

TITLE PAGE

RAW DATA PACKAGE FOR INTERIM REPORT #12 Analysis of Vegetation 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, 2005

PERFORMING LABORATORY / TESTING FACILITY

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PROJECT

Protocol Number: P0000760
Exygen Study Number: P0000760

Total Pages: 834

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APPENDIX I

PROTOCOL

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:

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3058 Research Drive
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Sponsor Representative:

Michael A. Santoro
Director of Regulatory Affairs
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DISTRIBUTION:

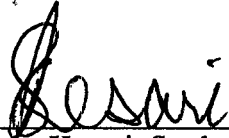
- 1) Jaisimha Kesari, Study Director, Weston Solutions
- 2) John M. Flaherty, Principal Investigator, Exygen Research
- 3) Michael A. Santoro, Sponsor Representative, 3M Company
- 4) Exygen Research Quality Assurance Unit

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

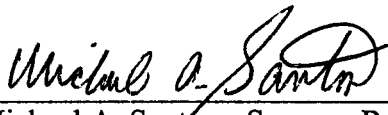
APPROVALS



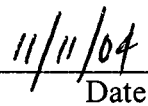
Jaisimha Kesari, Study Director
Weston Solutions




Date



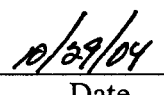
Michael A. Santoro, Sponsor Representative
3M Company



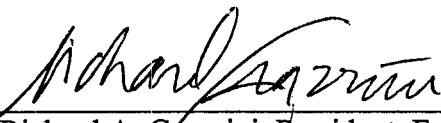
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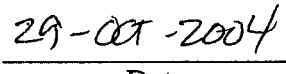
John M. Flaherty, Principal Investigator
Exygen Research



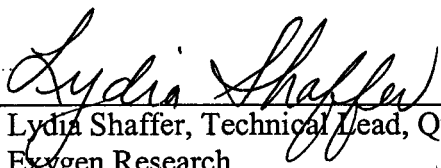
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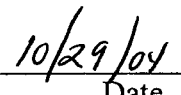
Richard A. Grazzini, President, Facility Management
Exygen Research



Date



Lydia Shaffer, Technical Lead, Quality Assurance Unit
Exygen Research



Date

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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

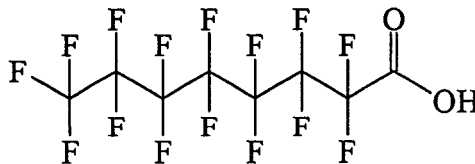
Molecular Weight: 414

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 ¹³C labeled perfluorooctanoic acid (¹³C PFOA) and was purchased by 3M from Perkin-Elmer Life and Analytical Sciences.

¹³C PFOA

Chemical Name: 1,2-¹³C perfluorooctanoic acid

Molecular Weight: 416

Lot Number: 3507-195

Purity: 97%

Transition Monitored: 415 → 370

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

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PRINCIPAL INVESTIGATOR

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john.flaherty@exygen.com

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

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 I). 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.

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 ¹³C 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

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}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}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}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}C PFOA results to establish precision and accuracy of PFOA samples. The following samples are to be prepared:

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

- 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).

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., $\mu\text{g/mL}$).

- 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.

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"

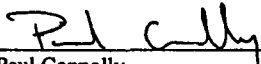
ANALYTICAL METHOD

Method Number: V0001780

**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

10/26/04

Date



John Flaherty
Vice President, Operations, Exygen Research

10/26/04

Date

Total Pages: 7

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 μ 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 μ L, 100-200 μ 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 μ L and 10-100 μ L), with disposable tips.
- 5.11 Waters Sep Pak Vac 6 cc (1g) tC18 SPE cartridges.

Exygen Research

Method Number V0001780

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, 5 μ
(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:

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>	<u>Flow Rate (mL/min)</u>
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 μ L (can be increased to as much as 50 μ 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.

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.

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	XCmmdyy-0
10	100	40	25	XCmmdyy-1
10	200	40	50	XCmmdyy-2
10	400	40	100	XCmmdyy-3
100	100	40	250	XCmmdyy-4
100	200	40	500	XCmmdyy-5
100	400	40	1000	XCmmdyy-6

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

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 C₁₈ 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 C₁₈ 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

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 ≥ 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.

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 (%) =

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

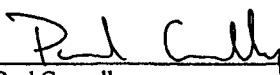
ANALYTICAL METHOD

Method Number: V0001781

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

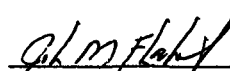
Analytical Testing Facility: Exygen Research
3058 Research Drive
State College, PA 16801

Approved By:



Paul Connolly
Technical Leader, LC-MS, Exygen Research

10/26/04
Date



John Flaherty
Vice President, Operations, Exygen Research

11/24/04
Date

Total Pages: 7

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 μ 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 μ L, 100-200 μ 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 μ L and 10-100 μ 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.

Oxygen Research

Method Number V0001781

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:

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>	<u>Flow Rate (mL/min)</u>
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.

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	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 8× its initial concentration, due to the concentration of the standard during the extraction (SPE).
XC = extracted calibration standard.

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 C₁₈ 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 C₁₈ 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

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 ≥ 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.

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.

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

ANALYTICAL METHOD

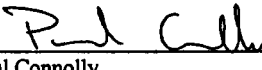
Method Number: V0001782

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

Analytical Testing Facility:

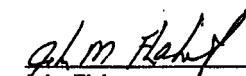
Exygen Research
3058 Research Drive
State College, PA 16801

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

Total Pages: 7

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 μ 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 μ L, 100-200 μ 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 μ L and 10-100 μ L), with disposable tips.
- 5.11 Waters Sep Pak Vac 6 cc (1g) tC18 SPE cartridges.
- 5.12 SPE vacuum manifold.

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 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.

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

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 C₁₈ 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 C₁₈ 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 C₁₈ 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.

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 ≥ 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.

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.

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

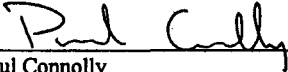
ANALYTICAL METHOD

Method Number: V0001783

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

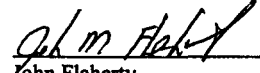
Analytical Testing Facility: Exygen Research
3058 Research Drive
State College, PA 16801

Approved By:



Paul Connolly
Technical Leader, LC-MS, Exygen Research

10/26/04
Date



John Flaherty
Vice President, Operations, Exygen Research

10/26/04
Date

Total Pages: 8

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-NH₂ – 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 μ 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.

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 Tissumizer.
- 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 (50-100uL, 100-200uL).
- 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 µL and 10-100 µL), 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: 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.

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 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.

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.

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-NH₂. 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.

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.

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 ≥ 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.

Oxygen Research

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 (%) =

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

ANALYTICAL METHOD

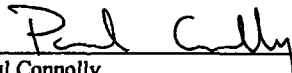
Method Number: V0001784

**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

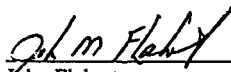
Approved By:



Paul Connolly
Technical Leader, LC-MS, Exygen Research

10/26/04

Date



John Flaherty
Vice President, Operations, Exygen Research

10/26/04

Date

Total Pages: 7

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-NH₂ – 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 μ 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.

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 (50-100uL, 100-200uL).
- 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 µL and 10-100 µL), 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, 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:

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>	<u>Flow Rate (mL/min)</u>
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.

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 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.

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.

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 ($\mu\text{g/mL}$)	Volume (mL)	Diluted to (mL)	Final Concentration ($\mu\text{g/mL}$)
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 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-NH₂. 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.

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.

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 ≥ 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 (%) =

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

ANALYTICAL METHOD

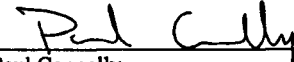
Method Number: V0001785

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

Analytical Testing Facility:

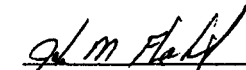
Exygen Research
3058 Research Drive
State College, PA 16801

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

Total Pages: 7

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 μ 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 μ L, 100-200 μ L).
- 5.7 125-mL LDPE narrow-mouth bottles.
- 5.8 2 mL clear HPLC vial kit.

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Method Number V0001785

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 μ L and 10-100 μ L), with disposable tips.
- 5.11 Waters Sep Pak Vac 6 cc (1g) tC18 SPE cartridges.
- 5.12 SPE vacuum manifold.
- 5.13 Tissuemizer.
- 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 μ (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:

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>	<u>Flow Rate (mL/min)</u>
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 μ L (can be increased to as much as 50 μ 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.

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 (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.

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 tissuemizer 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 C₁₈ 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 C₁₈ 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

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 ≥ 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 = 10

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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).

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

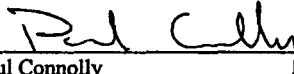
ANALYTICAL METHOD

Method Number: V0001786

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

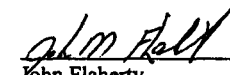
Analytical Testing Facility: Exygen Research
3058 Research Drive
State College, PA 16801

Approved By:



Paul Connolly
Technical Leader, LC-MS, Exygen Research

10/26/04
Date



John Flaherty
Vice President, Operations, Exygen Research

11/2/04
Date

Total Pages: 7

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 μ 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 μ L, 100-200 μ 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 μ L and 10-100 μ L), with disposable tips.
- 5.11 Waters Sep Pak Vac 6 cc (1g) tC18 SPE cartridges.
- 5.12 SPE vacuum manifold.
- 5.13 Vortexer.

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, 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:

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>	<u>Flow Rate (mL/min)</u>
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.

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.

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 C₁₈ 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 C₁₈ 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.

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 ≥ 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 (%) =

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

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.

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

PROTOCOL AMENDMENT

Amendment Number: 1

Effective Date: 01/19/05

Exygen 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.

[Signature]
Study Director Signature

3/9/05
Date

[Signature]
Principal Investigator Signature

2/2/05
Date

NA
Study Director Management Signature

NA
Date

[Signature]
Sponsor Signature (if required)

3/10/05
Date

Exygen QAU Review LJS 02/10/05

PROTOCOL AMENDMENT

Amendment Number: 2 Page 1 of 1
Effective Date: 03/07/05
Exygen Study Number: P0000760 Client Study Number: None

<u>DESCRIPTION OF AMENDED SECTION</u>	
Report, page 11 of 65	
<u>AMENDED TO</u>	
Instead of one final report, interim reports will be issued.	
<u>RATIONALE</u>	
Due to the excessive sizes of the data sets, interim reports will be issued to allow the client to receive data in a timelier manner. <i>manner 03/09/05 RAZ</i>	
<u>IMPACT ON STUDY</u>	
The client will be able to receive and review the data more quickly.	
Study Director Signature <i>[Signature]</i>	Date <u>3/9/05</u>
Principal Investigator Signature <i>[Signature]</i>	Date <u>3/9/05</u>
Study Director Management Signature <i>[Signature]</i>	Date <u>8-MAR-05</u>
Exygen Management Signature <i>[Signature]</i>	Date <u>3/10/05</u>
Sponsor Signature (if required) <i>[Signature]</i>	Date <u>3/10/05</u>
Exygen QAU Review <u>LJS 03/08/05</u>	

APPENDIX II

ANALYTICAL METHOD

ANALYTICAL METHOD

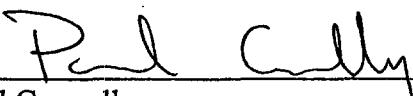
Method Number: V0001784

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

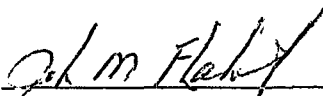
Approved By:



Paul Connolly
Technical Leader, LC-MS, Exygen Research

10/26/04

Date



John Flaherty
Vice President, Operations, Exygen Research

10/26/04

Date

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-NH₂ – 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 μ 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.

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 (50-100uL, 100-200uL).
- 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 μ L and 10-100 μ 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 μ (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:

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>	<u>Flow Rate (mL/min)</u>
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 μ L (can be increased to as much as 50 μ 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.

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 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.

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 $\mu\text{g/mL}$ 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 $\mu\text{g/mL}$ fortification solution of PFOA is prepared by bringing 1 mL of the 100 $\mu\text{g/mL}$ solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.

9.1.3 A 0.1 $\mu\text{g/mL}$ fortification solution of PFOA is prepared by bringing 10 mL of the 1.0 $\mu\text{g/mL}$ solution to a final volume of 100 with methanol in a 125 mL LDPE bottle.

9.1.4 A 0.01 $\mu\text{g/mL}$ fortification solution of PFOA is prepared by bringing 10 mL of the 0.1 $\mu\text{g/mL}$ 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 $\mu\text{g/mL}$ fortification solution.

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 ($\mu\text{g/mL}$)	Volume (mL)	Diluted to (mL)	Final Concentration ($\mu\text{g/mL}$)
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 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-NH₂. 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.

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.

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 ≥ 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 (%) =

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

APPENDIX III

STANDARD INFORMATION

Standards - Primary

SP0003800 > 3845

Description: Pentadecafluorooctanoic acid
 Type: Routine
 Sponsor Contact:
 Matrix: Solids (Gravimetric)
 Catalog #: 171468
 Lot #: 23116HB
 CAS #: 335-67-1
 Solvent: NONE
 Removed: False
 Condition Recv'd: Room Temperature
 Condition Stored: Room Temperature
 Current Location: B1-105
 Vendor: SIGMA-ALDRICH, INC.
 Company Name:
 Contact:
 Phone #:
 Notes:

Date Received: 12/08/2003
 Expires: 12/8/2005 12:00:00AM
 Special: No significant hazard
 Flammable: Non-combustible (will not burn)
 Health: Slightly toxic
 Reactive: No significant hazard
 Gross: 43.03219
 Gross Units: g
 Net Balance: 4.23143
 Net Units: g

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BY BAK DATE 08/03/05

Oxygen STUDY NO. P760
BAK 08/03/05

Aliquot - SP0003800

Addition	Component	Withdrawal	Units	Component	Purpose	Computer	Created On	Created By
5			g		INITIAL		12/8/2003 12:56:03PM	Risha, Karen
0082		0.10242	g	Balance 14	ANALYSIS	BALANCE5	12/8/2003 1:53:57PM	Risha, Karen
		0.10242	g	Balance 14	ANALYSIS	BALANCE5	3/2/2004 11:45:51AM	Sheehan, Amy
		0.010244	g	Balance 32	ANALYSIS	BALANCE7	6/1/2004 10:32:52AM	Decker, Emily
		0.010242	g	Balance 32	ANALYSIS	BALANCE7	11/17/2004 11:28:49AM	Sheehan, Amy
		0.01047	g	Balance 17	ANALYSIS	BALANCE8	11/22/2004 11:15:04AM	Decker, Emily
		0.10242	g	Balance 17	ANALYSIS	BALANCE8	12/20/2004 8:17:11AM	Sheehan, Amy
		0.10242	g	Balance 17	ANALYSIS	BALANCE8	1/19/2005 11:44:19AM	Sheehan, Amy



SP0003800 > 3845

Description: Pentadecafluorooctanoic acid
 Type: Routine
 Sponsor Contact:
 Matrix: Solids (Gravimetric)
 Catalog #: 171468
 Lot #: 23116HB
 CAS #: 335-67-1
 Solvent: NONE
 Removed: False
 Condition Recv'd: Room Temperature
 Condition Stored: Room Temperature
 Current Location: B1-105
 Vendor: SIGMA-ALDRICH, INC.
 Company Name:
 Contact:
 Phone #:
 Notes:

Date Received: 12/08/2003
 Expires: 12/8/2005 12:00:00AM
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 Gross: 43.03219
 Gross Units: g
 Net Balance: 4.23143
 Net Units: g

Oxygen STUDY NO. P760
BAK 08/03/05

Aliquot - SP0003800, cont'd

Addition	Component	Withdrawal	Units	Component	Purpose	Computer	Created On	Created By
			Notes: P760					
		0.01024	g	Balance 14	PREPARE	BALANCE5	2/22/2005 9:06:53AM	Connolly, Paul
			Notes:					
		0.10242	g	Balance 17	OTHER	BALANCE8	5/17/2005 10:55:17AM	Edwards, Chrissy
			Notes: Prepare stock for P760					
		0.10242	g	Balance 17	ANALYSIS	BALANCE8	6/16/2005 8:18:43AM	Sheehan, Amy
			Notes: Fluorochemicals					
		0.10242	g	Balance 17	ANALYSIS	BALANCE8	6/20/2005 3:09:46PM	Kravets, Brittany
			Notes: Stocks for fluorochemicals.					
		0.010434	g	Balance 32	PREPARE	BALANCE7	7/22/2005 10:54:43AM	Smith, Jennifer
			Notes:					

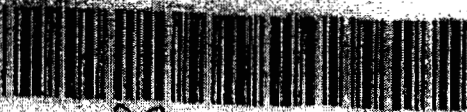
0003

Characterization - SP0003800

Purity 97.64 % Date Characterized 12/08/2003 Date Expires 12/08/2005 Characterized By Sigma-Aldrich Created By Risha, Karen

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SP3800



SIGMA-ALDRICH

Allentown Distribution Center
Aldrich Chemical Company, Inc.
6950 AMBASSADOR DRIVE
Allentown PA 18106

Oxygen STUDY NO. P760

Handwritten: 8/11/17/04

SHIP TO:
OXYGEN RESEARCH
KAREN
3048 RESEARCH DR
STATE COLLEGE PA 16801

Customer Service 800-325-3010 • <http://www.sigma-aldrich.com>

PAGE 1 of 1 DELIVERY REFERENCE

DATE	ORDER NO.	SOLD TO NAME	PURCHASE ORDER NUMBER	REFERENCE	
12/05/2003	49425591	OXYGEN RESEARCH	4031KR10	927024	
ROUTE		PERSON TO CONTACT			
AIRBORNE GROUND PCA SHIPPING POINT		NIM DOELITTLE			
STOCK NO.	SIGMA NO.	ORDERED	SHIPPED	BACKLOG	DESCRIPTION
171468-5G	23114AB			0	PENTADECYLFLUOROOCTANOIC ACID, 94% ENTRY OF OR: US
<p>The 2004-2005 Sigma-Aldrich General Catalog will be available soon. Get a FREE Metabolic Pathways Chart when you reserve your copy. www.sigma-aldrich.com</p>					

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SP3800



SIGMA-ALDRICH

Certificate of Analysis

Product Name	Pentadecafluorooctanoic acid
Product Number	17,146-8
CAS Number	335-67-1
Molecular Formula	$C_8HF_{15}O_2$
Molecular Weight	414.1

TEST	SPECIFICATION	LOT 23116HB RESULTS
APPEARANCE	WHITE TO OFF-WHITE POWDER	WHITE CRYSTALLINE POWDER
INFRARED SPECTRUM	CONFORMS TO STRUCTURE AND STANDARD AS ILLUSTRATED ON PAGE 507B OF EDITION I, VOLUME 1 OF 'THE ALDRICH LIBRARY OF FT-IR SPECTRA'	CONFORMS TO STRUCTURE AND STANDARD
TITRATION	95.5% - 104.5% (WITH NaOH)	101.1% (WITH NaOH)
GAS LIQUID CHROMATOGRAPHY	95.5% (MINIMUM)	97.7%
TITRATION	2% H ₂ O (MAXIMUM)	0.06% H ₂ O (WITH 'KARL FISCHER' REAGENT)
QUALITY CONTROL ACCEPTANCE DATE		JUNE, 2003

Ronnie J. Martin, Supervisor
Quality Control

Oxygen STUDY NO. P760

RB 11/17/04

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Protocol No.: _____ Exygen Study No.: _____ Page 1 of 1

STUDY TELEPHONE/COMMUNICATIONS LOG

Person: Ken Originator: Exygen x
Company: Aldrich Sponsor _____
Date: 12/08/03
Time: 1022

Notes:

I called Aldrich tech service at 1-800-231-8327 to ask the shelf life of the PFOA standard
I received today. Ken, an Aldrich rep in technical service, told me that the shelf life is
2 years from the date of receipt stored at room temperature.

