.1 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 1.0 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.

9.1.4 A 0.01 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 0.1 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.

9.1.5 The stock and fortification solutions are to be stored in a refrigerator at approximately 4°C and are stable for a maximum period of 6 months from the date of preparation.

**9.2 Standard Calibration Solutions**

9.2.1 LC/MS/MS calibration standards are prepared in methanol via dilution of the 1.0 µg/mL fortification solution.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS

9.2.2 The following is a typical example: additional concentrations may be prepared as needed.

| Concentration of Fortification Solution (µg/mL) | Volume (mL) | Diluted to (mL) | Final Concentration (µg/mL) |
|-------------------------------------------------|-------------|-----------------|-----------------------------|
| 1.0                                             | 5.0         | 100             | 0.05                        |
| 1.0                                             | 2.5         | 100             | 0.025                       |
| 1.0                                             | 1.0         | 100             | 0.01                        |
| 0.05                                            | 10          | 100             | 0.005                       |
| 0.025                                           | 10          | 100             | 0.0025                      |
| 0.1                                             | 10          | 100             | 0.001                       |
| 0.005                                           | 10          | 100             | 0.0005                      |

9.2.3 Store all calibration standards in 125-mL LDPE narrow-mouth bottles at 2°C to 6°C, up to six months.

9.2.4 Alternate volumes and concentrations of standards may be prepared as needed.

**10.0 Batch Set Up**

- 10.1 Each batch of samples extracted (typically 20 or less) must include at least one untreated control and two untreated controls fortified at known concentrations (lab control spikes) to verify procedural recovery for the batch.
- 10.2 Requirements for field and laboratory duplicates and spikes will be specified in the quality assurance plan for this project.

**11.0 Sample Extraction**

- 11.1 Weigh 5 g of frozen sample into 50 mL polypropylene centrifuge tubes (fortify as needed, replace lid and mix well).
- 11.2 Add 30 mL of acetonitrile and shake on a wrist action shaker for ~15 minutes.
- 11.3 Centrifuge the 50 mL polypropylene tubes containing sample at ~2000 rpm for ~10 minutes.
- 11.4 Pack and condition the SPE tubes and silanize the pear-shaped flasks.
- 11.5 Pack the 20 mL SPE tubes in sequence with 2 g florisil, 2 g silica gel, 2 g carbon, and 1 g LC-NH<sub>2</sub>. Condition the columns with 20 mL of methanol, then 20 mL of acetonitrile. Discard all washes. Do not allow the column to dry.
- 11.6 Silanize the 125 mL pear-shaped flasks by rinsing with the 30% dimethyldichlorosilane in toluene solution. Rinse the flask with toluene once, followed by methanol (three times). Dry the flasks completely before use, either by air-drying or with a stream of nitrogen.
- 11.7 Decant the extract on to a conditioned SPE column fitted inside the mouth of the pear-shaped flask. Collect the eluate in the 125 mL silanized pear-shape flask.

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**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS**

- 11.8 Add 20 mL of acetonitrile to the sample in the 50 mL centrifuge tube.
- 11.9 Shake the sample again for ~10 minutes on a wrist-action shaker.
- 11.10 Centrifuge the 50 mL polypropylene tubes containing sample at ~2000 rpm for ~5 minutes.
- 11.11 Decant the extract onto the same SPE column. Collect the eluate into the same pear-shaped flask and combine with the eluent from the initial extraction.
- 11.12 Repeat steps 11.8 through 11.11 again.
- 11.13 Add 3-4 drops of 1-octanol to the extract in the pear-shaped flask and evaporate at reduced pressure using a rotary evaporator (at < 40°C).
- 11.14 Make the final volume, by adding 2 mL of 2% ascorbic acid in methanol to the pear-shaped flask and swirl to mix/dissolve.
- 11.15 Transfer the extracts to HPLC vials using disposable pipets.
- 11.16 Analyze samples using electrospray LC/MS/MS.

**12.0 Chromatography**

- 12.1 Inject the same amount of each standard, sample and fortified sample into the LC/MS/MS system. A calibration standard must precede and follow all analyzed samples.
- 12.2 Standards of PFOA corresponding to at least five or more concentration levels must be included in an analytical set.
- 12.3 An entire set of extracted calibration standards must be included at the beginning and at the end of a sample set. Extracted standards must be interspersed between every 5-10 samples. As an alternative, an entire set of extracted calibration standards may be injected at the beginning of a set followed by extracted calibration standards interspersed every 5-10 samples (to account for a second set of extracted standards). In either case, extracted calibration standards must be the first and last injection in a sample set.
- 12.4 Use linear standard curves for quantitation. Linear standard curves are generated for the analyte by linear regression using 1/x weighting of peak area versus calibration standard concentration using MassLynx 3.3 (or equivalent) software system.
- 12.5 Sample response should not exceed standard responses. Any samples that exceed standard responses should be further diluted and reanalyzed.

**13.0 Acceptance Criteria**

- 13.1 Chromatogram must show a peak of a daughter ion at 369 amu from a parent of 413 amu. The 413 amu parent corresponds to the PFOA anion, while the daughter ion (369 amu) represents the loss of carbon dioxide.