Signature: *Shenasha* Date: 12/08/03

Exygen STUDY NO. P760

12/17/04

August 7, 2001/1

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Standards - Primary

SP0004184 > 4228

Description: Perfluorooctanoic Acid, Radiolabeled
 Type: Routine
 Sponsor Contact:
 Matrix: Solids (Gravimetric)
 Catalog #:
 Lot #: 3507-195
 CAS #: 335-67-1
 Solvent: NONE
 Removed: False
 Condition Recv'd: Dry Ice
 Condition Stored: Frozen
 Current Location: B1-105
 Client: Mark Ellefson
 Company Name:
 Contact:
 Phone #:
 Notes:

Date Received: 04/15/2004
 Expires: 3/29/2009 12:00:00AM
 Special:
 Flammable: Non-combustible (will not burn)
 Health: Highly toxic
 Reactive: No significant hazard
 Gross: 6.83947
 Gross Units: g
 Net Balance: 0.26279
 Net Units: g

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Exygen STUDY NO. P760
06/20/05

Aliquot - SP0004184

Addition	Component	Withdrawal	Units	Component	Purpose	Computer	Created On	Created By
0.5			g		INITIAL		4/15/2004 1:38:43PM	Edwards, Chrissy
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		0.010313	g	Balance 32	ANALYSIS	BALANCE7	11/17/2004 11:05:43AM	Sheehan, Amy
		0.10313	g	Balance 17	ANALYSIS	BALANCE8	1/19/2005 11:57:34AM	Sheehan, Amy
		0.10313	g	Balance 17	OTHER	BALANCE8	5/17/2005 11:39:07AM	Edwards, Chrissy
		0.010313	g	Balance 32	OTHER	BALANCE7	5/17/2005 12:10:42PM	Edwards, Chrissy



Purity
97 %

Date Characterized
04/07/2004

Date Expires
03/29/2009

Characterized By
Characterized by Perkin Elmer Life and
Analytical Sciences

Created By
Edwards, Chrissy

Oxygen STUDY NO. P760

06/20/05

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0089



SP0004184



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549 Albany Street
Boston, MA 02118, USA
www.perkinelmer.com

CUSTOM SERVICES

Caution: For Laboratory Use. A research chemical for research purposes only.

CERTIFICATE OF ANALYSIS

Compound Name: Perfluorooctanoic acid, [1,2-¹³C]
Lot Number: 3507-195
Molecular Formula: C₈HF₁₅O₂
Molecular Weight: 416
Physical Form: Solid
Identification: Compared with unlabeled standard by Mass Spectrum
Chemical Purity: >97% by Mass Spectrometry

Oxygen STUDY NO. P760
kg 11/17/04

Issued by: James Lacy
Manager

Date: 4/7/04

Exact Copy of Original
CML Initial 12 Apr 10 04 Date

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SP0004184

Exact Copy of Original

Initial Date 12/02/04

PerkinElmer
Life and Analytical Sciences.

Mass Spectrometry Laboratory, Boston, MA 02118

Thursday, February 19, 2004 12:14:42 PM

To: Kelley, Terence Lab/Dept: 030 Location: 575-5 Order#: 216289

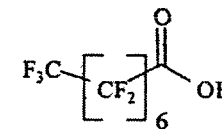
Compound Name Perfluorooctanoic acid, [1,2-13C]-

Formula: C8 H F15 O2 Molec. Wt: 416 Label: Unlabeled Conc: unknown

Catalog#: cus51073000mg Lot#: cold standard M.S. Mode: -ve APCI Infusion / Finnigan LCO Deca

Submitted on: 02/17/2004 Analyzed on: 02/21/2004 Solvent: Hexane MassSpec ID: 59

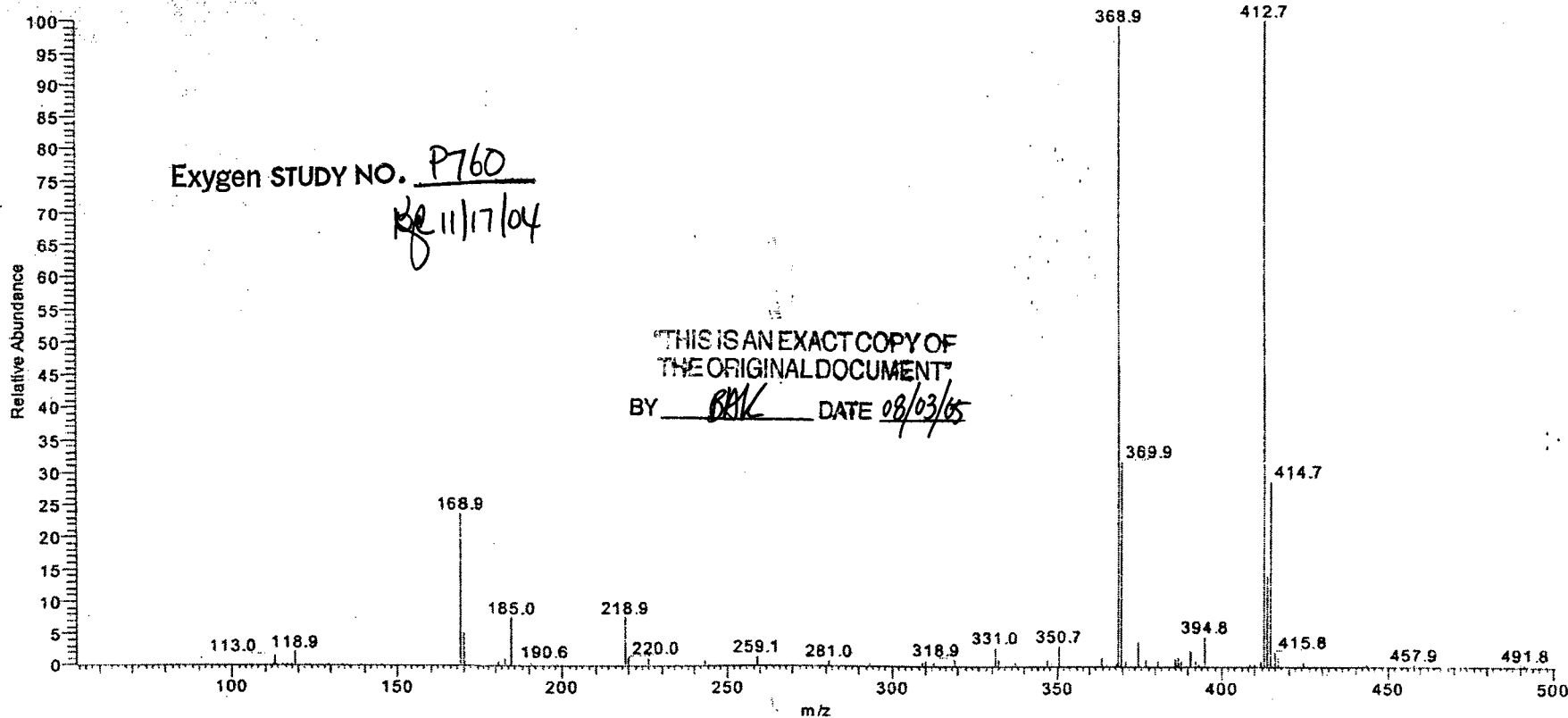
Notes / Results: ID is OK.



D:\Data\cus51073_040219114611 02/19/2004 11:46:11 AM APCI infusion in hexane

Perfluorooctanoic acid, [std]-3507-195

cus51073_040219114611 #47-101 RT: 0.76-1.65 AV: 55 NL: 3.13E4
T: -c Full ms [50.00-1000.00]



0091

SP0004184

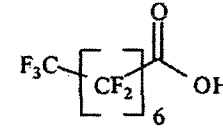
Exact Copy of Original
CWC 12 April 04
Initial Date

PerkinElmer
Life and Analytical Sciences.

Mass Spectrometry Laboratory, Boston, MA 02118

Thursday, February 19, 2004 12:14:42 PM

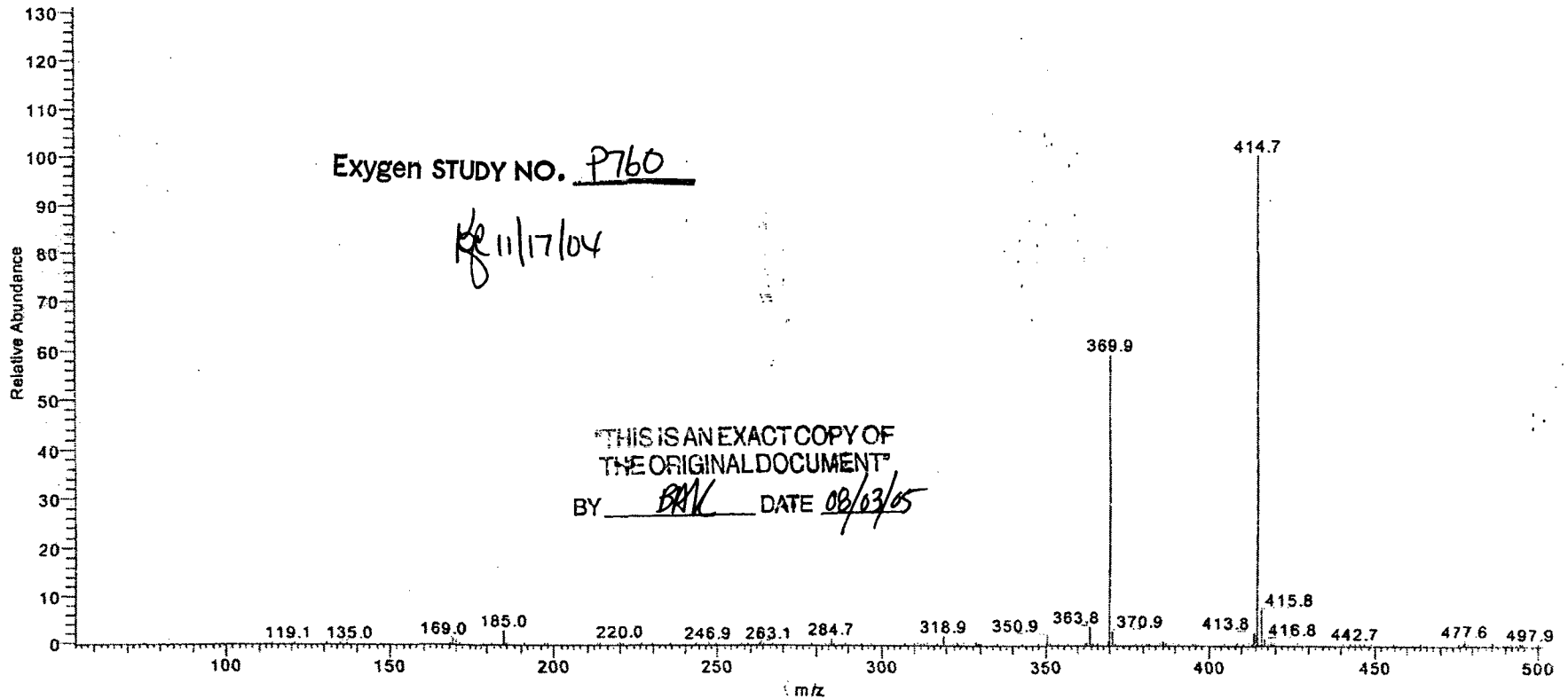
To	Kelley, Terence	Lab/Dept	030	Location	575-5	Order#	216289
Compound Name Perfluorooctanoic acid, [1,2-13C]-							
Formula	C8 H F15 O2	Molec. Wt.	416	Label	13C	Conc.	unknown
Catalog#	cus51073000mg	Lot#	3507-195	M.S. Mode	-ve.APCI Infusion / Finnigan LCQ Deca		
Submitted on	02/17/2004	Analyzed on	02/21/2004	Solvent	Hexane	MassSpec ID	58
Notes / Results	ID is OK.						



D:\Data\cus51073 02/18/2004 02:28:04 PM APCI infusion in hexane

Perfluorooctanoic acid, [1,2-13C]- 3507-195

cus51073 #43-66 RT: 0.66-1.06 AV: 24 NL: 1.11E6
T: -c Full ms [50.00-1000.00]



0092

3M Environmental Technology
and Services

PO Box 33331
St. Paul, MN 55133-3331
612 778 6442

3M

April 12, 2004

Oxygen Research
3048 Research Drive
State College, PA 16801
Attention: John Flaherty

Dear Mr. Flaherty:

Enclosed is approximately 0.5 grams of TCR-744, Perfluorooctanoic Acid, [1,2-¹³C]. Also enclosed are the associated Certificate of Analysis, TCR Receipt & General Information form, MSDS, and one Chain of Custody form (#6725). Please sign and date the Chain of Custody form then return the original to my attention at the following address:

3M Environmental Lab
Building 2-3E-09
P.O. Box 33331
St. Paul, MN 55133

If you have any questions or comments regarding this standard transfer, please contact me on:
(651) 778-6343

Sincerely,

Cindy M. Carlson

Cindy M. Carlson
Analyst

enclosure

Oxygen STUDY NO. P760

kg 11/17/04

"THIS IS AN EXACT COPY OF
THE ORIGINAL DOCUMENT"
BY BRR DATE 08/03/05

SP0004184

0093

3M Environmental Laboratory

Form 56778 - PWO

Shipping Address:
3M Bldg. 2-3E-09
935 Bush Avenue
St. Paul, MN 55108

Telephone:
Sample Receiving: (651) 778-4948
Alternate: (651) 778-4753
FAX: (651) 778-8176

Chain of Custody / Request for Laboratory Analytical

6725

SP0001

Project ID/Project Name	Validation of Method 231.2
Template #	Final Report Due Date
Project Lead	Mark Ellefson
Internal Due Date	
Dept. # (main)	832202
Class/Job/Project #	EXP-023-087

3M
E03-0472

Report Results	Contact Name	Mark Ellefson	Date Available	
	Company	3M	Date Due	
	Mailing Address	PO Box 33331	Contract Lab	
	City, State, Zip	St Paul, MN, 55133		
	Telephone #	651-778-6176	FAX #	651-778-6176

Special Instructions and/or Specific Regulatory Requirements:
(method, limit of detection, reporting units, etc.)

Analysis Requested:
Complete below. Attach any associated information.

Preservatives:					Total Number of Containers
HNO3	H2SO4	VOCs	None	Other	

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/Media	Enter the number of containers of each					(Enter an 'X' in the box below to indicate request)						
1	0.5 g of TCR-744																
2																	
3																	
4																	
5																	
6																	
7																	
8																	
9																	
10																	

Collected by (print):	Collector's signature:
1. Cindy Carlson Pace	Naresh Kumar Sengupta
11:35am 4/12/04	1100 4/15/04

Sample Condition Upon Receipt: Acceptable Other:

Temperature: °C Received on Ice

Other Associated CoCs: _____ Copies to: _____

Comments: Oxygen STUDY NO. P760

3M ENVIRONMENTAL LAB
935 BUSH AVE BLDG 2-3E-09
ST. PAUL, MN 55106

EXYGEN RESEARCH
3117 RESEARCH DRIVE
STATE COLLEGE, PA 16801
ATTN: SAMPLE CHECK-IN/JOHN FLAHERTY
814-231-8032

US DOT: THIS PACKAGE
CONFORMS TO 49CFR 173.4

34-7027-7194-9



UPS Ground



1Z 579 55E 03 1003 576 7

TRACKING NUMBER

1097 M

SHIPPING CHARGE
CORRECTED
DO NOT DUPLICATE

EXYGEN STUDY NO. P760

kg 11/17/04

SP0004184

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BY BAK DATE 08/03/05

0095

SP0004184

TCR Receipt & General Information

TCR #	TCR-744	TCR Substance #	TCR-744
Substance trade name or reference #	PFOA [1-2 ¹³ C]		
Substance/chemical name	Perfluorooctanoic Acid, Radiolabeled	Received from:	Perkin Elmer
Lot/batch #:	3507-195	Date Received:	03/29/2004
Location of synthesis, fabrication, or derivation records:	Perkin Elmer	Amount received (wt. or vol):	2g
Molecular Formula:	C ₈ F ₁₅ [¹³ C] ₂ [¹³ C]OOH	Number/size of containers:	1-125 mL plastic container
Condition:	White crystals	Shipper:	Airborne Express
Expiration date:	03/29/2009	MSDS (y/n)	<input type="radio"/> Y <input checked="" type="radio"/> N
Purity:	>97%	Records received:	Mass Spectral Data, Correspondence
3M #:	None		
Entered by (Initials)	Date Entered	Checked By	Date
CMC	03/29/2004	MLA	04/12/2004
Std Location/Storage	Initials	Date	Checked by (Initial/Date)
F-19	CMC	03/29/2004	MLA04/12/2004
Amount of Retain	Initials	Date	Checked by (Initial/Date)
None	CMC	03/29/2004	

Comments	Initials	Date	Checked By	Date
Received on dry ice	CMC	03/29/2004	MLA	04/12/2004
Added purity information from Certificate of Analysis	CMC	04/08/2004	MLA	04/12/2004

Attachments

Certificate of Analysis from PE.

Oxygen STUDY NO. P760

04/11/04

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 BY BM/ DATE 09/03/05

Exact Copy of Original
 CMC 12 April 04
 Initial Date

SP0004184

USE LOG

PFOA [1-2 13C]
Perfluorooctanoic Acid, Radiolabeled
TCR-744

Gross Wt./Vol. Before Withdrawal Balance ID 1	Amnt. withdrawn (mass or vol) Balance ID 2	Gross wt/vol after withdrawal Balance ID 1	Purpose (enter standard number or reason for removal)	Balance ID 1	Balance ID 2	Initials	Date
14.8850g	0.5116g	14.3530g	Sent to Oxygen	900	900	CMC	04/12/2004

Oxygen STUDY NO. P760

kg 11/17/04

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THE ORIGINAL DOCUMENT
BY BMC DATE 08/03/05

Exact Copy of Original

CMC 12 April 04
Initial Date

STANDARDS

SS0006991 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1000ug/mL

Standard ID: 18,254
Solvent: METHANOL
Category: Stock

Prepared By: Edwards, Chrissy
Prepared On: 5/17/2005 9:37:26AM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SP0002694	Heptadecafluorooctanesulfonic acid (PFOS)	0.10654	g
SP0003800	Pentadecafluorooctanoic acid	0.10242	g
SP0005726	Perfluorobutanesulfonate (TCR-99030-023)	0.11692	g
SP0004184	Perfluorooctanoic Acid, Radiolabeled	0.10313	g
SP0002401	PFHS	0.11136	g

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	1000.3600	µg/mL
Perfluorobutane Sulfonate	1000.1300	µg/mL
Perfluorohexane Sulfonate	1000.2400	µg/mL
Perfluorooctane Sulfonate	999.9840	µg/mL
Perfluorooctanoic Acid	1000.0300	µg/mL

Exygen STUDY NO. P 760

BML 08/03/05



STANDARDS

SS0006995 / 13C PFOA in Methanol 100 ug/mL

Standard ID: 18,260
Solvent: METHANOL
Category: Stock

Prepared By: Edwards, Chrissy
Prepared On: 5/17/2005 12:11:32PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 11:56:18AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SP0004184	Perfluorooctanoic Acid, Radiolabeled	0.01031	g

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	100.0360	µg/mL

Exygen STUDY NO. P760

BAK 08/03/05



STANDARDS

SS0006996 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 100 ug/mL

Standard ID: 18,261
Solvent: METHANOL
Category: Fortification

Prepared By: Edwards, Chrissy
Prepared On: 5/17/2005 12:44:26PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

<u>Preparation</u>			
<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006991	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1000ug/mL	10.00000	mL

<u>Final Concentrations</u>		
<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	100.0360	µg/mL
Perfluorobutane Sulfonate	100.0130	µg/mL
Perfluorohexane Sulfonate	100.0240	µg/mL
Perfluorooctane Sulfonate	99.9984	µg/mL
Perfluorooctanoic Acid	100.0030	µg/mL

Exygen STUDY NO. P760
BANK 08/03/05



STANDARDS

SS0006997 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 10 ug/mL

Standard ID: 18,262
Solvent: METHANOL
Category: Fortification

Prepared By: Edwards, Chrissy
Prepared On: 5/17/2005 12:48:24PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006996	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 100 ug/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acic	10.0036	µg/mL
Perfluorobutane Sulfonate	10.0013	µg/mL
Perfluorohexane Sulfonate	10.0024	µg/mL
Perfluorooctane Sulfonate	9.9998	µg/mL
Perfluorooctanoic Acid	10.0003	µg/mL

Exygen STUDY NO. P760
BRK 08/03/05



STANDARDS

SS0006998 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1 ug/mL

Standard ID: 18,263
Solvent: METHANOL
Category: Fortification

Prepared By: Edwards, Chrissy
Prepared On: 5/17/2005 12:48:37PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006997	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 10 ug/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	1.0004	µg/mL
Perfluorobutane Sulfonate	1.0001	µg/mL
Perfluorohexane Sulfonate	1.0002	µg/mL
Perfluorooctane Sulfonate	1.0000	µg/mL
Perfluorooctanoic Acid	1.0000	µg/mL

Exygen STUDY NO. P760

BAK 08/03/05



0102

STANDARDS

SS0007273 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 50 ng/mL

Standard ID: 18,605
Solvent: METHANOL
Category: Calibration

Prepared By: Edwards, Chrissy
Prepared On: 6/10/2005 9:15:50AM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 ng/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006998	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1 ug/mL	5.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	50.0180	ng/mL
Perfluorobutane Sulfonate	50.0065	ng/mL
Perfluorohexane Sulfonate	50.0120	ng/mL
Perfluorooctane Sulfonate	49.9992	ng/mL
Perfluorooctanoic Acid	50.0015	ng/mL