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**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS

- 13.2 Method blanks must not contain PFOA at levels greater than the LOQ. If a blank contains PFOA at levels greater than 0.5 ppb, then a new blank sample must be obtained and the entire set must be re-extracted.
- 13.3 Recoveries of control spikes and matrix spikes must be between 70-130% of their known values. If a control spike falls outside the acceptable limits, the entire set of samples should be re-extracted.
- 13.4 Any calibration standard found to be a statistical outlier by using the Huge Error Test, may be excluded from the calculation of the calibration curve. However, the total number of calibration standards that could be excluded must not exceed 20% of the total number of standards injected.
- 13.5 The correlation coefficient (R) for calibration curves generated must be  $\geq 0.992$  ( $R^2 \geq 0.985$ ). If calibration results fall outside these limits, then appropriate steps must be taken to adjust instrument operation, and the standards or the relevant set of samples should be reanalyzed.
- 13.6 Retention times between standards and samples must not drift more than  $\pm 4\%$  within an analytical run. If retention time drift exceeds this limit within an analytical run then the set must be reanalyzed.

**14.0 Calculations**

- 14.1 Use the following equation to calculate the amount of PFOA found (in ng/mL, based on peak area) using the standard curve (linear regression parameters) generated by the Mass Lynx software program:

$$\text{PFOA found (ng/mL)} = \frac{(\text{Peak area} - \text{intercept})}{\text{slope}}$$

- 14.2 Use the following equation to convert the amount of PFOA found in ng/mL to ng/g (ppb).

$$\text{PFOA found (ppb)} = \frac{[\text{PFOA found (ng/mL)} \times \text{final volume (mL)} \times \text{DF}]}{\text{sample weight (g)}}$$

DF = factor by which the final volume was diluted, if necessary.

- 14.3 For samples fortified with known amounts of PFOA prior to extraction, use the following equation to calculate the percent recovery.

Recovery (%) =

$$\frac{[\text{total analyte found (ng/g)} - \text{analyte found in control (ng/g)}]}{\text{analyte added (ng/g)}} \times 100$$

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Exygen Protocol Number: P0000760

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**ANALYTICAL METHOD**

Method Number: V0001785

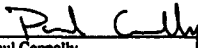
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**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS**

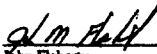
Analytical Testing Facility:

Exygen Research  
3058 Research Drive  
State College, PA 16801

Approved By:

  
Paul Connolly  
Technical Leader, LC-MS, Exygen Research

10/26/04  
Date

  
John Flaherty  
Vice President, Operations, Exygen Research

10/26/04  
Date

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Total Pages: 7

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001785

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS****1.0 Scope**

This method is to be employed for the isolation and quantitation of perfluorooctanoic acid by High Performance Liquid Chromatography coupled to a tandem Mass Spectrometric Detector (LC/MS/MS) in small mammal liver.

**2.0 Safety**

- 2.1 Always observe safe laboratory practices.
- 2.2 Consult the appropriate MSDS before handling any chemical for proper safety precautions.

**3.0 Sample Requirement**

- 3.1 At least 5 g of test sample for extraction.
- 3.2 Samples should be processed before extraction. Place the frozen sample in a food processor and homogenize with dry ice. Place the samples in containers and leave open in frozen storage overnight to allow for carbon dioxide sublimation. Seal and place the samples in frozen storage until time of analysis. Alternately, if there is an insufficient amount of sample (~less than 5 g), then no processing is necessary and the sample can be used as supplied.
- 3.3 Sample collection procedures will be specified in the sampling plan for this project.

**4.0 Reagents and Standards**

- 4.1 Water - HPLC grade
- 4.2 Methanol - HPLC grade
- 4.3 Acetonitrile - HPLC grade
- 4.4 Ammonium Acetate - A.C.S. Reagent Grade
- 4.5 Perfluorooctanoic Acid - Sigma-Aldrich

**5.0 Instrument and Equipment**

- 5.1 A high performance liquid chromatograph capable of pumping up to 2 solvents equipped with a variable volume injector capable of injecting 5-200  $\mu$ L connected to a tandem Mass Spectrometer (LC/MS/MS).
- 5.2 A device to collect raw data for peak integration and quantitation.
- 5.3 Analytical balance capable of reading to 0.00001 g.
- 5.4 50 mL disposable polypropylene centrifuge tubes.
- 5.5 15 mL disposable polypropylene centrifuge tubes.
- 5.6 Disposable micropipets (50-100 $\mu$ L, 100-200 $\mu$ L).
- 5.7 125-mL LDPE narrow-mouth bottles.
- 5.8 2 mL clear HPLC vial kit.

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Exygen Research

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**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS**

- 5.9 Disposable pipettes.  
5.10 Autopipettes (100-1000 µL and 10-100 µL), with disposable tips.  
5.11 Waters Sep Pak Vac 6 cc (1g) C18 SPE cartridges.  
5.12 SPE vacuum manifold.  
5.13 Tissue homogenizer.  
5.14 Wrist-action shaker.  
5.15 Centrifuge capable of spinning 15 mL polypropylene tubes at 3000 rpm.
- 6.0 Chromatographic System
- 6.1 Analytical Column: Fluorophase RP (Keystone Scientific), 2.1 mm x 50 mm, 5µ (P/N: 82505-052130)  
6.2 Temperature: 30°C  
6.3 Mobile Phase (A): 2 mM Ammonium Acetate in Water  
6.4 Mobile Phase (B): Methanol  
6.5 Gradient Program:

| Time (min) | % A | % B | Flow Rate (mL/min) |
|------------|-----|-----|--------------------|
| 0.0        | 65  | 35  | 0.3                |
| 1.0        | 65  | 35  | 0.3                |
| 8.0        | 25  | 75  | 0.3                |
| 20.0       | 25  | 75  | 0.3                |
| 22.5       | 65  | 35  | 0.3                |

- 6.6 Injection Volume: 15 µL (can be increased to as much as 50 µL).  
6.7 Quantitation: Peak Area - external standard calibration curve.  
6.8 Run Time: ~ 23 minutes.

The above conditions are intended as a guide and may be changed in order to optimize the HPLC system.

## 7.0 MS/MS System

- 7.1 Mode: Electrospray Negative MRM mode, monitoring 413 → 369 m/z for PFOA.

The above conditions are intended as a guide and may be changed in order to optimize the MS/MS system.

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Exygen Research

Method Number V0001785

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS****8.0 Preparation of Solutions****8.1 Mobile Phase**

- 8.1.1 2 mM ammonium acetate in water is prepared by adding 0.154 g of ammonium acetate to 1000 mL of water.

Alternate volumes may be prepared.

**9.0 Standard Preparation****9.1 Standard Stock/Fortification Solution**

- 9.1.1 Prepare a stock solution of ~100 µg/mL of PFOA by weighing 10 mg of analytical standard (corrected for purity) and dilute to 100 mL with methanol in a 125-mL LDPE bottle.
- 9.1.2 A 1.0 µg/mL fortification solution of PFOA is prepared by bringing 1 mL of the 100 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.3 A 0.1 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 1.0 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.4 The stock and fortification solutions are to be stored in a refrigerator at approximately 4°C and are stable for a maximum period of 6 months from the date of preparation.

**9.2 Standard Calibration Solutions**

- 9.2.1 LC/MS/MS calibration standards are prepared in methanol via dilution of the 0.1 µg/mL fortification solution.
- 9.2.2 The following is a typical example: additional concentrations may be prepared as needed.

| Concentration of Fortification Solution (µg/mL) | Volume (mL) | Diluted to (mL) | Final Concentration (ng/mL) |
|-------------------------------------------------|-------------|-----------------|-----------------------------|
| 100                                             | 5.0         | 100             | 5.0                         |
| 100                                             | 2.0         | 100             | 2.0                         |
| 100                                             | 1.0         | 100             | 1.0                         |
| 5.0                                             | 10          | 100             | 0.5                         |
| 2.0                                             | 10          | 100             | 0.2                         |
| 1.0                                             | 10          | 100             | 0.1                         |

- 9.2.3 Store all calibration standards in 125-mL LDPE narrow-mouth bottles at 2°C to 6°C, up to six months.

- 9.2.4 Alternate volumes and concentrations of standards may be prepared as needed.

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Exygen Research

Method Number V0001785

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS****10.0 Batch Set Up**

- 10.1 Each batch of samples extracted (typically 20 or less) must include at least one untreated control and two untreated controls fortified at known concentrations (lab control spike) to verify procedural recovery for the batch.
- 10.2 Requirements for field and laboratory duplicates and spikes will be specified in the quality assurance plan for this project.

**11.0 Sample Extraction**

- 11.1 Weigh 1 g of sample into a 50 mL polypropylene centrifuge tubes (fortify as needed, replace lid and mix well). Note that alternate weights of liver may be measured depending on the sample size available for use.
- 11.2 Add water to the sample for a final volume of 10 mL.
- 11.3 Homogenize sample using a tissue grinder for ~1 minute.
- 11.4 Transfer 1 mL of the sample using a disposable pipette into a 15 mL disposable centrifuge tube.
- 11.5 Add 5 mL of acetonitrile and shake for ~20 minutes on a wrist-action shaker.
- 11.6 Centrifuge the tubes at ~3000 rpm for ~5 minutes.
- 11.7 Decant the supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.
- 11.8 Condition the C<sub>18</sub> SPE cartridges (1 g, 6 mL) by passing 10 mL methanol followed by 5 mL of HPLC water (~2 drop/sec). Do not let column run dry.
- 11.9 Load the sample on conditioned C<sub>18</sub> SPE cartridge. Discard eluate.
- 11.10 Elute with ~2 mL of methanol. Collect 2 mL of eluate into a graduated 15 mL polypropylene centrifuge tube (final volume = 2 mL).
- 11.11 Analyze samples using electrospray LC/MS/MS.