Exygen STUDY NO. P760
BAK 08/03/05



STANDARDS

SS0007274 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 25 ng/mL

Standard ID: 18,606
Solvent: METHANOL
Category: Calibration

Prepared By: Edwards, Chrissy
Prepared On: 6/10/2005 9:17:56AM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 ng/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006998	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1 ug/mL	2.50000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	25.0090	ng/mL
Perfluorobutane Sulfonate	25.0033	ng/mL
Perfluorohexane Sulfonate	25.0060	ng/mL
Perfluorooctane Sulfonate	24.9996	ng/mL
Perfluorooctanoic Acid	25.0008	ng/mL

Exygen STUDY NO. P760

BRK 08/03/05



STANDARDS

SS0007275 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 10 ng/mL

Standard ID: 18,607
Solvent: METHANOL
Category: Calibration

Prepared By: Edwards, Chrissy
Prepared On: 6/10/2005 9:19:18AM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 ng/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006998	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1 ug/mL	1.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	10.0036	ng/mL
Perfluorobutane Sulfonate	10.0013	ng/mL
Perfluorohexane Sulfonate	10.0024	ng/mL
Perfluorooctane Sulfonate	9.9998	ng/mL
Perfluorooctanoic Acid	10.0003	ng/mL

Exygen STUDY NO. P760

BRK 08/03/05

0105



STANDARDS

SS0007276 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 5.0 ng/mL

Standard ID: 18,608
Solvent: METHANOL
Category: Calibration

Prepared By: Edwards, Chrissy
Prepared On: 6/10/2005 9:20:12AM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 ng/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0007273	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 50 ng/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	5.0018	ng/mL
Perfluorobutane Sulfonate	5.0006	ng/mL
Perfluorohexane Sulfonate	5.0012	ng/mL
Perfluorooctane Sulfonate	4.9999	ng/mL
Perfluorooctanoic Acid	5.0001	ng/mL

Exygen STUDY NO. P760
BRK 08/03/05

0106



STANDARDS

SS0007277 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 2.5 ng/mL

Standard ID: 18,609
Solvent: METHANOL
Category: Calibration

Prepared By: Edwards, Chrissy
Prepared On: 6/10/2005 9:21:21AM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 ng/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0007274	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 25 ng/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	2.5009	ng/mL
Perfluorobutane Sulfonate	2.5003	ng/mL
Perfluorohexane Sulfonate	2.5006	ng/mL
Perfluorooctane Sulfonate	2.5000	ng/mL
Perfluorooctanoic Acid	2.5001	ng/mL

Exygen STUDY NO. P760

BANK 08/03/05

0107



STANDARDS

SS0007278 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 1.0 ng/mL

Standard ID: 18,610
Solvent: METHANOL
Category: Calibration

Prepared By: Edwards, Chrissy
Prepared On: 6/10/2005 9:21:55AM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 ng/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0007275	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 10 ng/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	1.0004	ng/mL
Perfluorobutane Sulfonate	1.0001	ng/mL
Perfluorohexane Sulfonate	1.0002	ng/mL
Perfluorooctane Sulfonate	1.0000	ng/mL
Perfluorooctanoic Acid	1.0000	ng/mL

Exygen STUDY NO. P760

BML 08/03/05

0108



STANDARDS

SS0007279 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 0.5 ng/mL

Standard ID: 18,611
Solvent: METHANOL
Category: Calibration

Prepared By: Edwards, Chrissy
Prepared On: 6/10/2005 9:22:28AM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 ng/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0007276	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in MeOH 5.0 ng/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	0.5002	ng/mL
Perfluorobutane Sulfonate	0.5001	ng/mL
Perfluorohexane Sulfonate	0.5001	ng/mL
Perfluorooctane Sulfonate	0.5000	ng/mL
Perfluorooctanoic Acid	0.5000	ng/mL

Exygen STUDY NO. P760
BAL 08/03/05

0109



STANDARDS

SS0007403 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1 ug/mL

Standard ID: 18,763
Solvent: METHANOL
Category: Fortification

Prepared By: Edwards, Chrissy
Prepared On: 6/22/2005 1:20:05PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006997	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 10 ug/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	1.0004	µg/mL
Perfluorobutane Sulfonate	1.0001	µg/mL
Perfluorohexane Sulfonate	1.0002	µg/mL
Perfluorooctane Sulfonate	1.0000	µg/mL
Perfluorooctanoic Acid	1.0000	µg/mL

Exygen STUDY NO. P760

BANK 08/03/05

0110



STANDARDS

SS0007404 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 0.1 ug/mL

Standard ID: 18,764	Prepared By: Edwards, Chrissy
Solvent: METHANOL	Prepared On: 6/22/2005 1:29:42PM
Category: Fortification	Condition Stored: Refrigerated
	Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0007403	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1 ug/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	0.1000	µg/mL
Perfluorobutane Sulfonate	0.1000	µg/mL
Perfluorohexane Sulfonate	0.1000	µg/mL
Perfluorooctane Sulfonate	0.1000	µg/mL
Perfluorooctanoic Acid	0.1000	µg/mL

Exygen STUDY NO. P760

BAK 08/03/05

0111



STANDARDS

SS0007505 / 13C PFOA in Methanol 0.1 ug/mL

Standard ID: 18,883
Solvent: METHANOL
Category: Fortification

Prepared By: Kravets, Brittany
Prepared On: 7/1/2005 3:06:09PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 11:56:18AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006995	13C PFOA in Methanol ug/mL	100	0.10000 mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	0.1000	µg/mL

Exygen STUDY NO. P760
BRK 08/03/05

0112



STANDARDS

SS0007917 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 10 ug/mL

Standard ID: 19,549
Solvent: METHANOL
Category: Fortification

Prepared By: Kravets, Brittany
Prepared On: 8/4/2005 1:45:25PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0006996	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 100 ug/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	10.0036	µg/mL
Perfluorobutane Sulfonate	10.0013	µg/mL
Perfluorohexane Sulfonate	10.0024	µg/mL
Perfluorooctane Sulfonate	9.9998	µg/mL
Perfluorooctanoic Acid	10.0003	µg/mL

Exygen STUDY NO.

P760
BAK 09/20/05



STANDARDS

SS0007918 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1.0 ug/mL

Standard ID: 19,550	Prepared By: Kravets, Brittany
Solvent: METHANOL	Prepared On: 8/4/2005 1:50:26PM
Category: Fortification	Condition Stored: Refrigerated
	Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

Preparation

<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0007917	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 10 ug/mL	10.00000	mL

Final Concentrations

<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	1.0004	µg/mL
Perfluorobutane Sulfonate	1.0001	µg/mL
Perfluorohexane Sulfonate	1.0002	µg/mL
Perfluorooctane Sulfonate	1.0000	µg/mL
Perfluorooctanoic Acid	1.0000	µg/mL

Exygen STUDY NO. P760
BANK 09/20/05



STANDARDS

SS0007919 / PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 0.1 ug/mL

Standard ID: 19,551	Prepared By: Kravets, Brittany
Solvent: METHANOL	Prepared On: 8/4/2005 1:53:34PM
Category: Fortification	Condition Stored: Refrigerated
	Date Expires: 11/17/2005 9:31:46AM

Final Volume: 100 µg/mL

Preparation			
Stock	Description	Amount	Units
SS0007918	PFBS, PFHS, PFOS, PFOA, and 13C PFOA in Methanol 1.0 ug/mL	10.00000	mL

Final Concentrations		
Component	Concentration	Units
3C-Perfluorooctanoic Acid	0.1000	µg/mL
Perfluorobutane Sulfonate	0.1000	µg/mL
Perfluorohexane Sulfonate	0.1000	µg/mL
Perfluorooctane Sulfonate	0.1000	µg/mL
Perfluorooctanoic Acid	0.1000	µg/mL

Exygen STUDY NO.

P760
BAK 09/20/05

0115 Instance:

R0194325



STANDARDS

SS0007920 / 13C PFOA in Methanol 1.0 ug/mL

Standard ID: 19,552
Solvent: METHANOL
Category: Fortification

Prepared By: Kravets, Brittany
Prepared On: 8/4/2005 1:56:52PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 11:56:18AM

Final Volume: 100 µg/mL

Preparation				
<u>Stock</u>	<u>Description</u>		<u>Amount</u>	<u>Units</u>
SS0006995	13C PFOA in Methanol ug/mL	100	1.00000	mL

Final Concentrations		
<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	1.0004	µg/mL

Exygen STUDY NO. P760
BANK 09/20/05

0116



STANDARDS

SS0007921 / 13C PFOA in Methanol 0.1 ug/mL

Standard ID: 19,553
Solvent: METHANOL
Category: Fortification

Prepared By: Kravets, Brittany
Prepared On: 8/4/2005 1:59:41PM
Condition Stored: Refrigerated
Date Expires: 11/17/2005 11:56:18AM

Final Volume: 100 µg/mL

Preparation			
<u>Stock</u>	<u>Description</u>	<u>Amount</u>	<u>Units</u>
SS0007920	13C PFOA in Methanol ug/mL	1.0	10.00000 mL

Final Concentrations		
<u>Component</u>	<u>Concentration</u>	<u>Units</u>
3C-Perfluorooctanoic Acid	0.1000	µg/mL

Exygen STUDY NO: P7H0
BAK 01/20/05

0117



APPENDIX IV

SAMPLE INFORMATION

Login

Login Group: L0005367

Login #:	5478	Conform COC Sample:	False
Project:	P0000760	Conform COC:	False
Company Name:	Weston Solutions, Inc.	Conform Sample:	True
Submitted By:	Jaisimha Kesari, P.E., DEE	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	06/01/2005		
Due Date:			
Received Date:	06/01/2005		
Received By:	Robb, Shawn		
Spread Sample:			
Label:			
Exygen SD/PI:	Flaherty, John		
Project Title/Type:	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 / ROUTINE		

Login Notes:

Conform Notes: This sample (Grass) was requested by John Flaherty to be collected from our yard here at our facilities. Grass collected and logged into the Exylims by Shawn L. Robb. This sample will be used under project P0000760.

Packages / Containers

<u>Package</u>	<u>Carton</u>	<u>Date / Condition</u>	<u>Shipper / ID</u>	<u>Temp. Control/Temp.</u>	<u>Direction / Handled By</u>
306308		Received Date: 6/1/05 10:52 Package & Contents Uncompromised		None	RECEIVED Robb, Shawn

<u>Container #</u>	<u>Gross Weight</u>	<u>pH</u>	<u>Container Type</u>	<u>Preservative</u>	<u>Mfg. Lot</u>	<u>Mfg. ID</u>
C0073483	201.90 g		1 gallon ziploc bag	NONE		

Samples

<u>Sample ID</u>	<u>Container</u>	<u>Matrix</u>	<u>Fraction</u>	<u>Sample</u>	<u>Date Sampled</u>	<u>Date Due</u>
L0005367-0001	C0073483	SOLID	Vegetation	Control Grass		

Login Reviewed By: _____

Melt

Date/Time: _____

06/29/05 1430

0119



Login

Login Group: L0003823

Login #:	3933	Conform COC Sample:	True
Project:	P0000760	Conform COC:	True
Company Name:	Weston Solutions, Inc.	Conform Sample:	True
Submitted By:	Jaisimha Kesari, P.E., DEE	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	11/12/2004		
Due Date:			
Received Date:	10/23/2004		
Received By:	Ammerman, Mark		
Spread Sample:			
Label:			
Exygen SD/PI:	Flaherty, John		
Project Title/Type:	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 / ROUTINE		

Login Notes:

Conform Notes: The identifications on the chain of custody (ex DF06V01AVP00100410) does not match the identification on the containers (ex. DF06V01AVP0010041007). The identifications will be added to the chain to coincide with that of the containers. MSA 09/22/05

Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By	
P 4625		Received Date: 11/12/04 15:59 Package & Contents Uncompromised	FEDEX 8479 6714 4912	Dry Ice 0.0	RECEIVED Ammerman, Mark	
Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0049876	171.80 g		500 ml Clear Plastic Narrow	NONE		
C0049877	123.00 g		500 ml Clear Plastic Narrow	NONE		
C0049878	157.60 g		500 ml Clear Plastic Narrow	NONE		
C0049879	139.40 g		500 ml Clear Plastic Narrow	NONE		
C0049880	138.40 g		500 ml Clear Plastic Narrow	NONE		
C0049881	196.00 g		500 ml Clear Plastic Narrow	NONE		
C0049882	143.00 g		500 ml Clear Plastic Narrow	NONE		
C0049883	144.40 g		500 ml Clear Plastic Narrow	NONE		
C0049884	119.80 g		500 ml Clear Plastic Narrow	NONE		
C0049885	195.00 g		500 ml Clear Plastic Narrow	NONE		
C0049886	155.00 g		500 ml Clear Plastic Narrow	NONE		
C0049887	168.40 g		500 ml Clear Plastic Narrow	NONE		
C0049888	205.80 g		500 ml Clear Plastic Narrow	NONE		

0120



Login

Login Group: L0003823

Login #: 3933
Project: P0000760
Company Name: Weston Solutions, Inc.
Submitted By: Jaisimha Kesari, P.E., DEE
Login Type: Immediate Receipt of Samples
Started: True
Date Start: 11/12/2004
Due Date:
Received Date: 10/23/2004
Received By: Ammerman, Mark
Spread Sample:
Label:
Exygen SD/PI: Flaherty, John
Project Title/Type: 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 / ROUTINE

Conform COC Sample: True
Conform COC: True
Conform Sample: True
Conform Request: True

Login Notes:

Conform Notes: The identifications on the chain of custody (ex DF06V01AVP00100410)does not match the identification on the containers (ex. DF06V01AVP0010041007). The identifications will be added to the chain to coincide with that of the containers. MSA 09/22/05

Packages / Containers

<u>Package</u>	<u>Carton</u>	<u>Date / Condition</u>	<u>Shipper / ID</u>	<u>Temp. Control/Temp.</u>	<u>Direction / Handled By</u>	
PK0004625		Received Date: 11/12/04 15:59 Package & Contents Uncompromised	FEDEX 8479 6714 4912	Dry Ice 0.0	RECEIVED Ammerman, Mark	
<u>Container #</u>	<u>Gross Weight</u>	<u>pH</u>	<u>Container Type</u>	<u>Preservative</u>	<u>Mfg. Lot</u>	<u>Mfg. ID</u>
C0049889	130.20 g		500 ml Clear Plastic Narrow	NONE		
C0049890	193.80 g		500 ml Clear Plastic Narrow	NONE		
C0049891	144.20 g		500 ml Clear Plastic Narrow	NONE		
C0049892	170.70 g		500 ml Clear Plastic Narrow	NONE		
C0049893	182.00 g		500 ml Clear Plastic Narrow	NONE		
C0049894	205.30 g		500 ml Clear Plastic Narrow	NONE		
C0049895	149.80 g		500 ml Clear Plastic Narrow	NONE		
C0049896	124.80 g		500 ml Clear Plastic Narrow	NONE		
C0049897	170.80 g		500 ml Clear Plastic Narrow	NONE		

0121



Login

Samples

<u>Sample ID</u>	<u>Container</u>	<u>Matrix</u>	<u>Fraction</u>	<u>Sample</u>	<u>Date Sampled</u>	<u>Date Received</u>	<u>Date Due</u>
L0003823-0001	C0049876	SOLID	Vegetation	DF06V01AVP0010041007	10/07/2004	10/23/2004	
L0003823-0002	C0049877	SOLID	Vegetation	DF06V01PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0003	C0049878	SOLID	Vegetation	DF06V01RCP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0004	C0049879	SOLID	Vegetation	DF06V02AVP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0005	C0049880	SOLID	Vegetation	DF06V02PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0006	C0049881	SOLID	Vegetation	DF06V02RCP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0007	C0049882	SOLID	Vegetation	DF09V01AVP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0008	C0049883	SOLID	Vegetation	DF09V01PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0009	C0049884	SOLID	Vegetation	DF09V01RCP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0010	C0049885	SOLID	Vegetation	DF09V02RCP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0011	C0049886	SOLID	Vegetation	DF09V02PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0012	C0049887	SOLID	Vegetation	DF8bV01PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0013	C0049888	SOLID	Vegetation	DF8bV01RCP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0014	C0049889	SOLID	Vegetation	DF8bV02PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0015	C0049890	SOLID	Vegetation	DF8bV02RCP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0016	C0049891	SOLID	Vegetation	DF14V01AVP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0017	C0049892	SOLID	Vegetation	DF14V01PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0018	C0049893	SOLID	Vegetation	DF14V02AVP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0019	C0049894	SOLID	Vegetation	DF14V02PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0020	C0049895	SOLID	Vegetation	DBKGV01AVP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0021	C0049896	SOLID	Vegetation	DBKGV01PAP0010041007	10/07/2004	10/23/2004	11/12/2004
L0003823-0022	C0049897	SOLID	Vegetation	DBKGV01RCP0010041007	10/07/2004	10/23/2004	11/12/2004

0122





CHAIN OF CUSTODY/ANALYSIS REQUEST FORM

Exygen Research Sample Receiving • 3117 Research Drive • State College, PA 16801, USA
 T: 814.231.8032 • F: 814.231.1580 • exygenresearch.com

Page 1 of 3

PROJECT INFORMATION

Client (name & address):

WESTON SOLUTIONS INC
1400 WESTON Way, BLDG 5-2
WEST CHESTER, PA 19380
 Phone: 610 701 3761
 Fax: 610 701 7401
 Sampler: TF

Project Manager (Name & E-mail Address):

JAI KESARI
J.KESARI@WESTONSOLUTIONS.COM
 Project Name: DECATYR AL
 P.O. #: _____
 Quotation #: _____

Please fill out this form *completely* to ensure correct analysis and proper handling of your samples.

ANALYSES REQUESTED

PFOA							
XX							
XX							
XX							
XX							
XX							
XX							
XX							

0124

SAMPLE ANALYSIS

ExyLIMS#	Client Sample Identification	Collection Date	Collection Time	Grab	Composite	Number of Containers	Specify Matrix	Comments
	DF06 V01 AVP001 004 10 07	10/07/04	11:30	X			PLANT	
	DF06 V01 PAP001 004 10 07	10/07/04	11:30	X			PLANT	
	DF06 V01 RCP001 004 10 07	10/07/04	11:30		X		PLANT	
	DF06 V02 AVP001 004 10 07	10/07/04	12:00	X			PLANT	
	DF06 V02 PAP001 004 10 07	10/07/04	12:00	X			PLANT	
	DF06 V02 RCP001 004 10 07	10/07/04	12:00	X			PLANT	
	DF09 V01 AVP001 004 10 07	10/07/04	10:40	X			PLANT	
	DF09 V01 PAP001 004 10 07	10/07/04	10:40	X			PLANT	

LAB USE ONLY

CHAIN OF CUSTODY

Relinquished by	Date	Time
	10/20/04	17:30

Cooler ID # client Cooler Temp. (°C) 0.0

Received by	Date	Time
	10/20/04	17:30
	10/20/04	22:00

LAB USE ONLY

OTHER INFORMATION

PROJECT REQUIREMENTS

Results Deadline: _____

Laboratory Report Options:

- Sample results only
- Add case narrative
- Add quality control summary
- Add calibration summary
- Add raw data
- Other _____



CHAIN OF CUSTODY/ANALYSIS REQUEST FORM

Exygen Research Sample Receiving • 3117 Research Drive • State College, PA 16801, USA
T: 814.231.8032 • F: 814.231.1580 • exygenresearch.com

PROJECT INFORMATION

Client (name & address):

WESTON SOLUTIONS INC
1400 WESTON WAY, BLDG 5-2
WEST CHESTER PA 19380

Phone: 610 701 3761

Fax: 610 701 7401

Sampler: TF

Project Manager (Name & E-mail Address):

JAI KESARI
J.KESARI@WESTONSOLUTIONS.COM

Project Name: DECATUR, ALABAMA

P.O. #: _____

Quotation #: _____

Please fill out this form *completely* to ensure correct analysis and proper handling of your samples.