**12.0 Chromatography**

- 12.1 Inject the same amount of each standard, sample and fortified sample into the LC/MS/MS system. A calibration standard must precede and follow all analyzed samples.
- 12.2 Standards of PFOA corresponding to at least five or more concentration levels must be included in an analytical set.
- 12.3 An entire set of calibration standards must be included at the beginning and at the end of a sample set. Standards must be interspersed between every 5-10 samples. As an alternative, an entire set of calibration standards may be injected at the beginning of a set followed by calibration standards interspersed every 5-10 samples (to account for a second set of standards). In either case, calibration standards must be the first and last injection in a sample set.
- 12.4 Use linear standard curves for quantitation. Linear standard curves are generated for the analyte by linear regression using 1/x weighting of peak area

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001783

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS**

versus calibration standard concentration using MassLynx 3.3 (or equivalent) software system.

- 12.5 Sample response should not exceed standard responses. Any samples that exceed standard responses should be further diluted and reanalyzed.

**13.0 Acceptance Criteria**

- 13.1 Chromatogram must show a peak of a daughter ion at 369 amu from a parent of 413 amu. The 413 amu parent corresponds to the PFOA anion, while the daughter ion (369 amu) represents the loss of carbon dioxide.
- 13.2 Method blanks must not contain PFOA at levels greater than the LOQ. If a blank contains PFOA at levels greater than 10 ng/g, then a new blank sample must be obtained and the entire set must be re-extracted.
- 13.3 Recoveries of control spikes and matrix spikes must be between 70-130% of their known values. If a control spike falls outside the acceptable limits, the entire set of samples should be re-extracted. Any matrix spike outside 70-130% should be evaluated by the analyst to determine if re-extraction is warranted.
- 13.4 Any calibration standard found to be a statistical outlier by using the Huge Error Test, may be excluded from the calculation of the calibration curve. However, the total number of calibration standards that could be excluded must not exceed 20% of the total number of standards injected.
- 13.5 The correlation coefficient (R) for calibration curves generated must be  $\geq 0.992$  ( $R^2 \geq 0.983$ ). If calibration results fall outside these limits, then appropriate steps must be taken to adjust instrument operation, and the standards or the relevant set of samples should be reanalyzed.
- 13.6 Retention times between standards and samples must not drift more than  $\pm 4\%$  within an analytical run. If retention time drift exceeds this limit within an analytical run then the set must be reanalyzed.

**14.0 Calculations**

- 14.1 Use the following equation to calculate the amount of PFOA found (in ng/mL, based on peak area) using the standard curve (linear regression parameters) generated by the Mass Lynx software program:

$$\text{PFOA found (ng/mL)} = \frac{(\text{Peak area} - \text{intercept})}{\text{slope}} \times \text{DF} \times \text{aliquot factor}$$

DF = factor by which the final volume was diluted, if necessary.  
Aliquot factor = 10

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001785

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Liver by LC/MS/MS

- 14.2 For samples fortified with known amounts of PFOA prior to extraction, use the following equation to calculate the percent recovery.

Recovery (%) =

$$\frac{[\text{total analyte found (ng/mL)} - \text{analyte found in control (ng/mL)}]}{\text{analyte added (ng/mL)}} \times 100$$

- 14.3 Use the following equation to convert the amount of PFOA found in ng/mL to ng/g (ppb).

$$\text{PFOA found (ppb)} = \frac{[\text{PFOA found (ng/mL)} \times \text{final volume (mL)}]}{\text{sample weight (g)}}$$

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Exygen Protocol Number: P0000760

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**ANALYTICAL METHOD**

Method Number: V0001786

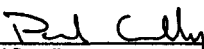
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**Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS**

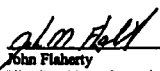
Analytical Testing Facility:

Exygen Research  
3058 Research Drive  
State College, PA 16801

Approved By:

  
Paul Connolly  
Technical Leader, LC-MS, Exygen Research

10/26/04  
Date

  
John Flaherty  
Vice President, Operations, Exygen Research

10/26/04  
Date

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Total Pages: 7

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001786

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS****1.0 Scope**

This method is to be employed for the isolation and quantitation of perfluorooctanoic acid by High Performance Liquid Chromatography coupled to a tandem Mass Spectrometric Detector (LC/MS/MS) in small mammal serum.

**2.0 Safety**

- 2.1 Always observe safe laboratory practices.
- 2.2 Consult the appropriate MSDS before handling any chemical for proper safety precautions.

**3.0 Sample Requirement**

- 3.1 At least 1 mL of test sample for extraction.
- 3.2 No sample processing is needed for serum samples. However, frozen serum samples must be allowed to completely thaw to room temperature before use.
- 3.3 Sample collection procedures will be specified in the sampling plan for this project.

**4.0 Reagents and Standards**

- 4.1 Water - HPLC grade
- 4.2 Methanol - HPLC grade
- 4.3 Acetonitrile - HPLC grade
- 4.4 Ammonium Acetate - A.C.S. Reagent Grade
- 4.5 Perfluorooctanoic Acid - Sigma-Aldrich

**5.0 Instrument and Equipment**

- 5.1 A high performance liquid chromatograph capable of pumping up to 2 solvents equipped with a variable volume injector capable of injecting 5-200  $\mu$ L connected to a tandem Mass Spectrometer (LC/MS/MS).
- 5.2 A device to collect raw data for peak integration and quantitation.
- 5.3 Analytical balance capable of reading to 0.00001 g.
- 5.4 50 mL disposable polypropylene centrifuge tubes.
- 5.5 15 mL disposable polypropylene centrifuge tubes.
- 5.6 Disposable micropipets (50-100 $\mu$ L, 100-200 $\mu$ L).
- 5.7 125-mL LDPE narrow-mouth bottles.
- 5.8 2 mL clear HPLC vial kit.
- 5.9 Disposable pipettes.
- 5.10 Autopipettes (100-1000  $\mu$ L and 10-100  $\mu$ L), with disposable tips.
- 5.11 Waters Sep Pak Vac 6 cc (1g) C18 SPE cartridges.
- 5.12 SPE vacuum manifold.
- 5.13 Vortexer.