ANALYSES REQUESTED

RFOA

0.00

SAMPLE ANALYSIS

ExyLIMS#	Client Sample Identification	Collection Date	Collection Time	Grab	Composite	Number of Containers	Specify Matrix	Comments
	DF09 V01 RCP0010041007	10/27/04	1040	X			PLANT	
	DF09 V02 RCP0010041007	10/27/04	1110	X			PLANT	
	DF09 V02 PAF0010041007	10/27/04	11:10	X			PLANT	
	DF8b V01 PAF0010041007	10/27/04	1230	X			PLANT	
	DF8b V01 RCP0010041007	10/27/04	1230	X			PLANT	
	DF8b V02 PAF0010041007	10/27/04	1300	X			PLANT	
	DF8b V02 RCP0010041007	10/27/04	1300	X			PLANT	
	DF14 V01 AVP0010041007	10/27/04	1330	X			PLANT	

LAB USE ONLY

CHAIN OF CUSTODY

Cooler ID # _____ Cooler Temp. (°C) _____

Relinquished by	Date	Time
<u>[Signature]</u>	<u>10/29/04</u>	<u>1230</u>

Received by	Date	Time
<u>[Signature]</u>	<u>10/29/04</u>	<u>1730</u>
<u>[Signature]</u>	<u>10/29/04</u>	<u>2230</u>

LAB USE ONLY

PROJECT REQUIREMENTS

Results Deadline: _____

Laboratory Report Options:

- Sample results only
- Add case narrative
- Add quality control summary
- Add calibration summary
- Add raw data
- Other _____

OTHER INFORMATION

PROJECT INFORMATION

Client (name & address):
WESTON SOLUTIONS INC
1400 WESTON WAY BLDG 5-2
WEST CHESTER, PA 19380
 Phone: 610 701 3761
 Fax: 610 701 7401
 Sampler: TF

Project Manager (Name & E-mail Address):
JAI KESARI
J.KESARI@WESTONSOLUTIONS.COM
 Project Name:
DECATUR, AL
 P.O. #:
 Quotation #:

ANALYSES REQUESTED

Please fill out this form *completely* to ensure correct analysis and proper handling of your samples.

SAMPLE ANALYSIS

ExyLIMS#	Client Sample Identification	Collection Date	Collection Time	Grab	Composite	Number of Containers	Specify Matrix	Comments
	DF14 V01 PAP0010041007	10/07/04	13:30	X			PLANT	
	DF14 V02 AVP0010041007	10/07/04	14:00	X			PLANT	
	DF14 V02 PAP0010041007	10/07/04	14:00	X			PLANT	
	DBKG V01 AVP0010041007	10/07/04	14:40	X			PLANT	
	DBKG V01 PAP0010041007	10/07/04	14:30	X			PLANT	
	DBKG V01 RCP0010041007	10/07/04	14:30		X		PLANT	

PFOA

0126

LAB USE ONLY

CHAIN OF CUSTODY

Cooler ID # client Cooler Temp. (°C) 0

Relinquished by	Date	Time
<i>[Signature]</i>	10/29/04	1730

Received by	Date	Time
<i>[Signature]</i>	10/29/04	
<i>[Signature]</i>	10/29/04	2230

LAB USE ONLY

PROJECT REQUIREMENTS

Results Deadline: _____

Laboratory Report Options:

- Sample results only
- Add case narrative
- Add quality control summary
- Add calibration summary
- Add raw data
- Other _____

OTHER INFORMATION



3058 Research Drive Phone: 814-272-1039
State College, PA 16801 Fax: 814-231-1580

SAMPLE CONDITION UPON RECEIPT FORM

Protocol # NA

Exygen Study # *

Date & Time Received 10/23/14 2230

Condition of Samples dry ice - active

Temporary Storage Location walker freezer 10

Initials & Date -DHA 10/23/14

Waybill # 847967144912

* Waiting for study number.

DHA 10/23/14

FedEx Express **US Airbill**

847967144912

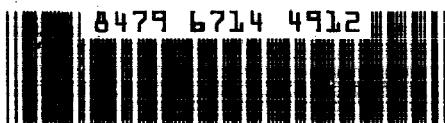
0200

FedEx Retrieval Copy

1 From
 Date: 10/20/04 Sender's FedEx Account Number: 211537680
 Sender's Name: TIM FRINAK Phone: 256 552 6187
 Company: WESTON SOLUTIONS INC % 3M
 Address: 1400 STATE DOCKS Rd
 City: DECATUR State: AL ZIP: 35601

2 Your Internal Billing Reference 02181-129-081-0001

3 To
 Recipient's Name: SAMPLE RECEIVING Phone: 814 231-8032
 Company: EXYGEN RESEARCH
 Recipient's Address: 3048 RESEARCH DR
 Address: _____
 City: STATE COLLEGE State: PA ZIP: 16801



4a Express Package Service
 1X FedEx Priority Overnight
 FedEx Standard Overnight
 FedEx 2Day
 FedEx Express Saver

4b Express Freight Service
 FedEx Freight Ground
 FedEx 2Day Freight
 FedEx 3Day Freight

5 Packaging
 FedEx Envelope
 FedEx Pak
 FedEx Box
 Other

6 Special Handling
 3 SATURDAY Delivery
 HOLD Weekday at FedEx Location
 HOLD Saturday at FedEx Location

Does this shipment include dangerous goods?
 4 Yes
 No
 Shipper's Declaration not required

7 Payment Bill to: Enter FedEx Acct. No. or Credit Card No. below.
 1X Sender Acct. No. in Section 1 will be billed.
 2 Recipient
 3 Third Party
 4 Credit Card
 5 Cash/Check

Total Packages: 1 Total Weight: _____
 Total Charges: _____

8 Sign to Authorize Delivery Without Signatures

By signing this airbill, you authorize the carrier to deliver your shipment without a signature.
 Fed. Date 11/03 • Part #158281 • ©1994-2003 FedEx • PRINTED IN U.S.A. MWIA 04

8128

467

Login

Login Group: L0003839

Login #:	3949	Conform COC Sample:	True
Project:	P0000760	Conform COC:	True
Company Name:	Weston Solutions, Inc.	Conform Sample:	True
Submitted By:	Jaisimha Kesari, P.E., DEE	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	11/16/2004		
Due Date:			
Received Date:	10/22/2004		
Received By:	Ammerman, Mark		
Spread Sample:			
Label:			
Exygen SD/PI:	Flaherty, John		
Project Title/Type:	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 / ROUTINE		

Login Notes:
Conform Notes:

Packages / Containers

<u>Package</u>	<u>Carton</u>	<u>Mail Date / Condition</u>	<u>Shipper / ID</u>	<u>Temp. Control/Temp.</u>	<u>Direction / Handled By</u>
PK0004648		11/16/2004 8:49:37AM Package & Contents Uncompromised	FEDEX 8479 6714 4912	Dry Ice 0.0	RECEIVED Ammerman, Mark

<u>Container #</u>	<u>Gross Weight</u>	<u>pH</u>	<u>Container Type</u>	<u>Preservative</u>	<u>Mfg. Lot</u>	<u>Mfg. ID</u>
C0050021	62.70 g		1 gallon ziploc bag	NONE		
C0050022	47.90 g		1 gallon ziploc bag	NONE		
C0050023	62.60 g		1 gallon ziploc bag	NONE		
C0050024	80.50 g		1 gallon ziploc bag	NONE		
C0050025	101.10 g		1 gallon ziploc bag	NONE		
C0050026	44.10 g		1 gallon ziploc bag	NONE		
C0050027	61.30 g		1 gallon ziploc bag	NONE		
C0050028	78.10 g		1 gallon ziploc bag	NONE		
C0050029	52.70 g		1 gallon ziploc bag	NONE		

0129



Samples

<u>Sample ID</u>	<u>Container</u>	<u>Matrix</u>	<u>Fraction</u>	<u>Sample</u>	<u>Date Sampled</u>	<u>Date Received</u>	<u>Date Due</u>
L0003839-0001	C0050021	SOLID	Vegetation	DF09V01SSP0010041007	10/07/2004	10/22/2004	
L0003839-0002	C0050022	SOLID	Vegetation	DF09V02UGP0010041007	10/07/2004	10/22/2004	11/16/2004
L0003839-0003	C0050023	SOLID	Vegetation	DF06V01SSP0010041007	10/07/2004	10/22/2004	11/16/2004
L0003839-0004	C0050024	SOLID	Vegetation	DF06V02SSP0010041007	10/07/2004	10/22/2004	11/16/2004
L0003839-0005	C0050025	SOLID	Vegetation	DF8bV01UGP0010041007	10/07/2004	10/22/2004	11/16/2004
L0003839-0006	C0050026	SOLID	Vegetation	DF8bV02UGP0010041007	10/07/2004	10/22/2004	11/16/2004
L0003839-0007	C0050027	SOLID	Vegetation	DF14V01SSP0010041007	10/07/2004	10/22/2004	11/16/2004
L0003839-0008	C0050028	SOLID	Vegetation	DF14V02SSP0010041007	10/07/2004	10/22/2004	11/16/2004
L0003839-0009	C0050029	SOLID	Vegetation	DBKGV01SSP0010041007	10/07/2004	10/22/2004	11/16/2004



PROJECT INFORMATION

Client (name & address):

WESTON SOLUTIONS INC
1400 WESTON WAY BLDG 5-2
WEST CHESTER, PA 19380

Phone: 610 701 3761

Fax: 610 701 7401

Sampler: TF

Project Manager (Name & E-mail Address):

JAI KESARI
J.KESARI@WESTONSOLUTIONS.COM

Project Name:

DECATUR, AL

P.O. #:

Quotation #:

Please fill out this form *completely* to ensure correct analysis and proper handling of your samples.

ANALYSES REQUESTED

0131

SAMPLE ANALYSIS

ExyLIMS#	Client Sample Identification	Collection Date	Collection Time	Grab	Composite	Number of Containers	Specify Matrix	Comments
	DF09V0155Pool10041007	10/27/04	10:40	X			BIO	
	DF09V02UGPool10041007	10/27/04	11:10	X			BIO	
	DF06V0155Pool10041007	10/27/04	11:30	X			BIO	
	DF06V0255Pool10041007	10/27/04	12:00	X			BIO	
	DF86V01UGPool10041007	10/27/04	12:30	X			BIO	
	DF86V02UGPool10041007	10/27/04	13:00	X			BIO	

PFOA
X
X
X
X
X

LAB USE ONLY

CHAIN OF CUSTODY

Relinquished by	Date	Time
<u>[Signature]</u>	<u>10/29/04</u>	<u>1730</u>

Cooler ID # 1111 Cooler Temp. (°C) 00

Received by	Date	Time
<u>[Signature]</u>	<u>10/29/04</u>	<u>1730</u>
<u>[Signature]</u>	<u>10/29/04</u>	<u>2270</u>

LAB USE ONLY

PROJECT REQUIREMENTS

Results Deadline: _____

Laboratory Report Options:

- Sample results only
- Add case narrative
- Add quality control summary
- Add calibration summary
- Add raw data
- Other _____

OTHER INFORMATION

Hold For Further Instruction.

PROJECT INFORMATION

Client (name & address):

WESTON SOLUTIONS INC
1400 WESTON Way Bldg 5-2
WEST CHESTER PA 19380

Phone: 610 701 3761

Fax: 610 701 7401

Sampler: TF

Project Manager (Name & E-mail Address):

JAI KESARI
J.KESARI@WESTONSOLUTIONS.COM

Project Name:

DECATUR, AL

P.O. #: _____

Quotation #: _____

Please fill out this form *completely* to ensure correct analysis and proper handling of your samples.

ANALYSES REQUESTED

0139

XXX PFOA

SAMPLE ANALYSIS

ExyLIMS#	Client Sample Identification	Collection Date	Collection Time	Grab	Composite	Number of Containers	Specify Matrix	Comments						
	DF14V01SSP0010041007	10/07/04	1330	X			Bio							
	DF14V02SSP0010041007	10/07/04	1400	X			BIO							
	DBKGV01SSP0010041007	10/07/04	1430	X			BIO							

LAB USE ONLY

CHAIN OF CUSTODY

Relinquished by	Date	Time
<u>T. J. Kesari</u>	<u>10/20/04</u>	<u>1730</u>

Cooler ID # clant Cooler Temp. (°C) 0.0

Received by	Date	Time
<u>Fred</u>	<u>10/20/04</u>	<u>1730</u>
	<u>10/23/04</u>	<u>2230</u>

LAB USE ONLY

PROJECT REQUIREMENTS

Results Deadline: _____

Laboratory Report Options:

- Sample results only
- Add case narrative
- Add quality control summary
- Add calibration summary
- Add raw data
- Other _____

OTHER INFORMATION

HO FOR FURTHER INSTRUCTIONS



3058 Research Drive Phone: 814-272-1039
State College, PA 16801 Fax: 814-231-1580

SAMPLE CONDITION UPON RECEIPT FORM

Protocol # NA

Exygen Study # *

Date & Time Received 10/23/14 2230

Condition of Samples dry ice - active

Temporary Storage Location walkin freezer 10

Initials & Date -MVA 10/23/14

Waybill # 84796714 4912

** waiting for study number.*

MVA 10/23/14

THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT

DATE 10/20/04

FedEx Express US Airbill

847967144912

0200

Form ID 616

FedEx Retrieval Copy

1 From
 Date 10/20/04 Sender's FedEx Account Number 211537680
 Sender's Name TIM FRINAK Phone 256 552 6187
 Company WESTON SOLUTIONS INC % 3M
 Address 1400 STATE DOCKS Rd
 City DECATUR State AL ZIP 35601

2 Your Internal Billing Reference 02181-129-081-0001

3 To
 Recipient's Name SAMILE RECEIVING Phone 814 231-8032
 Company EXYGEN RESEARCH
 Address 3048 RESEARCH DR
 City STATE COLLEGE State PA ZIP 16801

4a Express Package Service
 FedEx Priority Overnight Next business morning 5
 FedEx Standard Overnight Next business day 6
 FedEx 2Day Second business day 20
 FedEx Express Saver Third business day 20
FedEx Envelope rate not available. Minimum charge. One-pound rate.

4b Express Freight Service
 FedEx 1Day Freight* Next business day** 8
 FedEx 2Day Freight Second business day** 83
 FedEx 3Day Freight Third business day**
* Call for Confirmation. ** To most locations.

5 Packaging
 FedEx Envelope* 2
 FedEx Pak* 3
 FedEx Box 4
 Other
* Includes FedEx Small Pak, FedEx Large Pak, and FedEx Tube Pak.

6 Special Handling
 SATURDAY Delivery Available ONLY for FedEx Priority Overnight, FedEx 2Day, FedEx 1Day Freight, and FedEx 2Day Freight to select ZIP codes 3
 HOLD Weekday at FedEx Location Pick up to 16:00. FedEx Priority Overnight, FedEx 2Day, FedEx 1Day Freight, and FedEx 2Day Freight to select ZIP codes 1
 HOLD Saturday at FedEx Location Available ONLY for FedEx Priority Overnight and FedEx Priority 2Day to select ZIP codes 31

Does this shipment contain dangerous goods? One box must be checked.
 No 4
 Yes Apses attached Shipper's Use Only
 Yes Suspended Distribution Shipper's Use Only
 Dry Ice

7 Payment Bill to: Enter FedEx Acct No. or Credit Card No. below
 Sender Acct No. in Section 1 to be billed 2
 Recipient 3
 Third Party 4
 Credit Card 5
 Cash On Delivery 6

FedEx Acct No. Credit Card No. Total Packages 1 Total Weight Total Charges
 Our facility is limited to \$100 unless you declare a higher value. See the FedEx Service Guide for details.

8 Sign to Authorize Delivery Without a Signature
 By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.
 FedEx Signature Required **467**



0134

SAMPLE PROCESSING RECORD

Oxygen Study Number: P0000760
 Matrix: Solid/vegetation
 Samples removed from: NA
 Date: 6/1/05 Initials: ERL

Oxygen Sample ID	Sample Weight (g)	Oxygen Sample ID	Sample Weight (g)
<u>C0073483</u>	<u>201.9</u>		
ERL 6/1/05		ERL 6/1/05	
ERL 6/1/05		ERL 6/1/05	
ERL 6/1/05		ERL 6/1/05	

Samples weighed on balance number: Balance 8
 Date: 6/1/05 Initials: ERL

Samples were processed on (Equipment/Unit #): Robot Case 1 (E-10000517)
 Date 6/1/05 Initials ERL

Processed samples were placed in: Walkin Coolr 3
 Date 6/1/05 Initials ERL

Comments:

ERL 08/10/05

SAMPLE PROCESSING RECORD

Exygen Study Number: Proco 760
Matrix: Vegetation
Samples removed from: F-10
Date: 11-16-04 Initials: GL

Exygen Sample ID	Sample Weight (g)	Exygen Sample ID	Sample Weight (g)
C0049876	171.80	C0049881	196.00
C0049877	123.00	C0049882	143.00
C0049878	157.60	C0049883	144.40
C0049879	139.40	C0049884	119.80
C0049880	138.40	C0049885	195.00

Samples weighed on balance number: Samples weighed when log in to 11-16-04

Date: 11-16-04 Initials: GL

Samples were processed on (Equipment/Unit #): Robot Coupe 1

Date 11-16-04 Initials GL

Processed samples were placed in: F-10

Date 11-16-04 Initials GL

Comments:

BAK 07/27/05

SAMPLE PROCESSING RECORD

Oxygen Study Number: P0000760
 Matrix: Vegetation
 Samples removed from: F-10
 Date: 11-16-04 Initials: ec

Oxygen Sample ID	Sample Weight (g)	Oxygen Sample ID	Sample Weight (g)
C0049886	155.00	C0049891	144.20
C0049887	168.40	C0049892	170.70
C0049888	205.80	C0049893	182.00
C0049889	130.20	C0049894	205.30
C0049890	193.80	C0049895	149.80

Samples weighed on balance number: Samples weighed when in ec 11-16-04

Date: 11-16-04 Initials: ec

Samples were processed on (Equipment/Unit #): Robot coupe 1

Date: 11-16-04 Initials: ec

Processed samples were placed in: F-10

Date: 11-16-04 Initials: ec

Comments:

BAK 07/27/05

SAMPLE PROCESSING RECORD

Exygen Study Number: Pocos 760
Matrix: Vegetation
Samples removed from: F-10
Date: 11-16-04 Initials: ec

Exygen Sample ID	Sample Weight (g)	Exygen Sample ID	Sample Weight (g)
<u>C0049896</u>	<u>124.80</u>	<u>---</u>	<u>---</u>
<u>C0049897</u>	<u>170.80</u>	<u>---</u>	<u>---</u>
<u>---</u>	<u>---</u>	<u>---</u>	<u>---</u>
<u>---</u>	<u>---</u>	<u>---</u>	<u>---</u>
<u>---</u>	<u>---</u>	<u>---</u>	<u>---</u>

Samples weighed on balance number: Samples weighed when bag in ec 11-16-04

Date: 11-16-04 Initials: ec

Samples were processed on (Equipment/Unit #): Robot coupe 1

Date: 11-16-04 Initials: ec

Processed samples were placed in: F 10

Date: 11-16-04 Initials: ec

Comments:

BAC 07/27/05

SAMPLE PROCESSING RECORD

Oxygen Study Number: Passo 760
 Matrix: Vegetation
 Samples removed from: F-10
 Date: 11-16-04 Initials: gc

Oxygen Sample ID	Sample Weight (g)	Oxygen Sample ID	Sample Weight (g)
C0050021	62.70	C0050026	44.10
C0050022	47.90	C0050027	61.30
C0050023	62.60	C0050028	78.10
C0050024	80.50	C0050029	52.70
C0050025	101.10	—	—

Samples weighed on balance number: Samples weighed when Key in cc 11-16-04
 Date: 11-16-04 Initials: gc

Samples were processed on (Equipment/Unit #): Robot Coupe 1
 Date 11-16-04 Initials gc

Processed samples were placed in: F-10
 Date 11-16-04 Initials gc

Comments:

BANK 07/27/05

APPENDIX V
CORRESPONDENCE

Karen Risha

08/15/05

From: Karen Risha
Sent: Monday, August 15, 2005 10:26 AM
To: Jai Kesari (E-mail); Kent Lindstrom (E-mail)
Cc: Charles T. Young (E-mail)
Subject: Interim Report #12-Decatur Vegetation P760 and P1131

Exygen STUDY NO. P760

Hi Jai and Kent;
Attached please find the drafts of interim report #12 (Decatur Vegetation for PFOA (P760) and the Sulfonates (P1131)).
Please let me know of any corrections you would like made.
Sincerely,
Karen



Vegetation Interim
Report #12 ...



Vegetation Interim
Report #12 ...

Karen Risha
LC/MS Scientist and PI
Exygen Research
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State College, PA 16801
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E: karen.risha@exygen.com
exygen.com

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From: Karen Risha
Sent: Monday, September 19, 2005 2:08 PM
To: Brittany Kravets
Subject: FW: DRAFT WESTON Comments on Draft Interim Report #12 - Analysis of Vegetation Samples (P760)

-----Original Message-----

From: krlindstrom@mmm.com [mailto:krlindstrom@mmm.com]
Sent: Monday, September 19, 2005 1:04 PM
To: Karen Risha; John Flaherty
Cc: Kesari, Jai; Charles.Young@WestonSolutions.com; wkreagen@mmm.com; gahohenstein@mmm.com
Subject: Fw: DRAFT WESTON Comments on Draft Interim Report #12 - Analysis of Vegetation Samples (P760)

Karen,

Attached is a series of emails between 3M and Weston. Please review the this entire email while updating the P0000760 Vegetation Report. Note within this email that Weston comments from 09/08/05, 3M comments on 09/14/05, and comments in the immediate text of this email (09/19/05) each require changes. After you make these requested changes, please go ahead and finalize the report. However, please forward an electronic copy of the final report to Weston while it is concurrently being prepared for signatures. This will allow Weston the opportunity to quickly review the report modifications without slowing down the process of delivering a final report to Weston on or before Sept 26, 2005.

This part of this email will address the assessed accuracy of the data. Based on discussions involving Jai and 3M lab management, Weston has determined that individual samples results that have an assessed accuracy of less than +/- 60% cannot be reported due to quality control failures. The report must simply state the results as NR, where NR is defined as Not Reported due to quality control result failures. It will be up to the Study Director to evaluate the completeness of the data set with these NR results to determine if additional work will be required. That decision will be communicated to you by Weston.

Based on the Matrix Spike data presented in Table II, the following sample accuracy assessments are to be reported. This should be reported by adding a column to Table I to present the information.

C0049876 (+/- 60%), C0049878 (+/- 30%), C0049879 (+/- 50%), C0049881 (+/- 60%), C0049882 (+/- 50%), C0049883 (+/- 50%), C0049884 (+/- 30%), C0049885 (+/- 30%), C0049888 (+/- 30%), C0049890 (+/- 50%), C0049891 (+/- 30%), C0049892 (+/- 60%), C0049893 (+/- 40%), C0049894 (+/- 40%), C0049895 (+/- 40%), C0049896 (+/- 30%), C0049897 (+/- 30%), C0050021 (+/- 50%), C0050022 (+/- 30%), C0050023 (+/- 30%), C0050026 (+/- 60%), C0050029 (+/- 50%).

The following samples were processed with matrix spike concentrations (per the protocol), but these matrix spikes levels were too low relative to endogenous levels to be used for evaluating the quantitiveness of results. In these cases, the 13C PFOA surrogate spike that was in the range of sample results and at a concentration of at least 10% relative to the measured endogenous PFOA was evaluated. Based on this approach, the following sample accuracy assessments are to be reported:

C0049880 (+/- 40%), C0049886 (+/- 40%), C0049887 (+/- 60%), C0049889 (+/- 60%), C0050025 (+/- 50%)

Four samples out of 31 cannot be reported from this data set due to quality control failures. After these samples are changed to NR, the summary statistics in the Tables and the text must be updated. These samples include the following:

C0049877, C0050024, C0050027, C0050028.

Kent R. Lindstrom
Senior Research Chemist
3M Environmental, Health, and Safety Operations Building 02-3E-09
935 Bush Avenue
St. Paul, MN 55106
Tel: (651) 778-5352
Fax: (651) 778-6176
krlindstrom@mmm.com

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----- Forwarded by Kent R. Lindstrom/US-Corporate/3M/US on 09/19/2005 09:47 AM -----

"Young, Charles
T."
<Charles.Young@WestonSolutions.com>
om>
09/15/2005 10:31
AM

<krlindstrom@mmm.com>
<wkreagen@mmm.com>
"Kesari, Jai"
<J.Kesari@WestonSolutions.com>
Subject
RE: DRAFT WESTON Comments on Draft
Interim Report #12 - Analysis of
Vegetation Samples (P760)

Kent:

The subject vegetation data package for PFOA is one of the data packages targeted for finalization by 9/25/05 that Exygen has not yet received comments. Based on our discussion via teleconference today, the samples with assessed accuracies of +/- 30%, +/- 50% and +/- 60% need to be identified to Exygen in our combined review and direction issued to revise to NR those samples that fall outside of the +/- 60% acceptance range.

I assume that the intermediate +/- 40% assessed accuracy may be applied where appropriate for this package and others currently under review.

Charlie

-----Original Message-----

From: krlindstrom@mmm.com [mailto:krlindstrom@mmm.com]
Sent: Wednesday, September 14, 2005 2:22 PM
To: Young, Charles T.
Cc: Kesari, Jai; gahohenstein@mmm.com; wkreagen@mmm.com
Subject: Re: DRAFT WESTON Comments on Draft Interim Report #12 - Analysis of Vegetation Samples (P760)

Jai, Charlie -

At the end of this email I will detail required edits that I found, but first would like to address observations on the accuracy of the analytical results. I agree with Charlie that sample specific accuracy assessments take place. It will be relatively straight forward to evaluate the PFOA spike data to assess those results that are +/-30% or +/-50%, but the accuracy statement of results where spike data are outside of those limits will need close examination by Jai to determine the data usability compared to the data quality objectives. There certainly are methods (e.g. SOM01.1 (5/2005) EPA CLP Semivolatiles Method) where matrix spike and surrogate recoveries outside of +/-30% or +/-50% are reported, but Jai will need to make that call. This can be discussed at our scheduled 9AM CST conference call on Thurs Sept 15th.

In addition to Charlie's comments / requested changes below, here are what I consider to be needed corrections.

Section 3.0, first sentence includes "...methods entitled,". The word "methods" should not be plural.

Section 4.0 includes information about two sample shipments, both on October 23, 2004. The way that this is presented makes it look like the dates should be different or they could be combined and referenced as one shipment.

I agree with the Weston comment seeking additional information regarding the type, purpose, and results of the control samples in Section 4.0. The Weston comment below inadvertently references liver and serum rather than vegetation.

In Section 6.1.1, the last sentence includes "samples bags" where this should read "sample bags".

In Section 6.1.3, using the term "SPE tubes" suggests to me that the analytes are being extracted from the sample (retained on the packing materials) when the samples are passed through the tubes. Please clarify that these tubes are intended to remove matrix interference and not to retain the PFOA and 13C PFOA. Please also clarify that the tubes are packed at Exygen and not purchased commercially. It should also state that additional details about the packing materials are in the raw data or those additional details should be provided here. The general roles of each type of packing material might also be useful. If the "SPE" description is removed, then the "SPE" references in 6.1.4 should be updated.

In Section 6.6, the last sentence of the first paragraph switches from discussion about the curve to the quantitation of environmental samples. Please clarify the last sentence refers to the concentration determined in unknown samples rather than prepared standards.

In addition to the sample Descriptions / Exygen ID discrepancy between Tables I, II, and III that Weston noted below, the following samples show inconsistent IDs: DF06-V02-RCP001-0-041007, DF09-V02-RCP001-0-041007, DF8b-V02-RCP001-0-041007, and DF09-V02-PAP001-0-041007. Also, DF8b-V02-pap001-0-041007 should use consistent capital lettering in Table II. In Table III the sample description associated with C0049885 does not appear to be correct. Also in Table III, Spk H associated with C0059893 has an incorrect numerical ID. Please review all sample IDs for consistency between Tables I, II, and III in addition to making the corrections as noted. Sample DF06-V02-SSP001-0-041007, Table III, needs narration either as to why the surrogate was recovered at less than 5% or the results not reported at all.

Kent R. Lindstrom
Senior Research Chemist
3M Environmental, Health, and Safety Operations Building 02-3E-09
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"Young, Charles

T."

To <Charles.Young@WestonSolutions.com> "Kent Lindstrom"
<krlindstrom@mmm.com>

cc 09/08/2005 12:04 PM "Kesari, Jai"
<J.Kesari@WestonSolutions.com>

Subject DRAFT WESTON Comments on Draft
Interim Report #12 - Analysis of
Vegetation Samples (P760)

Kent: Please contact me when you have had an opportunity to discuss these.
Thanks, Charlie

WESTON has reviewed of the Draft Interim Report #12 - Analysis of Vegetation Samples (P760) for PFOA and has the following comments:

In Section 1.0 - Summary, the LOQ and LOD for PFOA in vegetation are stated as 0.5 ng/g and 0 ng/g respectively. The smallest standard amount is cited as 0.0005 ug/ml in Subsection 6.4. Does this correspond to a 0.5 ng/g LOD?

In Section 4.0 - Analytical Test Samples, the first sentence indicates that a vegetation sample used as the control was obtained from Exygen's facility. While the vegetation species and method of collection need not be included in the interim report, the report does not describe the purpose of analyzing control liver and serum samples and does not present the results of the analyses of the control tissues or matrix spikes of the control tissues other than the chromatograms shown in several of the figures. At a minimum, some explanation of the control sample analyses should be provided in Section 7.0 - Experimental Design.

In Subsection 6.2 - Preparation of Standards and Fortification Solutions, the last sentence in the first paragraph needs to end in a period.

In the Subsection 6.3 - Chromatography text, statements regarding control vegetation results should probably added for consistency with interim reports on other matrices.

In Subsection 6.4 - Instrument Sensitivity, the smallest standard amount is cited as 0.0005 ug/ml. Does this correspond to a 0.5 ng/g LOD?

In Subsection 6.6 - Quantitation and Example Calculation, equation 2 should indicate that the concentration is on a wet weight basis (i.e, PFOA found (ppb; wet weight)). The example calculation shown on should also include the weight basis in the parentheses.

In Table I, the heading for the PFOA column states the results are Dry Weight. Please revise to Wet Weight.

The Client Sample ID for the Exygen C0049890 and C0049890 Rep samples are listed as DF8b-V01-RCP001-0-041007 and DF8b-V01-RCP001-0-041007*.

The Client Sample IDs should be changed to DF8b-V02-RCP001-0-041007 and DF8b-V02-RCP001-0-041007* respectively. The correct Client Sample IDs are reflected in Tables II and III.

The footnotes to both Table I and Table II define ND as "ND = Not detected = Peak Area 0". Please revise to state the LOD as a non-zero number in concentration units of ng/g.

Finally, matrix spike and surrogate spike recoveries in Tables II and III for some vegetation samples are below the 70 - 130% range. Some are significantly lower. I will defer to Kent with respect to whether sample by sample accuracy assessment (and tabulation in Table I) may be warranted for the vegetation samples.

Charlie

Charles T. Young
Senior Technical Manager
Weston Solutions, Inc.
1400 Weston Way (4-2)
West Chester, PA 19380
(610) 701-3787
fax (610) 701-3187

APPENDIX VI

STUDY PERSONNEL LOG AND EXPLANATION OF ERROR CODING

STUDY PERSONNEL LOG

EXYGEN PROJECT#: P760 CLIENT PROJECT#: -

PRINTED NAME	SIGNATURE	INITIALS
KAREN RISHA	<i>Karen Risha</i>	KR
Christine E Edwards	<i>Christine E Edwards</i>	CEE
Amy Sheehan	<i>Amy Sheehan</i>	AMS
Paul Connolly	<i>Paul Connolly</i>	PC
Mark Armenta	<i>Mark Armenta</i>	MA
BRITTANY KRAVETS	<i>Brittany Kravets</i>	BK
James R. Miller	<i>James R. Miller</i>	JRM
Mark D. Newby	<i>Mark D. Newby</i>	MN
EDWARD KAISER	<i>Edward Kaiser</i>	EK
ED CARNS	①	①
John M Flaherty	<i>J M Flaherty</i>	JMF
Sharareh Zolghadr	<i>Sharareh Zolghadr</i>	SZ
Eric W. Edwards	<i>Eric W. Edwards</i>	EWE
Ellen Dashem	<i>Ellen Dashem</i>	EDD
Peikun Liu	<i>Peikun Liu</i>	PL
Carlyle Horrell	<i>Carlyle Horrell</i>	CH
Brian McAllister	<i>Brian McAllister</i>	BMA
Mindy Cressley	<i>Mindy Cressley</i>	MLC

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Centre Analytical Laboratories, Inc.

3048 Research Drive
Phone: (814) 231-8032

State College, PA 16801
Facsimile: (814) 231-1253

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BY BAC DATE 08/03/05

Master Signature Log

February 1993

Printed Name	Signature	Initials
① EDWARD E. CARUS	<i>Edward E. Carus</i>	EC
M. MICHAEL ARJMAND	<i>Michael Arjmand</i>	MA
Susan K. Heeman	<i>Susan K. Heeman</i>	<i>SKH</i>
Gail L. Keller	<i>Gail L. Keller</i>	GJK
GAYA WICKREMESINHE	<i>Gayawickremesinhe</i>	GW
Bijan Zolghadr	<i>Bijan</i>	BZ
Sulie Burton	<i>Sulie Burton - Petit</i>	SB JBP
Mary Ellen Beattie	<i>Mary Ellen Beattie</i>	MB
RICHARD A. GRAZINI	<i>Richard A. Grazini</i>	RAG or RG
Beatrice Marengo	<i>Beatrice Marengo</i>	BM
Jennifer L. Horney	<i>Jennifer L. Horney</i>	JLH
Michelle L. Madsen	<i>Michelle L. Madsen</i>	MLM
Philippe J. Ourisson	<i>Philippe J. Ourisson</i>	PJO
Michael L. D'Amant	<i>Michael L. D'Amant</i>	MDA
Samuel F. Stover	<i>Samuel F. Stover</i>	SFS
ROBERT J. DOOLITTLE	<i>Robert J. Doolittle</i>	RJD
JIAN-XIN HUANG	<i>Jianxin Huang</i>	JXH
Xiaoming Zhu	<i>Xiaoming Zhu</i>	X.Z
Brandi L. Grebe	<i>Brandi L. Grebe</i>	BLG
Ralph O. Mumma	<i>Ralph O. Mumma</i>	ROM
Sharon A. Webster	<i>Sharon A. Webster</i>	SW
M. ALI REZAIANI	<i>M. Ali Rezaiani</i>	AR
MITRA REZAIANI	<i>Mitra Rezaiani</i>	MR

① INCURRED FOR SIGNATURE & INITIALS OF ED CARUS. 08/06/10/05

0149

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BY RF DATE 06/10/05



3058 Research Drive
State College, PA 16801

Phone: 814-272-1039
Fax: 814-231-1580

STUDY PERSONNEL LOG

EXYGEN PROJECT#: P760 CLIENT PROJECT#: —

PRINTED NAME	SIGNATURE	INITIALS
ERIC HOOVER	(2)	(2)

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BY BAK DATE 08/03/05

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Master Signature Log

Printed Name	Signature	Initials
Katherine Siegel	Katherine Siegel	KJS
Christine Bastian	Christine Bastian	CEB
Amy Dick	Amy Dick	AED
Nancy L. Schuster	Nancy L. Schuster	NLS
Anthony G. Parzocquin	Anthony G. Parzocquin	AGP
Adam E HARRIS	Adam E Harris	AEH
Leon L. Kappler	Leon L Kappler	L L K
Eric W Edwards	Eric W Edwards	EWE / ee
Felicia L. Kemp	Felicia L Kemp	FK
Ty W. Kahler	Ty W. Kahler	TWK
Kelly A. Booker	Kelly A Booker	KAB
Elizabeth A. Hendershot	Elizabeth A. Hendershot	EAH
Deni-Jo Stanisic	Deni-Jo Stanisic	DJS
Lian-zhen Gu	Lian-zhen Gu	L Z G
Terence Hall	Terence Hall	TH
DAVID J SALASKE	David J Salaske	DJS / AS
WILLIAM CANTARA	William Cantara	WC
Stacey E Robinson	Stacey E Robinson	SER
Casey W. Hemmis	Casey W. Hemmis	CH
PAUL SWIDERSKY	Paul Swidersky	PS
DAVID DETTLER	David Dettler	DD
Sharon Lupo	Sharon A. Lupo	SL
② Eric Hoover	Eric Hoover	EH

② INCLUDED FOR SIGNATURE + INITIALS OF ERIC HOOVER - BAK 07/21/05

ERROR CORRECTION CODE LIST

Project Number: P 760

The following abbreviations may be used to explain corrections.

Code	Meaning
(cc)	Change for greater clarity
(ce)	Calculation error
(ne)	Numerical transcription error
(re)	Recording error
(sm)	Stray mark with no other feasible explanation
(sp)	Spelling error
(te)	Transcription error
(wd)	Wrong date
(wl)	Wrong location
(wo)	Inadvertent write over
(ee)	Entry not initialed and dated at time of entry
(ir)	Entry inadvertently not recorded at time of initial observation
(le)	Late entry

Printed from SOP V0000365-4.

Initials: BKK Date: 08/03/05

APPENDIX VII

LIST OF MAJOR EQUIPMENT

**List of Major Equipment
Exygen Study No P760**

Balances: Analytical, Mettler, Model AT261, Mettler Instrument Corporation, Hightstown, NJ

Laboratory Glassware: General Laboratory

LC/MS/MS Instrument: PE SCIEX API 4000 Biomolecular Mass Analyzer

HPLC System: Hewlett Packard (HP) Series 1100, Wilmington, DE

HPLC Column: Fluorphase RP, 50 mm x 2.1 mm, 5 μ , Thermo Electron Corporation

Centrifuge: Allegra 6R, Beckman

Vacuum Manifold: IST VacMaster 20, Internation Sorbent Technology, Mid Glamorgan, U.K.

SPE Columns: Waters Sep Pak Vac 6cc (1g) tC18, Waters Corporation

APPENDIX VIII

SAMPLE RAW DATA AND CHROMATOGRAPHY

Tuning Information

Note to File

Overall Summary

Vegetation Set #1: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #1, Data Set 071205D, Extracted on
07/12/05 (Analyzed on 07/14-15/05)

Vegetation Set #1, Data Set 071205DR, Extracted on
07/12/05 (Analyzed on 07/24/05)

Vegetation Set #2: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #2, Data Set 071205E, Extracted on
07/12/05 (Analyzed on 07/15/05)

Vegetation Set #2, Data Set 071205ER, Extracted on
07/12/05 (Analyzed on 07/26-27/05)

Vegetation Set #3: Internal Chain of Custody/Fortification
Sheets

APPENDIX VIII

SAMPLE RAW DATA AND CHROMATOGRAPHY

Tuning Information

Note to File

Overall Summary

Vegetation Set #1: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #1, Data Set 071205D, Extracted on
07/12/05 (Analyzed on 07/14-15/05)

Vegetation Set #1, Data Set 071205DR, Extracted on
07/12/05 (Analyzed on 07/24/05)

Vegetation Set #2: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #2, Data Set 071205E, Extracted on
07/12/05 (Analyzed on 07/15/05)

Vegetation Set #2, Data Set 071205ER, Extracted on
07/12/05 (Analyzed on 07/26-27/05)

Vegetation Set #3: Internal Chain of Custody/Fortification
Sheets

APPENDIX VIII Continued

SAMPLE RAW DATA AND CHROMATOGRAPHY

Vegetation Set #3, Data Set 071305C, Extracted on
07/13/05 (Analyzed on 07/19-20/05)

Vegetation Set #3, Data Set 071305CR, Extracted on
07/13/05 (Analyzed on 07/27-28/05)

Vegetation Set #3, Data Set 071305CR2, Extracted on
07/13/05 (Analyzed on 07/29/05)

Vegetation Set #4: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #4, Data Set 071405C, Extracted on
07/14/05 (Analyzed on 07/20-21/05)

Vegetation Set #5: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #5, Data Set 071505C, Extracted on
07/15/05 (Analyzed on 07/21/05)

Vegetation Set #6: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #6, Data Set 072805A, Extracted on
07/28/05 (Analyzed on 07/29-30/05)

APPENDIX VIII Continued

SAMPLE RAW DATA AND CHROMATOGRAPHY

Vegetation Set #6, Data Set 072805AR, Extracted on
07/28/05 (Analyzed on 08/02-03/05)

Vegetation Set #6, Data Set 072805AR2, Extracted on
07/28/05 (Analyzed on 08/08/05)

Vegetation Set #7: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #7, Data Set 072805B, Extracted on
07/28/05 (Analyzed on 07/28-29/05)

Vegetation Set #7, Data Set 072805BR, Extracted on
07/28/05 (Analyzed on 08/01-02/05)

Vegetation Set #8: Internal Chain of Custody/Fortification
Sheets

Vegetation Set #8, Data Set 080805C, Extracted on
08/08/05 (Analyzed on 08/09/05)

Vegetation Set #8, Data Set 080805CR, Extracted on
08/08/05 (Analyzed on 08/10/05)

Tuning Information

Performed By: **AMB 07/26/05**

Printing Time: 7:25:38

Printing Date: Tuesday, July 26, 2005

Unit Resolution Optimization for Quad 1, Positive

Tuesday, July 26, 2005, 07:21

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q1_FOS_PPG_03Mar03_gv.dam

Threshold: 200 cps
 Search Range: 5.0 amu
 Width At (percent max): 50.0
 Target Peak Width: 0.70
 Peak Width Tolerance: 0.100
 Max Iterations: 20

Original Resolution Table

Mass	Offset
59.050	0.030
175.000	0.087
906.673	0.417
1254.925	0.560
1545.134	0.683
2010.469	0.886

First Resolution Table

Mass	Offset
59.050	0.030
175.000	0.087
906.673	0.417
1254.925	0.560
1545.134	0.683
2010.469	0.886

Iteration 0

Mass	Offset	Found At	Intensity	Width
59.050	0.030	59.039	82326309	0.790
175.000	0.087	175.008	59210156	0.699
906.673	0.417	906.663	54896440	0.617
1254.925	0.560	1254.927	8809357	0.632
1545.134	0.683	1545.099	2045223	0.636
2010.469	0.886	2010.464	1461919	0.625

Successful resolution optimization.