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001786

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS

- 5.14 Wrist-action shaker.  
5.15 Centrifuge capable of spinning 15 mL polypropylene tubes at 3000 rpm.

**6.0 Chromatographic System**

- 6.1 Analytical Column: Fluophase RP (Keystone Scientific), 2.1 mm x 50 mm, 5 $\mu$  (P/N: 82505-052130)  
6.2 Temperature: 30°C  
6.3 Mobile Phase (A): 2 mM Ammonium Acetate in Water  
6.4 Mobile Phase (B): Methanol  
6.5 Gradient Program:

| Time (min) | % A | % B | Flow Rate (mL/min) |
|------------|-----|-----|--------------------|
| 0.0        | 65  | 35  | 0.3                |
| 1.0        | 65  | 35  | 0.3                |
| 8.0        | 25  | 75  | 0.3                |
| 20.0       | 25  | 75  | 0.3                |
| 22.5       | 65  | 35  | 0.3                |

- 6.6 Injection Volume: 15  $\mu$ L (can be increased to as much as 50  $\mu$ L).  
6.7 Quantitation: Peak Area -- external standard calibration curve.  
6.8 Run Time: ~ 23 minutes.

The above conditions are intended as a guide and may be changed in order to optimize the HPLC system.

**7.0 MS/MS System**

- 7.1 Mode: Electrospray Negative MRM mode, monitoring 413  $\rightarrow$  369 m/z for PFOA.

The above conditions are intended as a guide and may be changed in order to optimize the MSMS system.

**8.0 Preparation of Solutions****8.1 Mobile Phase**

- 8.1.1 2 mM ammonium acetate in water is prepared by adding 0.154 g of ammonium acetate to 1000 mL of water.

Alternate volumes may be prepared.

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001786

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS

**9.0 Standard Preparation****9.1 Standard Stock/Fortification Solution**

- 9.1.1 Prepare a stock solution of ~100 µg/mL of PFOA by weighing 10 mg of analytical standard (corrected for purity) and dilute to 100 mL with methanol in a 125-mL LDPE bottle.
- 9.1.2 A 1.0 µg/mL fortification solution of PFOA is prepared by bringing 1 mL of the 100 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.3 A 0.1 µg/mL fortification solution of PFOA is prepared by bringing 10 mL of the 1.0 µg/mL solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.
- 9.1.4 The stock and fortification solutions are to be stored in a refrigerator at approximately 4°C and are stable for a maximum period of 6 months from the date of preparation.

**9.2 Standard Calibration Solutions**

- 9.2.1 LC/MS/MS calibration standards are prepared in methanol via dilution of the 0.1 µg/mL fortification solution.
- 9.2.2 The following is a typical example: additional concentrations may be prepared as needed.

| Concentration of Fortification Solution (ng/mL) | Volume (mL) | Diluted to (mL) | Final Concentration (ng/mL) |
|-------------------------------------------------|-------------|-----------------|-----------------------------|
| 100                                             | 5.0         | 100             | 5.0                         |
| 100                                             | 2.0         | 100             | 2.0                         |
| 100                                             | 1.0         | 100             | 1.0                         |
| 5.0                                             | 10          | 100             | 0.5                         |
| 2.0                                             | 10          | 100             | 0.2                         |
| 1.0                                             | 10          | 100             | 0.1                         |

9.2.3 Store all calibration standards in 125-mL LDPE narrow-mouth bottles at 2°C to 6°C, up to six months.

9.2.4 Alternate volumes and concentrations of standards may be prepared as needed.

**10.0 Batch Set Up**

- 10.1 Each batch of samples extracted (typically 20 or less) must include at least one untreated control and two untreated controls fortified at known concentrations (lab control spike) to verify procedural recovery for the batch.
- 10.2 Requirements for field and laboratory duplicates and spikes will be specified in the quality assurance plan for this project.

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001786

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS

**11.0 Sample Extraction**

- 11.1 Measure 1 mL of sample into a 50 mL polypropylene centrifuge tubes (fortify as needed, replace lid and mix well). Note that alternate volumes of serum may be measured depending on the sample size available for use.
- 11.2 Add water to the sample for a final volume of 20 mL. Cap tightly
- 11.3 Vortex for ~1 minute.
- 11.4 Transfer 1 mL of the sample using a disposable pipette into a 15 mL disposable centrifuge tube.
- 11.5 Add 5 mL of acetonitrile and shake for ~20 minutes on a wrist-action shaker.
- 11.6 Centrifuge the tubes at ~3000 rpm for ~5 minutes.
- 11.7 Decant the supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.
- 11.8 Condition the C<sub>18</sub> SPE cartridges (1 g, 6 mL) by passing 10 mL methanol followed by 5 mL of HPLC water (~2 drop/sec). Do not let column run dry
- 11.9 Load the sample on conditioned C<sub>18</sub> SPE cartridge. Discard eluate.
- 11.10 Elute with ~2 mL of methanol. Collect 2 mL of eluate into a graduated 15 mL polypropylene centrifuge tube (final volume = 2 mL).
- 11.11 Analyze samples using electrospray LC/MS/MS.