New Resolution Table

Mass	Offset
59.050	0.030
175.000	0.087
906.673	0.417
1254.925	0.560
1545.134	0.683
2010.469	0.886

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
59.050	59.039	0.011	0.790	-0.090	82326309.00	1.19
175.000	175.008	-0.008	0.699	0.001	59210156.50	3.67
906.673	906.663	0.010	0.617	0.083	54896440.50	0.46
1254.925	1254.927	-0.002	0.632	0.068	8809357.56	4.02
1545.134	1545.099	0.035	0.636	0.064	2045223.88	7.63
2010.469	2010.464	0.005	0.625	0.075	1461919.15	-11.42

Original Calibration Table

Mass	Dac
59.050	1099
175.000	3310
906.673	17246
1254.925	23879
1545.134	29406
2010.469	38248

New Calibration Table

Mass	Dac
59.050	1099
175.000	3310

Acq. Date: n/a
 Exygen Research

Oxygen STUDY NO: P760

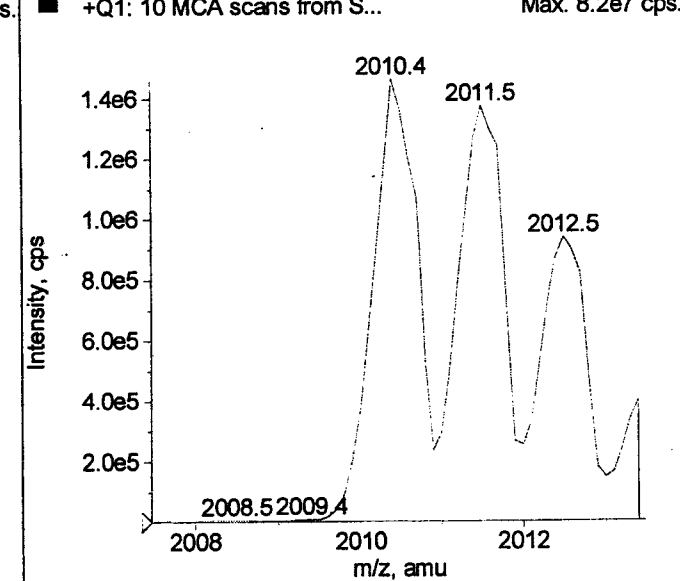
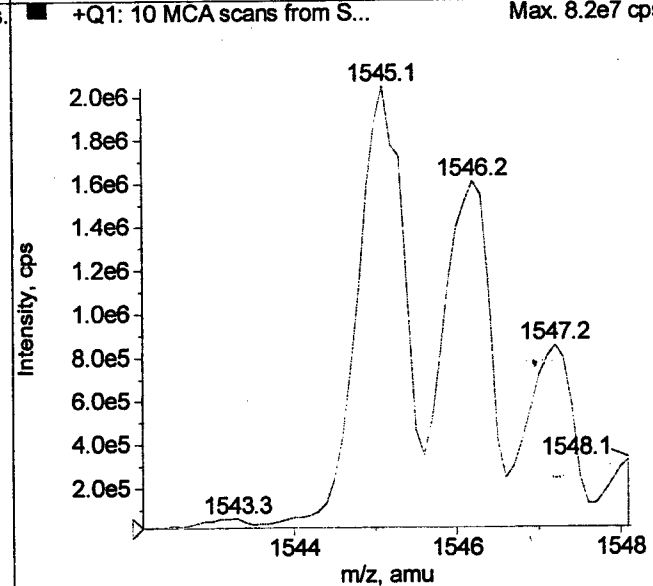
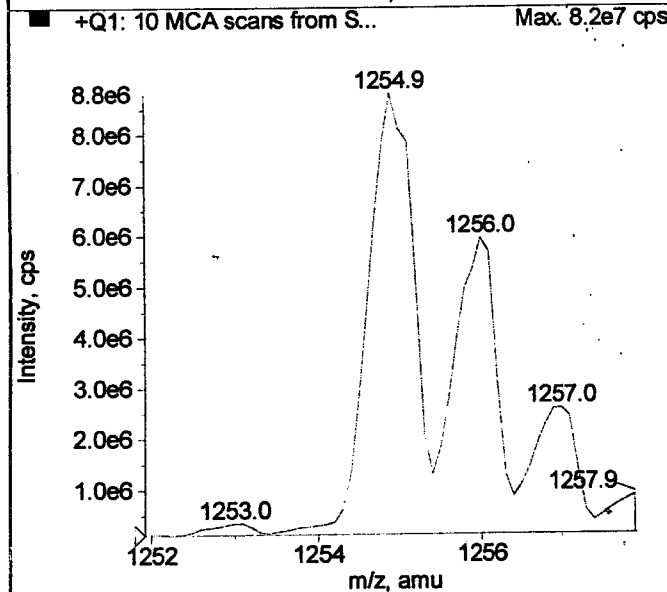
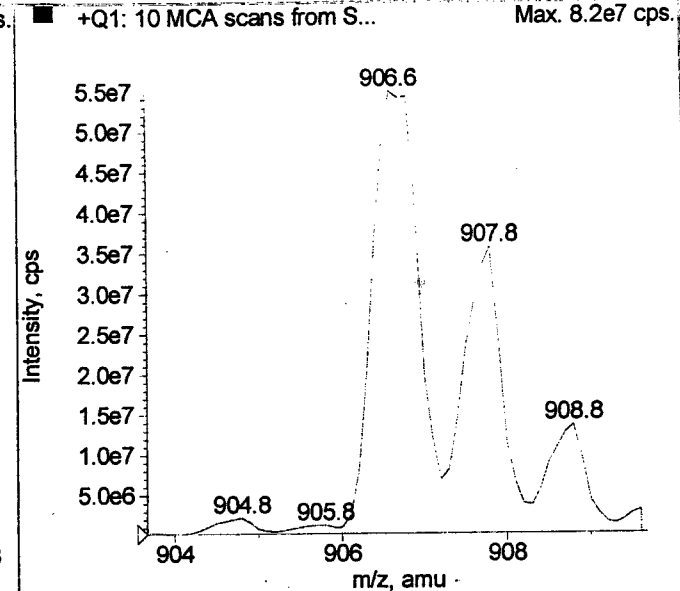
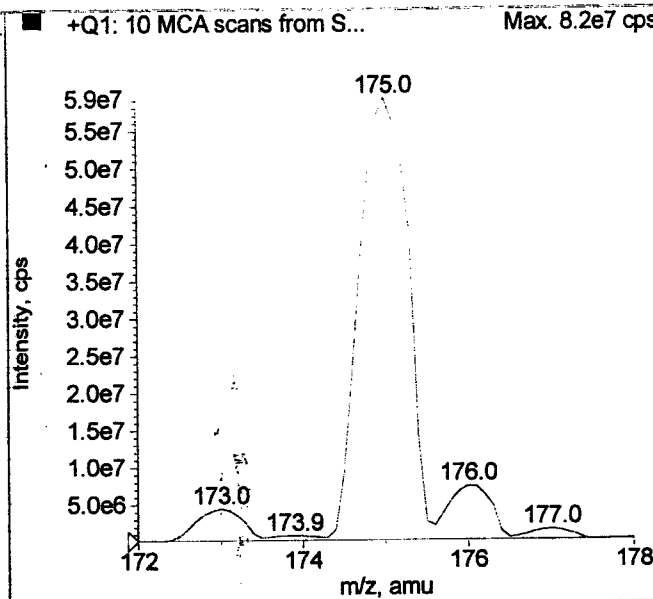
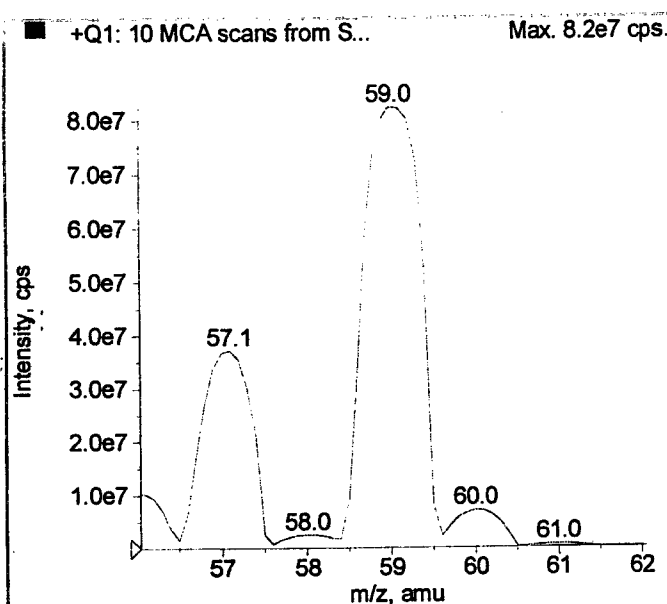
0160

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BY BAK DATE 08/08/05

5.673	17246
1254.925	23879
1545.134	29405
2010.469	38248

File path of acquired data for resolution optimization :
D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.07.26.07.21.33



Acq. Date: Tuesday, July 26, 2005
Exygen Research

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THE ORIGINAL DOCUMENT

Exygen STUDY NO. P760

BY BAK DATE 08/08/05

0169

Performed By: AMB 07/26/05

Printing Time: 7:42:42

Printing Date: Tuesday, July 26, 2005

Unit Resolution Optimization for Quad 3, Positive

Tuesday, July 26, 2005, 07:41

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q3 POS PPG 051205.dam

Threshold: 200 cps

Search Range: 5.0 amu

Width At (percent max): 50.0

Target Peak Width: 0.70

Peak Width Tolerance: 0.100

Max Iterations: 20

Original Resolution Table

Mass	Offset
59.050	0.055
175.000	0.118
906.673	0.430
1254.925	0.578
1545.134	0.698
1836.344	0.832

First Resolution Table

Mass	Offset
59.050	0.055
175.000	0.118
906.673	0.430
1254.925	0.578
1545.134	0.698
1836.344	0.832

The masses selected in the reference table do not match the masses in the acquisition method.

Iteration 0

Mass	Offset	Found At	Intensity	Width
59.050	0.055	59.066	33432064	0.633
175.000	0.118	175.007	4903586	0.626
906.673	0.430	906.651	6809328	0.646
1254.925	0.578	1254.899	2812269	0.624
1545.134	0.698	1545.124	753941	0.656
1836.344	0.832	1836.348	71549	0.691

Successful resolution optimization.

New Resolution Table

Mass	Offset
59.050	0.055
175.000	0.118
906.673	0.430
1254.925	0.578
1545.134	0.698
1836.344	0.832

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
59.050	59.066	-0.016	0.633	0.067	33432064.75	-2.34
175.000	175.007	-0.007	0.626	0.074	4903586.44	-1.58
906.673	906.651	0.022	0.646	0.054	6809328.13	1.86
1254.925	1254.899	0.026	0.624	0.076	2812269.38	2.55
1545.134	1545.124	0.010	0.656	0.044	753941.34	1.99
1836.344	1836.348	-0.004	0.691	0.009	71549.14	-8.43

Original Calibration Table

Mass	Dac
59.050	1100
175.000	3314
906.673	17261
1254.925	23900
1545.134	29432
1836.344	34981

New Calibration Table

Mass	Dac
59.050	1100

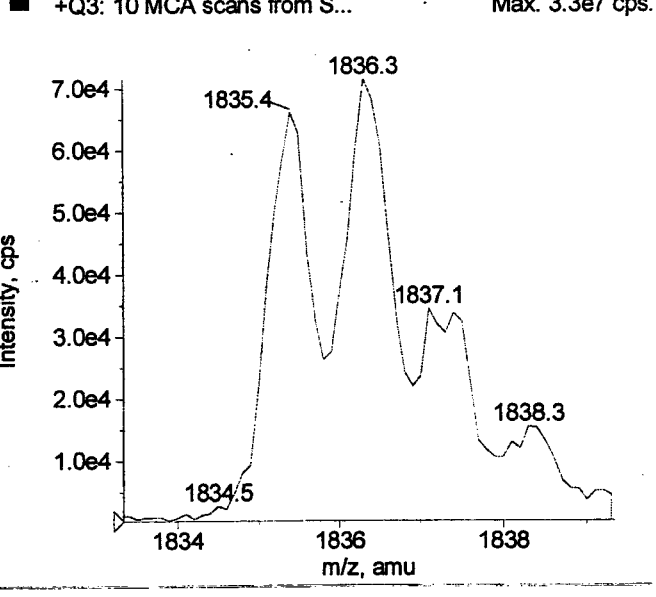
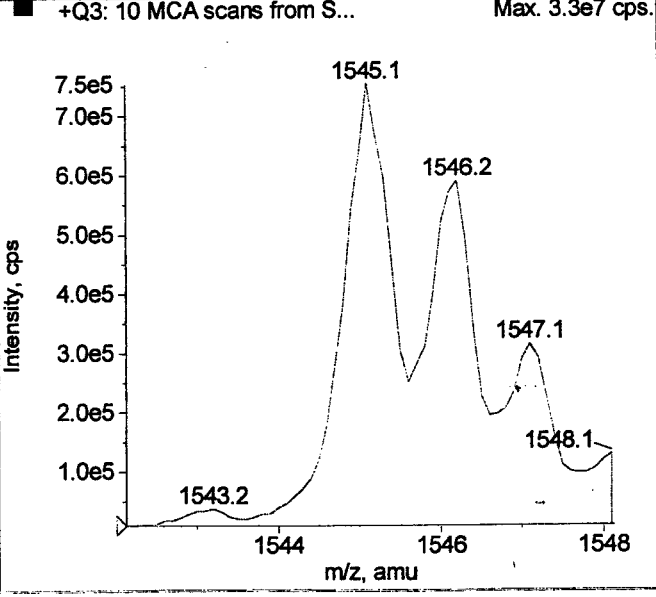
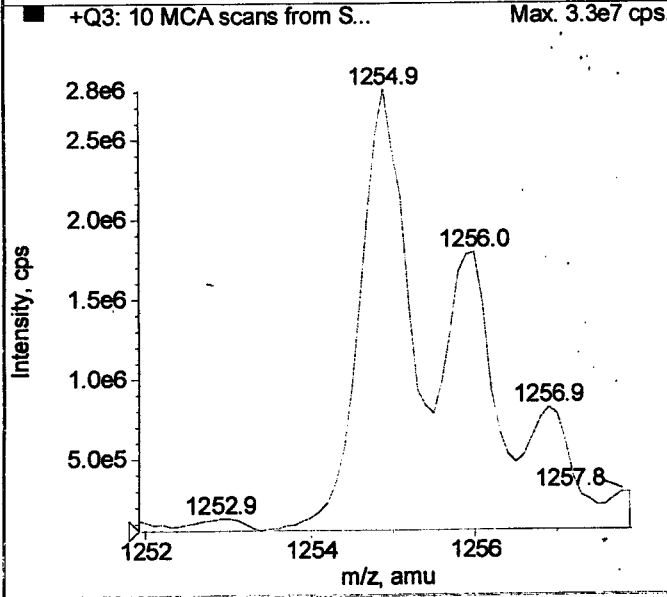
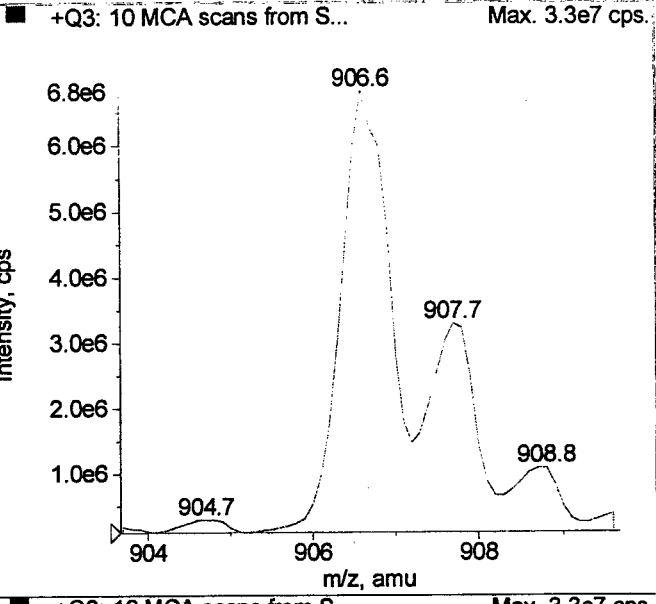
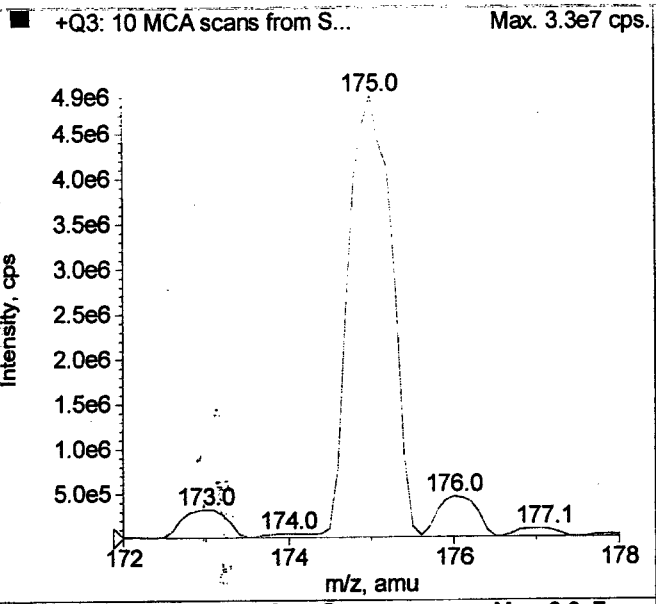
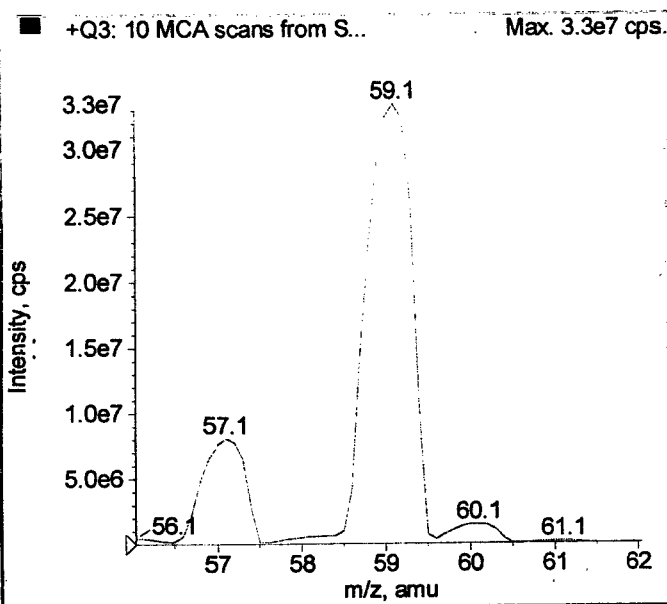
Acq. Date: n/a
Oxygen Research

Oxygen STUDY NO. P760

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BY BRV DATE 08/08/05

0.000	3314
206.673	17261
1254.925	23900
1545.134	29432
1836.344	34981

File path of acquired data for resolution optimization :
D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.07.26.07.41.42



0165

Acq. Date: Tuesday, July 26, 2005
Exygen Research

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BY BAL DATE 08/09/05

Exygen STUDY NO. P760

Resolution Optimization for Quad 1, Negative

Tuesday, July 26, 2005, 07:47

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q1 NEG PPG 081803.dam

Threshold: 200 cps
Search Range: 5.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
44.998	0.053
585.385	0.289
933.636	0.438
1165.804	0.532
1223.845	0.557
1572.097	0.706

First Resolution Table

Mass	Offset
44.998	0.053
585.385	0.289
933.636	0.438
1165.804	0.532
1223.845	0.557
1572.097	0.706

Iteration 0

Mass	Offset	Found At	Intensity	Width
44.998	0.053	44.975	81749553	0.616
585.385	0.289	585.356	3760141	0.633
933.636	0.438	933.629	16479352	0.608
1165.804	0.532	1165.786	8954152	0.655
1223.845	0.557	1223.830	6509409	0.640
1572.097	0.706	1572.134	901154	0.593

Iteration 1

Mass	Offset	Found At	Intensity	Width
44.998	0.053	44.972	81322553	0.623
585.385	0.289	585.349	3900238	0.626
933.636	0.438	933.633	15619456	0.620
1165.804	0.532	1165.800	9096959	0.664
1223.845	0.557	1223.837	7524956	0.625
1572.097	0.696	1572.079	1044566	0.816

Iteration 2

Mass	Offset	Found At	Intensity	Width
44.998	0.053	44.971	81005251	0.625
585.385	0.289	585.360	3782609	0.652
933.636	0.438	933.638	14804699	0.650
1165.804	0.532	1165.807	9226697	0.648
1223.845	0.557	1223.820	6983490	0.634
1572.097	0.701	1572.121	997395	0.682

Successful resolution optimization.

New Resolution Table

Mass	Offset
44.998	0.053
585.385	0.289
933.636	0.438
1165.804	0.532
1223.845	0.557
1572.097	0.701

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
44.998	44.971	0.027	0.625	0.075	81005251.50	-2.10
585.385	585.360	0.025	0.652	0.048	3782609.84	59.33

Performed By: AMB 07/26/05

Printing Time: 8:01:07

Printing Date: Tuesday, July 26,
2005

933.636	933.638	-0.002	0.650	0.050	14804699.75	54.81
1165.804	1165.807	-0.003	0.648	0.052	9226697.94	48.52
1223.845	1223.820	0.025	0.634	0.066	6983490.25	42.83
1572.097	1572.121	-0.024	0.682	0.018	997395.80	47.13

Original Calibration Table

Mass	Dac
44.998	833
585.385	11123
933.636	17759
1165.804	22182
1223.845	23286
1572.097	29919

New Calibration Table

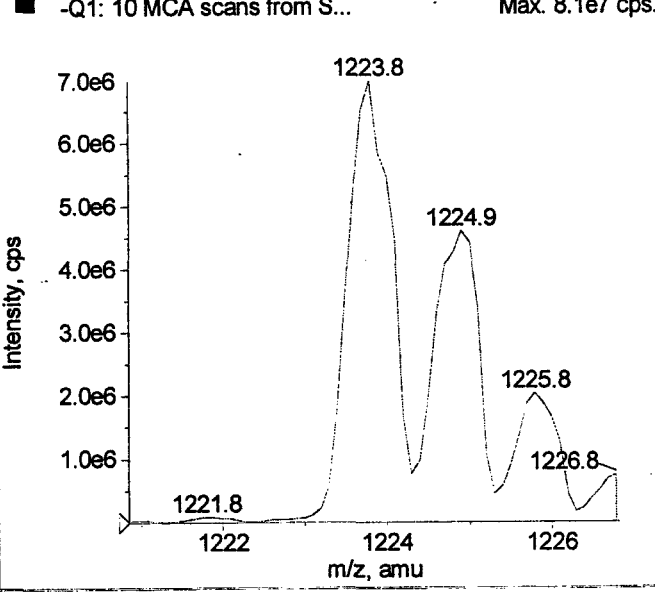
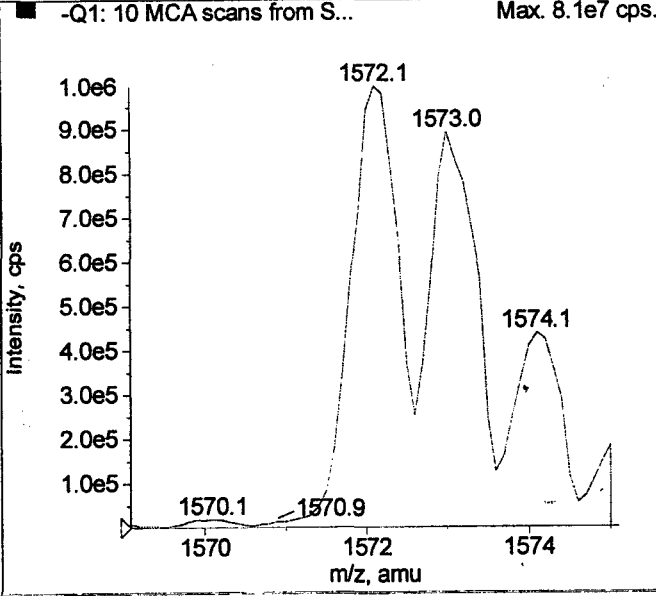
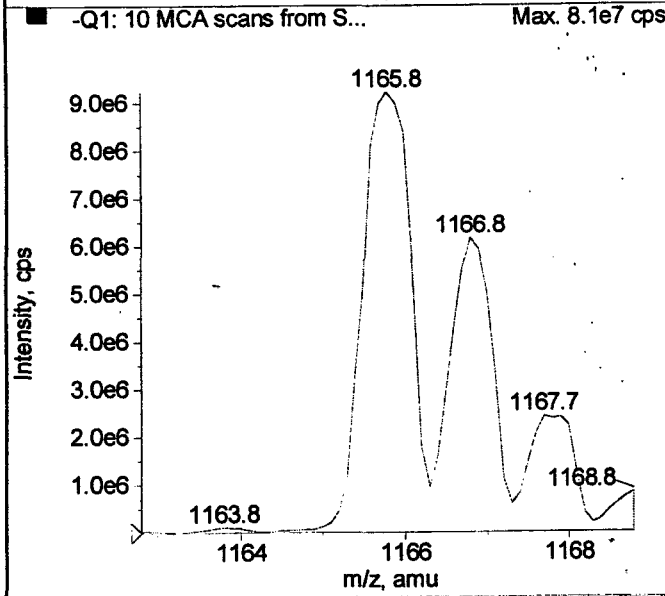
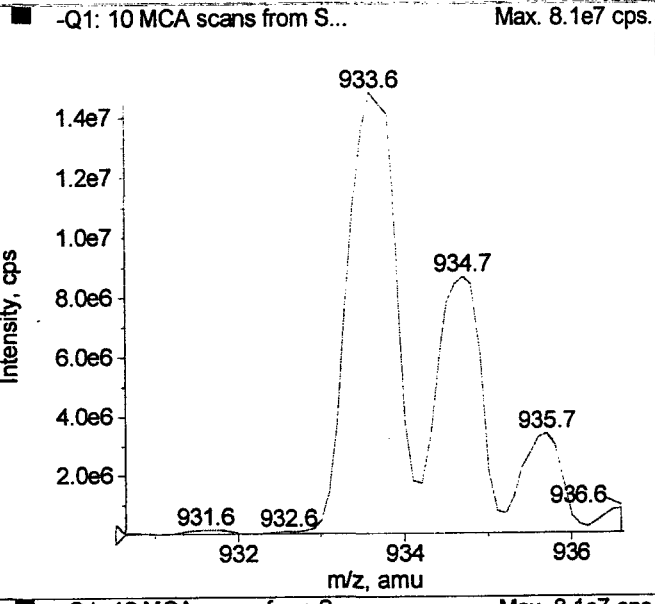
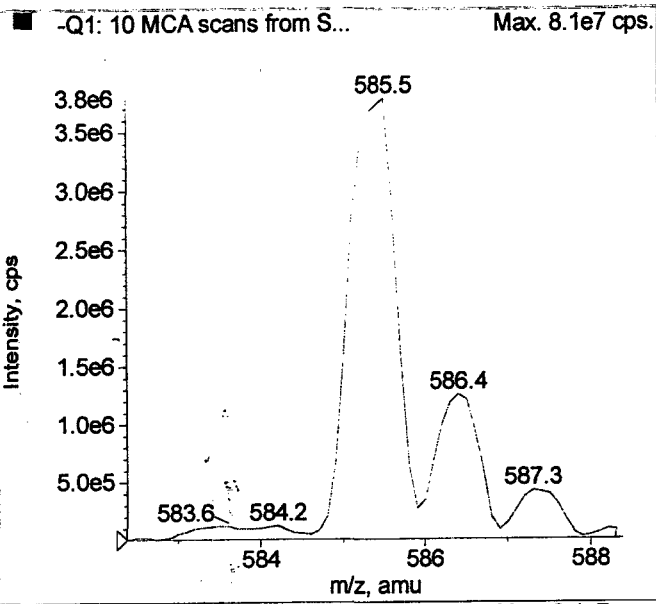
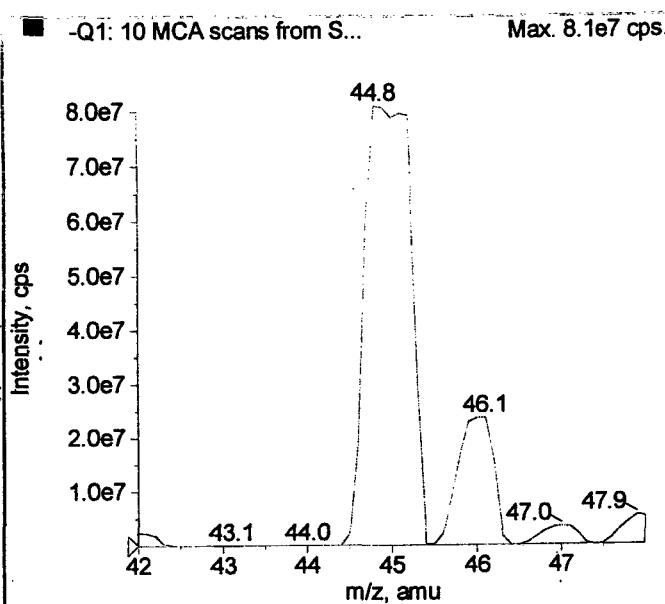
Mass	Dac
44.998	832
585.385	11123
933.636	17759
1165.804	22182
1223.845	23286
1572.097	29919

File path of acquired data for resolution optimization :

D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.07.26.07.47.33

Acq. Date: n/a
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0167



8910

Acq. Date: Tuesday, July 26, 2005
Exygen Research

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Exygen STUDY NO. P760

Resolution Optimization for Quad 3, Negative

Tuesday, July 26, 2005, 08:27

Method File:
D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q3 NEG PPG 081803.dam

Threshold: 200 cps
Search Range: 5.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
44.998	0.050
585.385	0.290
933.636	0.435
1165.804	0.537
1223.845	0.561
1572.097	0.707

First Resolution Table

Mass	Offset
44.998	0.050
585.385	0.290
933.636	0.435
1165.804	0.537
1223.845	0.561
1572.097	0.707

Iteration 0

Mass	Offset	Found At	Intensity	Width
44.998	0.050	45.005	79898509	0.677
585.385	0.290	585.368	1569936	0.679
933.636	0.435	933.608	9252174	0.666
1165.804	0.537	1165.790	4808521	0.623
1223.845	0.561	1223.841	2878910	0.620
1572.097	0.707	1572.099	488725	0.693

Successful resolution optimization.

New Resolution Table

Mass	Offset
44.998	0.050
585.385	0.290
933.636	0.435
1165.804	0.537
1223.845	0.561
1572.097	0.707

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
44.998	45.005	-0.007	0.677	0.023	79898509.00	0.71
585.385	585.368	0.017	0.679	0.021	1569936.61	-4.23
933.636	933.608	0.028	0.666	0.034	9252174.63	-3.38
1165.804	1165.790	0.014	0.623	0.077	4808521.53	2.05
1223.845	1223.841	0.004	0.620	0.080	2878910.05	4.67
1572.097	1572.099	-0.002	0.693	0.007	488725.52	-20.99

Original Calibration Table

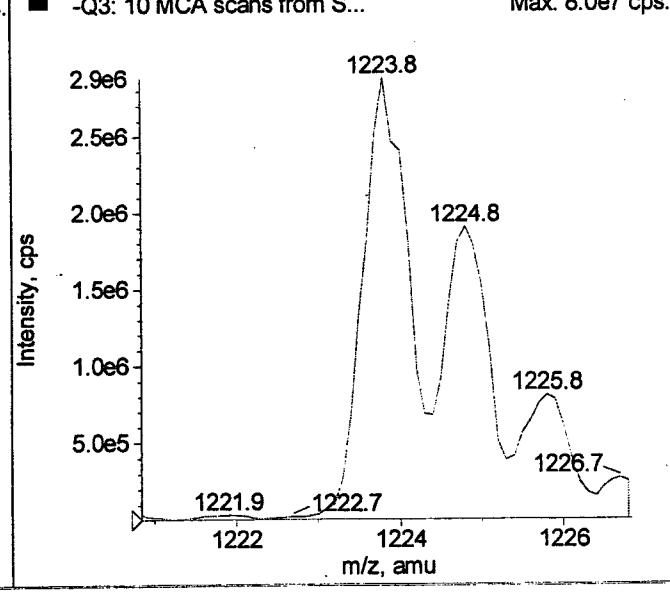
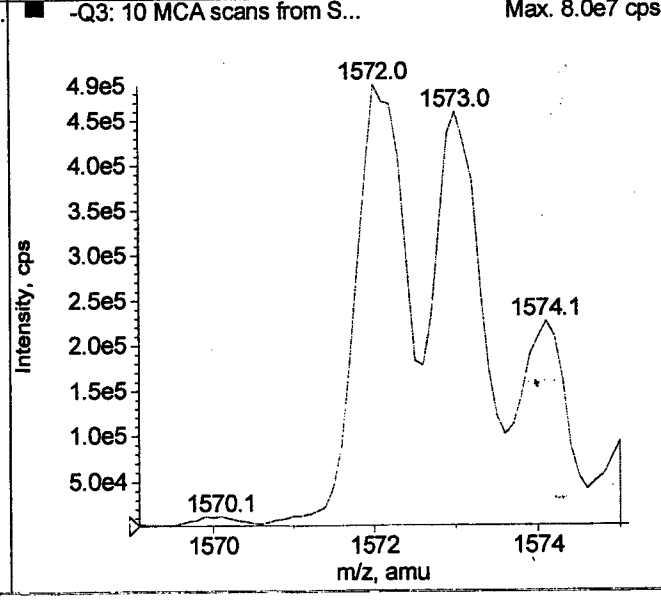
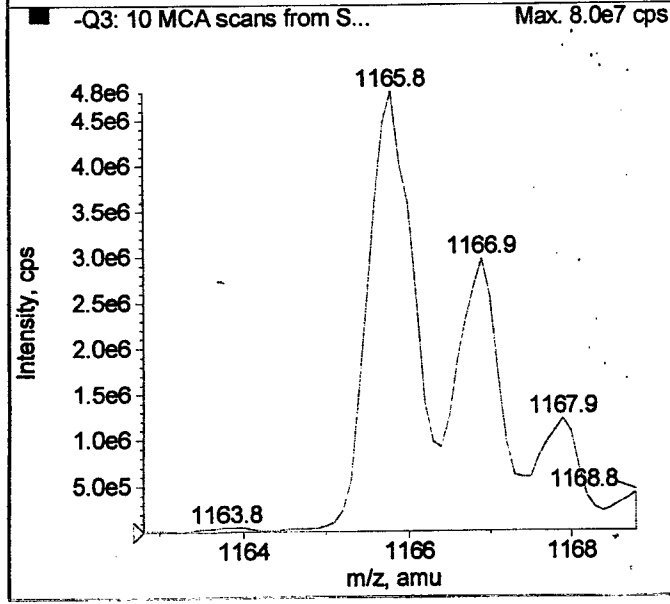
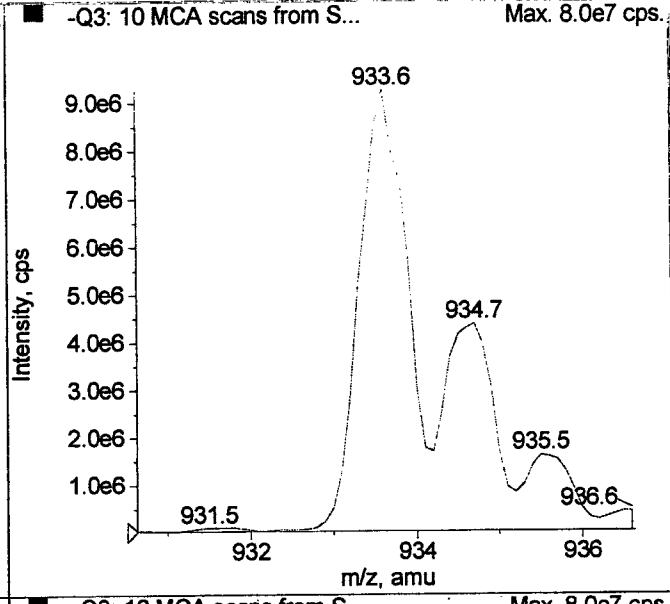
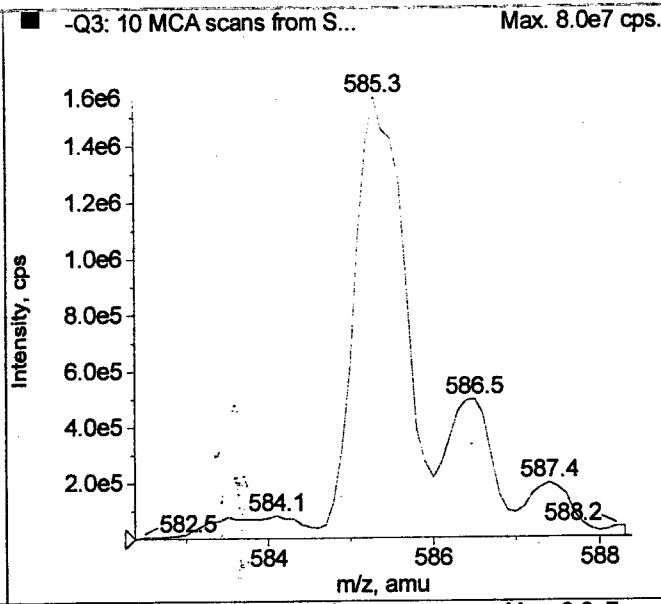
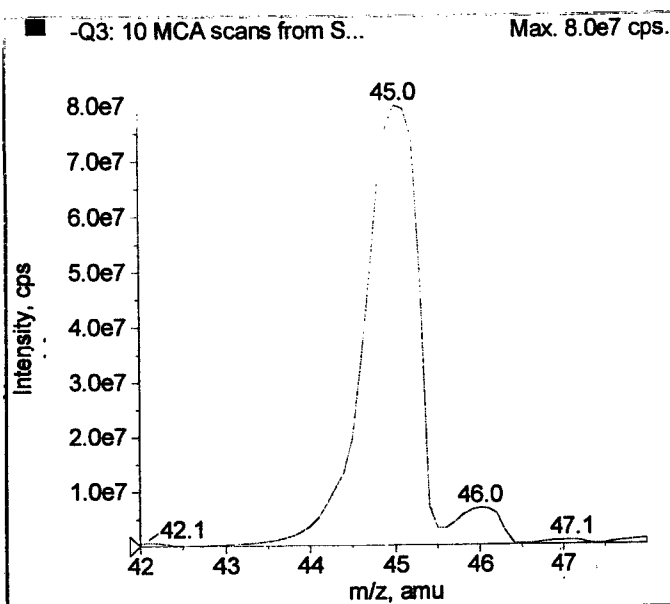
Mass	Dac
44.998	834
585.385	11134
933.636	17774
1165.804	22201
1223.845	23307
1572.097	29946

New Calibration Table

Mass	Dac
44.998	834
585.385	11134

3.636	17773
1165.804	22201
1223.845	23307
1572.097	29946

File path of acquired data for resolution optimization :
D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.07.26.08.27.48



0171

Acq. Date: Tuesday, July 26, 2005
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Exygen STUDY NO. P760

Performed By: *Arms 06/24/05*

Printing Time: 13:05:00

Printing Date: Friday, June 24, 2005

Unit Resolution Optimization for Quad 1, Positive

Friday, June 24, 2005, 12:57

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q1 POS PPG_03Mar03 gv.dam

Threshold: 200 cps
 Search Range: 5.0 amu
 Width At (percent max): 50.0
 Target Peak Width: 0.70
 Peak Width Tolerance: 0.100
 Max Iterations: 20

Original Resolution Table

Mass	Offset
59.050	0.050
175.000	0.107
906.673	0.427
1254.925	0.570
1545.134	0.688
2010.469	0.886

First Resolution Table

Mass	Offset
59.050	0.050
175.000	0.107
906.673	0.427
1254.925	0.570
1545.134	0.688
2010.469	0.886

Iteration 0

Mass	Offset	Found At	Intensity	Width
59.050	0.050	59.069	82115045	0.742
175.000	0.107	175.024	71734878	0.628
906.673	0.427	906.648	74345577	0.689
1254.925	0.570	1254.954	15669557	0.679
1545.134	0.688	1545.129	4858306	0.743
2010.469	0.886	2010.457	8446448	0.797

Successful resolution optimization.