**12.0 Chromatography**

- 12.1 Inject the same amount of each standard, sample and fortified sample into the LC/MS/MS system. A calibration standard must precede and follow all analyzed samples.
- 12.2 Standards of PFOA corresponding to at least five or more concentration levels must be included in an analytical set.
- 12.3 An entire set of calibration standards must be included at the beginning and at the end of a sample set. Standards must be interspersed between every 5-10 samples. As an alternative, an entire set of calibration standards may be injected at the beginning of a set followed by calibration standards interspersed every 5-10 samples (to account for a second set of standards). In either case, calibration standards must be the first and last injection in a sample set.
- 12.4 Use linear standard curves for quantitation. Linear standard curves are generated for the analyte by linear regression using 1/x weighting of peak area versus calibration standard concentration using MassLynx 3.3 (or equivalent) software system.
- 12.5 Sample response should not exceed standard responses. Any samples that exceed standard responses should be further diluted and reanalyzed.

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001786

**ANALYTICAL METHOD**

Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS

**13.0 Acceptance Criteria**

- 13.1 Chromatogram must show a peak of a daughter ion at 369 amu from a parent of 413 amu. The 413 amu parent corresponds to the PFOA anion, while the daughter ion (369 amu) represents the loss of carbon dioxide.
- 13.2 Method blanks must not contain PFOA at levels greater than the LOQ. If a blank contains PFOA at levels greater than 10 ng/mL, then a new blank sample must be obtained and the entire set must be re-extracted.
- 13.3 Recoveries of control spikes and matrix spikes must be between 70-130% of their known values. If a control spike falls outside the acceptable limits, the entire set of samples should be re-extracted. Any matrix spike outside 70-130% should be evaluated by the analyst to determine if re-extraction is warranted.
- 13.4 Any calibration standard found to be a statistical outlier by using the Huge Error Test, may be excluded from the calculation of the calibration curve. However, the total number of calibration standards that could be excluded must not exceed 20% of the total number of standards injected.
- 13.5 The correlation coefficient (R) for calibration curves generated must be  $\geq 0.992$  ( $R^2 \geq 0.985$ ). If calibration results fall outside these limits, then appropriate steps must be taken to adjust instrument operation, and the standards or the relevant set of samples should be reanalyzed.
- 13.6 Retention times between standards and samples must not drift more than  $\pm 4\%$  within an analytical run. If retention time drift exceeds this limit within an analytical run then the set must be reanalyzed.

**14.0 Calculations**

- 14.1 Use the following equation to calculate the amount of PFOA found (in ng/mL, based on peak area) using the standard curve (linear regression parameters) generated by the Mass Lynx software program:

$$\text{PFOA found (ng/mL)} = \frac{(\text{Peak area} - \text{intercept})}{\text{slope}} \times \text{DF} \times \text{aliquot factor}$$

DF = factor by which the final volume was diluted, if necessary.  
Aliquot factor = 20

- 14.2 For samples fortified with known amounts of PFOA prior to extraction, use the following equation to calculate the percent recovery.

Recovery (%) =

$$\frac{[\text{total analyte found (ng/mL)} - \text{analyte found in control (ng/mL)}]}{\text{analyte added (ng/mL)}} \times 100$$

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Exygen Protocol Number: P0000760

Exygen Research

Method Number V0001786

**ANALYTICAL METHOD****Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Small Mammal Serum by LC/MS/MS**

- 14.3 Use the following equation to convert the amount of PFOA found in ng/mL to ppb.

$$\text{PFOA found (ppb)} = \frac{[\text{PFOA found (ng/mL)} \times \text{final volume (mL)}]}{\text{sample volume (mL)}}$$

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3058 Research Drive  
State College, PA 16801Phone: 814-272-1039  
Fax: 814-231-1580**PROTOCOL AMENDMENT**Amendment Number: 1Effective Date: 01/19/05Exygen Study Number: P0000760 Client Study Number: None

Page 1 of 1

**DESCRIPTION OF AMENDED SECTION**

- 1) Analytical Procedure Summary V0001780: Section 9.1
- 2) Verification of Analytical Procedure

**AMENDED TO**

- 1) Add to Section 9.1: Section 9.1.6, Alternate weights of standards may be used to prepare alternate concentrations of stock solutions as necessary. Alternate levels of fortification solutions may also be prepared.
- 2) Low and high spiking levels of the analytes for each matrix may be altered depending on sample size available for extraction and/or to cover analyte concentrations expected in the samples.

**RATIONALE**

- 1) Higher concentrations of standards need to be prepared in order to spike the sample bottles at higher levels.
- 2) The sample size available for small mammal liver and serum was smaller than expected. Spiking at the pre-determined levels in the protocol puts the spiked concentration lower than the detection limit. Also, the analyte levels in the ground water samples are expected to greatly exceed the pre-determined spiking levels listed in the protocol. When the levels in the samples greatly exceed the spiking levels, an accurate recovery value cannot be calculated for the QC sample. Higher spiking levels in the bottles will cover the analyte concentrations expected in the water samples.

**IMPACT ON STUDY**

The LOQ is 100 ng/g for a 0.1 g sample of small mammal liver and is 1000 ng/mL for a 0.01 mL sample of small mammal serum.  
Higher levels of spiking for the water samples will ensure that more QC recovery data can be used.

Study Director Signature

Principal Investigator Signature

Study Director Management Signature

Sponsor Signature (if required)

Date

Date

Date

Date

Exygen QAU Review LJS 02/01/05

LIBRARY ID: VV0001226-6

ADMINISTRATIVE FORM

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## PROTOCOL AMENDMENT

Amendment Number:

2

Page 1 of 1

Effective Date:

03/07/05

Exygen Study Number

P0000760

Client Study Number:

NoneDESCRIPTION OF AMENDED SECTION

Report, page 11 of 65

AMENDED TO

Instead of one final report, Interim reports will be issued.

RATIONALEDue to the excessive sizes of the data sets, interim reports will be issued to allow the client to receive data in a timelier manner. *manner as before*IMPACT ON STUDY

The client will be able to receive and review the data more quickly.

Study Director Signature

Date

Principal Investigator Signature

Date

Study Director Management Signature

Date

Exygen Management Signature

Date

Sponsor Signature (if required)

Date

Exygen-QAU Review LJS 03/08/05

LIBRARY ID: V0001228-8

ADMINISTRATIVE FORM

06-02-2005 09:11am Free-NESTON SOLUTIONS

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## PROTOCOL AMENDMENT

Amendment Number:

3

Page 1 of 1

Effective Date:

04/13/05

Exygen Study Number

P760

Client Study Number:

NA

## DESCRIPTION OF AMENDED SECTION

Analytical Procedure Summary: V0001780: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS." Sections 9.2 and 11.0 of the method.

## AMENDED TO

Section 9.2. Non-extracted calibration standards may be prepared in order to quantitate samples directly injected onto the LC/MS/MS system.

Section 11.0. Samples may be analyzed without going through the extraction procedure. Instead, they may be diluted, if appropriate, and directly injected onto the LC/MS/MS system for analysis.

## RATIONALE

Some extracted results between laboratory and field spikes were not in agreement and/or were unusable. Directly injecting the extracts may add some insight into the matrix effects on the data results.

## IMPACT ON STUDY

More usable data results may be obtained.

Study Director Signature

Date

Principal Investigator Signature

Date

Study Director Management Signature

Date

Exygen Management Signature

Date

Sponsor Signature (if required)

Date

Exygen QAU Review 06/02/05

LIBRARY ID: V0001228-8

ADMINISTRATIVE FORM

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PRINT TIME JUN. 2. 9:30AM

JUL. 25. 2005 8:56AM EXYGEN RESEARCH

NO. 774 P. 4

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## PROTOCOL AMENDMENT

Amendment Number:

4

Page 1 of 1

Effective Date:

07/18/05

Exygen Study Number

P760

Client Study Number:

NA

DESCRIPTION OF AMENDED SECTION

Verification of Analytical Procedure, page 10 of protocol.

AMENDED TO

The field duplicate can be used for the laboratory spikes and replicate when the primary sample volume is limited.

RATIONALE

The sample size for a water sample is 200 mL. If a sample site requires re-extraction for any reason, there would not be enough of the primary sample to repeat two laboratory spikes and a replicate. The field duplicate is technically the same sample as the primary sample and therefore, can be used for laboratory spikes and replicates as needed.

IMPACT ON STUDY

No negative impact on the study. Using the duplicate sample allows for the full QC of the sample site to be completed.

Study Director Signature

Date

Principal Investigator Signature

Date

Study Director Management Signature

Date

Exygen Management Signature

Date

Sponsor Signature (if required)

Date

Exygen QAU Review *m.m. 7/22/05*

LIBRARY ID: V0001226-8

ADMINISTRATIVE FORM



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# PROTOCOL AMENDMENT

Amendment Number: 5 Page 1 of 1  
Effective Date: 09/13/05  
Exygen Study Number P760 Client Study Number: NA

## DESCRIPTION OF AMENDED SECTION

Analytical Procedure Summary: V0001780: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS."

## AMENDED TO

The reanalysis of two water samples, DF09-GW-131R-0-040126 and DFB-GW-135L-0-040121 will be performed using the following procedure:

An aliquot of the water sample is spiked with the surrogate standard and/or other matrix spike compounds as appropriate. Add methanol to the fortified aqueous sample. Mix and aliquot for LC/MS/MS analysis. The LC conditions include the use of a Betasil C18, 2.1 x 50 mm column, a gradient of 30% B to 100% B in 10 minutes, where A=2mM ammonium acetate in water and B=Methanol (6 min equilibration). Use a 5 µL injection volume and a 0.3 mL/min flow rate. LC conditions may be further modified as appropriate for the system being used. An interim report will be issued that will include the exact details of the reanalysis.

## RATIONALE

DF09-GW-131R-0-040126 and DF8b-GW-135L-0-040121 could not be reported quantitatively in interim report #3 due to quality control sample failures. The study director and sponsor have requested reanalysis using the above conditions.

## IMPACT ON STUDY

No negative impact on the study. More usable data may be obtained.

Study Director Signature

Date

*[Signature]*

9/14/05

Principal Investigator Signature

Date

N/A

-

Study Director Management Signature

Date

*[Signature]*

14-Sep-05

Exygen Management Signature

Date

Sponsor Signature (if required)

Date

Exygen QAU Review m.w. 9/13/05

LIBRARY ID: V0001226-8

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651 733 1958

09/14 '05 09:27 NO.915 02/02

EP.13.2005

1:51PM

EXYGEN RESEARCH

NO. 234 P. 6

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## PROTOCOL AMENDMENT

Amendment Number:

5

Page 1 of 1

Effective Date:

09/13/05

Exygen Study Number

P760

Client Study Number:

NA

DESCRIPTION OF AMENDED SECTION

Analytical Procedure Summary: V0001780: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS."

AMENDED TO

The reanalysis of two water samples, DFC9-GW-131R-0-040126 and DFB-GW-135L-0-040121 will be performed using the following procedure:

An aliquot of the water sample is spiked with the surrogate standard and/or other matrix spike compounds as appropriate. Add methanol to the fortified aqueous sample. Mix and aliquot for LC/MS/MS analysis. The LC conditions include the use of a Betasil C18, 2.1 x 50 mm column, a gradient of 30% B to 100% B in 10 minutes, where A=2mM ammonium acetate in water and B=Methanol (5 min equilibration). Use a 5 µL injection volume and a 0.3 mL/min flow rate. LC conditions may be further modified as appropriate for the system being used. An interim report will be issued that will include the exact details of the reanalysis.

RATIONALE

DFC9-GW-131R-0-040126 and DFB-GW-135L-0-040121 could not be reported quantitatively in interim report #3 due to quality control sample failures. The study director and sponsor have requested reanalysis using the above conditions.

IMPACT ON STUDY

No negative impact on the study. More usable data may be obtained.

Study Director Signature

Date

Principal Investigator Signature

Date

Study Director Management Signature

Date

Exygen Management Signature

Date

Sponsor Signature (if required)

Date

Exygen QAU Review *M. ~ 9/13/05*

LIBRARY ID: V0001226-6

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SEP 13 2003 02:19 FROM: 3M ENV. LAB 651 778 6176  
SEP. 13. 2005 1:51PM EXYGEN RESEARCH

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NO. 294 P. 2



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**PROTOCOL AMENDMENT**

Amendment Number: 5 Page 1 of 1  
Effective Date: 09/13/05  
Exygen Study Number: P760 Client Study Number: NA

**DESCRIPTION OF AMENDED SECTION**

Analytical Procedure Summary: V0001780: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water by LC/MS/MS."

**AMENDED TO**

The reanalysis of two water samples, DF09-GW-131R-0-040126 and DFB-GW-135L-0-040121 will be performed using the following procedure:  
An aliquot of the water sample is spiked with the surrogate standard and/or other matrix spike compounds as appropriate. Add methanol to the fortified aqueous sample. Mix and aliquot for LC/MS/MS analysis. The LC conditions include the use of a Betasil C18, 2.1 x 50 mm column, a gradient of 30% B to 100% B in 10 minutes, where A=2mM ammonium acetate in water and B=Methanol (6 min equilibration). Use a 5 µL injection volume and a 0.3 mL/min flow rate. LC conditions may be further modified as appropriate for the system being used. An interim report will be issued that will include the exact details of the reanalysis.

**RATIONALE**

DF09-GW-131R-0-040126 and DFB-GW-135L-0-040121 could not be reported quantitatively in interim report #3 due to quality control sample failures. The study director and sponsor have requested reanalysis using the above conditions.

**IMPACT ON STUDY**

No negative impact on the study. More usable data may be obtained.

Study Director Signature 

Date 9/14/05

Principal Investigator Signature

Date

Study Director Management Signature

Date

Exygen Management Signature

Date

Sponsor Signature (if required)

Date

Exygen QAU Review m.w. 9/13/05

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## PROTOCOL AMENDMENT

Amendment Number: 6 Page 1 of 1  
Effective Date: 09/20/05  
Exygen Study Number P760 Client Study Number: NA

DESCRIPTION OF AMENDED SECTION

The identification of the two water samples listed in protocol amendment No 5 are incorrect.

AMENDED TO

The two water samples for reanalysis should be DF09-GW-131R-0-040120 and DF8b-GW-135R-0-040121.

RATIONALE

The correct samples are identified for reanalysis.

IMPACT ON STUDY

No negative impact on the study.

Study Director Signature

Date

9/20/05

Principal Investigator Signature

NA

Date

-

Study Director Management Signature

Date

23-SEP-05

Exygen Management Signature

Date

Sponsor Signature (if required)

Date

Exygen QAU Review MW 9/20/05