New Resolution Table

Mass	Offset
59.050	0.050
175.000	0.107
906.673	0.427
1254.925	0.570
1545.134	0.688
2010.469	0.886

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
59.050	59.069	-0.019	0.742	-0.042	82115045.00	-0.15
175.000	175.024	-0.024	0.628	0.072	71734878.00	1.12
906.673	906.648	0.025	0.689	0.011	74345577.00	-2.27
1254.925	1254.954	-0.029	0.679	0.021	15669557.50	2.41
1545.134	1545.129	0.005	0.743	-0.043	4858306.53	4.36
2010.469	2010.457	0.012	0.797	-0.097	8446448.63	-2.95

Original Calibration Table

Mass	Dac
59.050	1098
175.000	3309
906.673	17244
1254.925	23875
1545.134	29401
2010.469	38261

New Calibration Table

Mass	Dac
59.050	1098
175.000	3309

Acq. Date: n/a
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906.673	17244
1254.925	23876
1545.134	29401
2010.469	38261

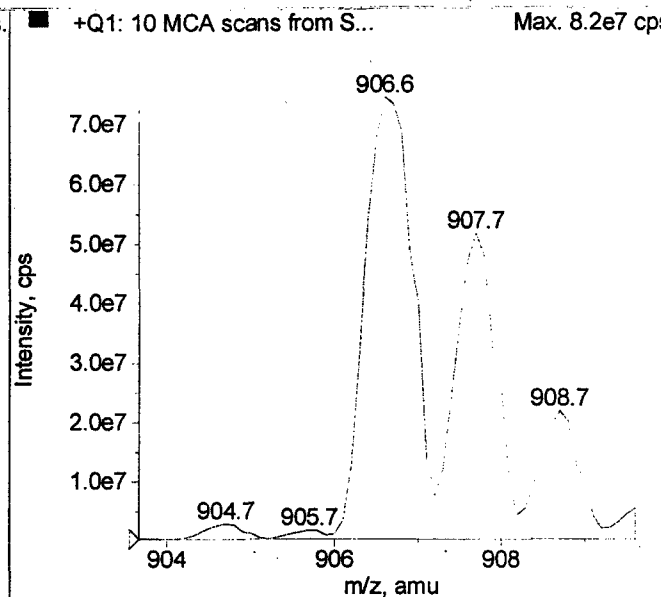
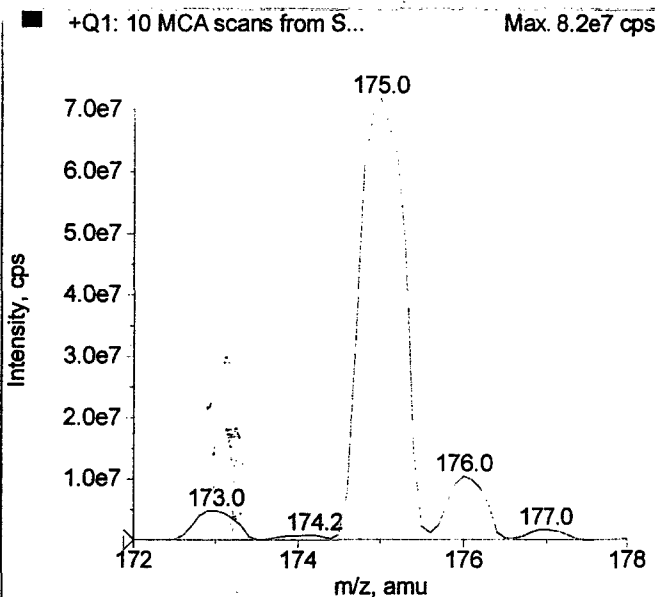
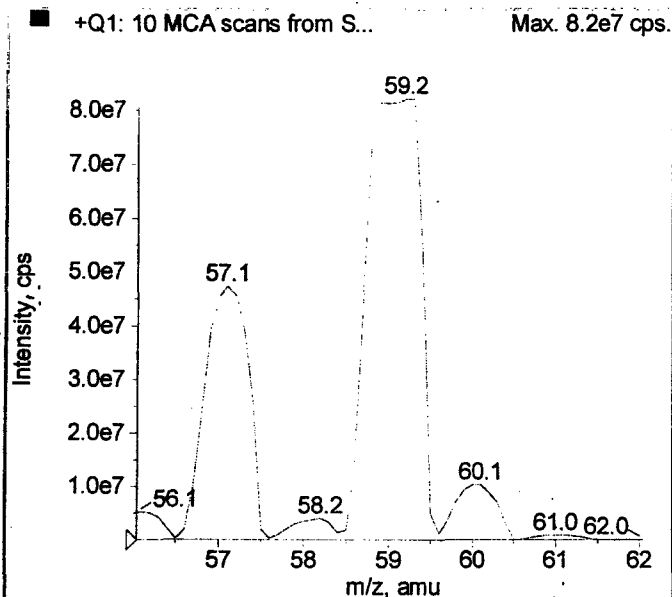
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Acq. Date: n/a
Exygen Research

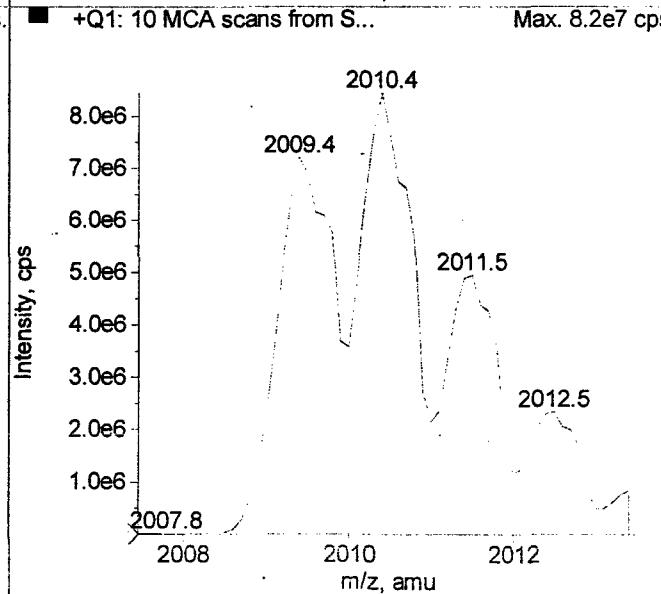
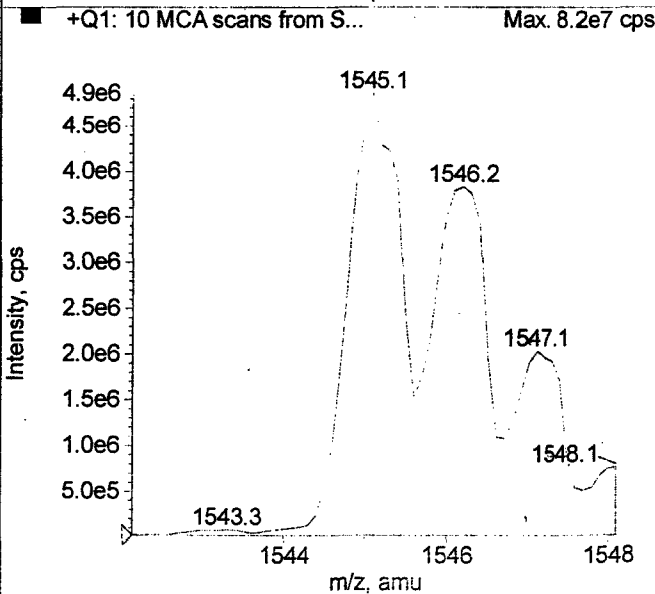
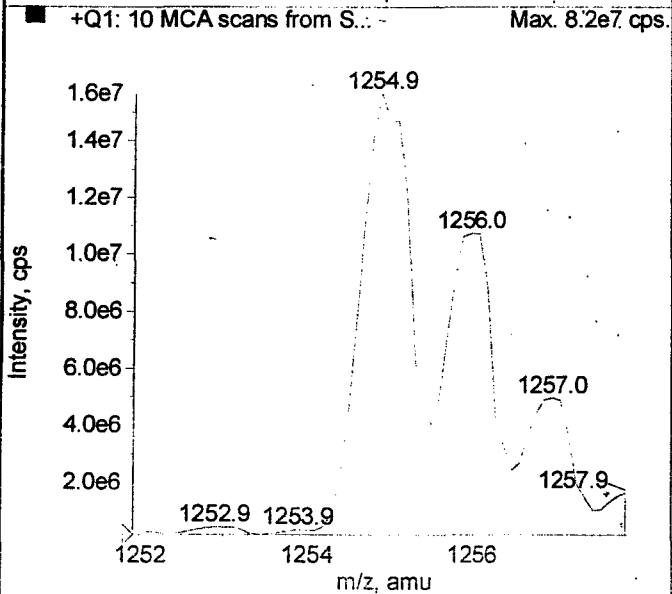
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P760



Performed By: AMES 06/24/05

Printing Date: Friday, June 24,
2005

Unit Resolution Optimization for Quad 3, Positive

Friday, June 24, 2005, 13:18

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q3 POS PPG 051205.dam

Threshold: 200 cps

Search Range: 5.0 amu

Width At (percent max): 50.0

Target Peak Width: 0.70

Peak Width Tolerance: 0.100

Max Iterations: 20

Original Resolution Table

Mass	Offset
59.050	0.055
175.000	0.118
906.673	0.430
1254.925	0.578
1545.134	0.698
2010.469	0.961

First Resolution Table

Mass	Offset
59.050	0.055
175.000	0.118
906.673	0.430
1254.925	0.578
1545.134	0.698
2010.469	0.961

Iteration 0

Mass	Offset	Found At	Intensity	Width
59.050	0.055	59.061	44080428	0.646
175.000	0.118	174.978	7841854	0.635
906.673	0.430	906.663	10734372	0.684
1254.925	0.578	1254.935	5237993	0.678
1545.134	0.698	1545.162	1676251	0.720
2010.469	0.961	2010.457	7166	0.539

Iteration 1

Mass	Offset	Found At	Intensity	Width
59.050	0.055	59.062	45743796	0.647
175.000	0.118	174.981	8302709	0.634
906.673	0.430	906.663	11152019	0.688
1254.925	0.578	1254.933	5547157	0.685
1545.134	0.698	1545.164	1853899	0.727
2010.469	0.951	2010.401	18795	0.723

Successful resolution optimization.

New Resolution Table

Mass	Offset
59.050	0.055
175.000	0.118
906.673	0.430
1254.925	0.578
1545.134	0.698
2010.469	0.951

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
59.050	59.062	-0.012	0.647	0.053	45743796.50	1.09
175.000	174.981	0.019	0.634	0.066	8302709.50	-0.43
906.673	906.663	0.010	0.688	0.012	11152019.56	-1.17
1254.925	1254.933	-0.008	0.685	0.015	5547157.47	-2.92
1545.134	1545.164	-0.030	0.727	-0.027	1853899.05	-6.77
2010.469	2010.401	0.068	0.723	-0.023	18795.43	52.88

Original Calibration Table

Mass	Dac
59.050	1100
175.000	3314

Acq. Date: n/a
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906.673	17259
1254.925	23897
1545.134	29428
2010.469	38309

New Calibration Table

Mass	Dac
59.050	1100
175.000	3314
906.673	17259
1254.925	23897
1545.134	29429
2010.469	38308

File path of acquired data for resolution optimization :

D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.06.24.13.18.15

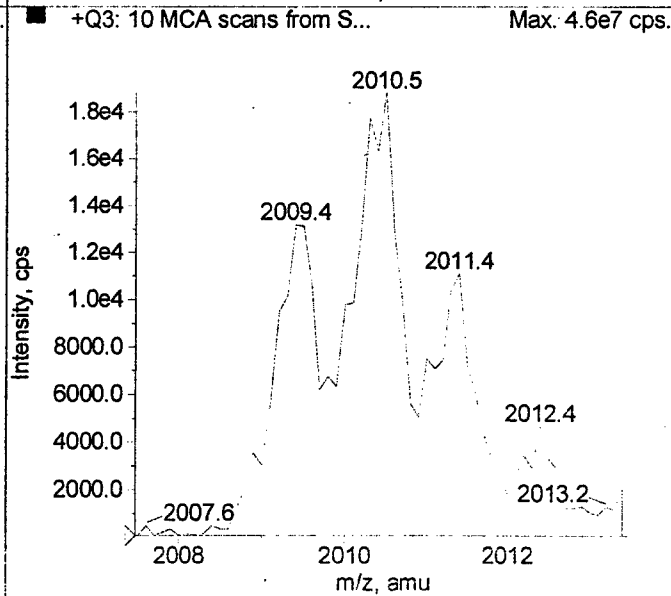
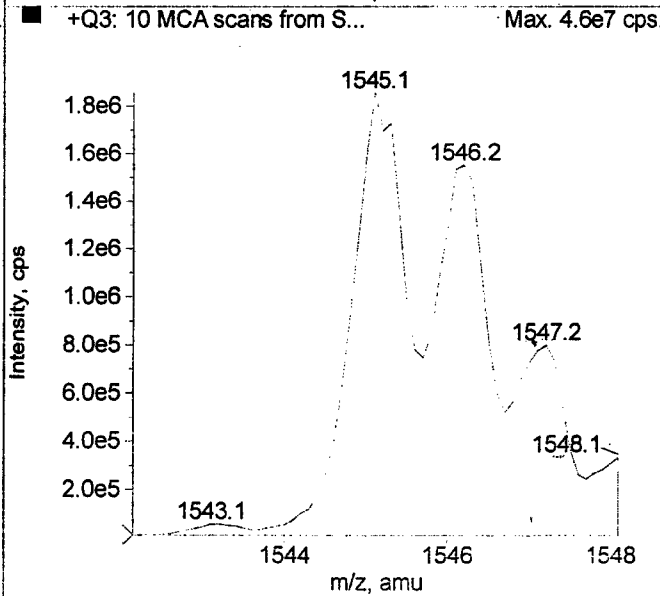
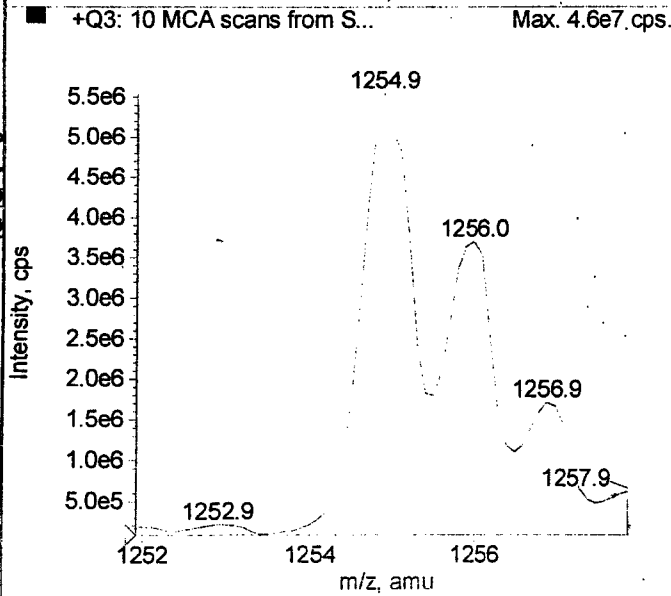
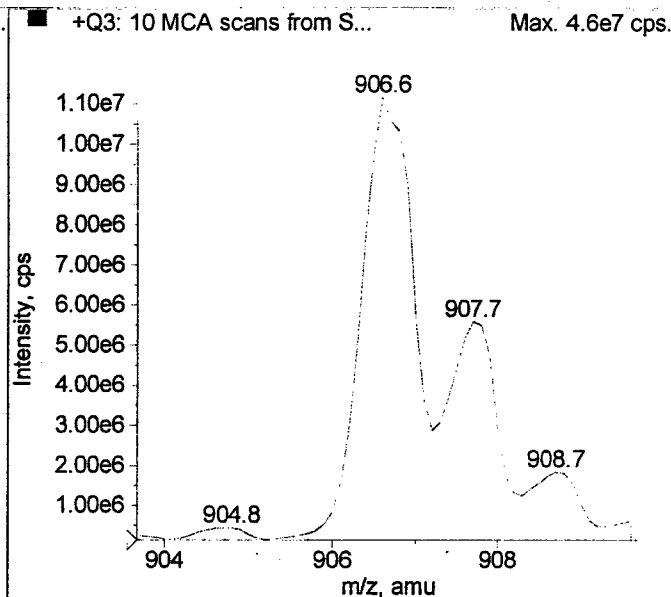
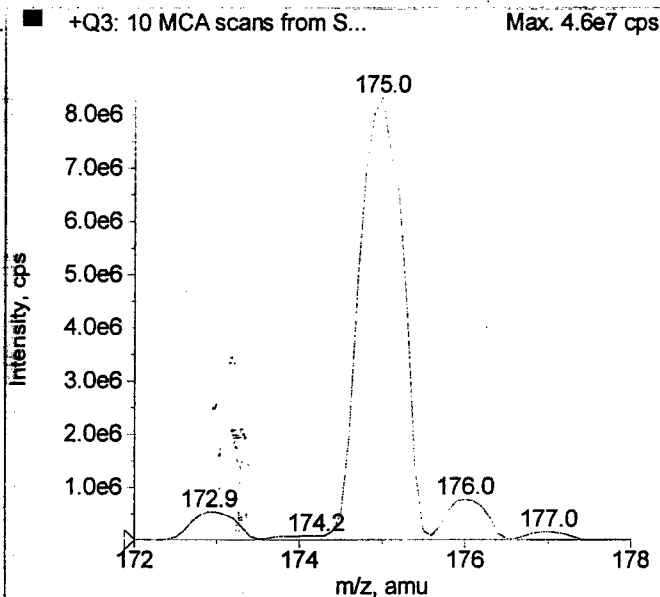
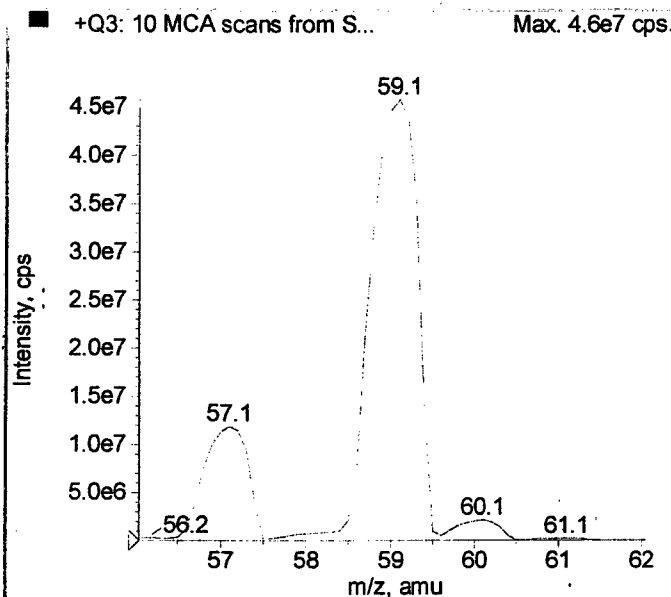
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4210

Unit Resolution Optimization for Quad 1, Negative

Friday, June 24, 2005, 13:49

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q1 NEG PPG 081803.dam

Threshold: 200 cps
Search Range: 5.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
44.998	0.053
585.385	0.289
933.636	0.438
1165.804	0.532
1572.097	0.706
1863.306	0.830
2037.431	0.895
2211.557	0.970

First Resolution Table

Mass	Offset
44.998	0.053
585.385	0.289
933.636	0.438
1165.804	0.532
1572.097	0.706
1863.306	0.830
2037.431	0.895
2211.557	0.970

Iteration 0

Mass	Offset	Found At	Intensity	Width
44.998	0.053	44.990	80111398	0.731
585.385	0.289	585.383	4947099	0.688
933.636	0.438	933.628	23958562	0.657
1165.804	0.532	1165.758	14590182	0.697
1572.097	0.706	1572.061	1369618	0.688
1863.306	0.830	1863.313	2213323	0.638
2037.431	0.895	2037.469	2320109	0.730
2211.557	0.970	2211.512	1360537	0.769

Successful resolution optimization.

New Resolution Table

Mass	Offset
44.998	0.053
585.385	0.289
933.636	0.438
1165.804	0.532
1572.097	0.706
1863.306	0.830
2037.431	0.895
2211.557	0.970

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
44.998	44.990	0.008	0.731	-0.031	80111398.00	-2.37
585.385	585.383	0.002	0.688	0.012	4947099.78	19.76
933.636	933.628	0.008	0.657	0.043	23958562.75	11.92
1165.804	1165.758	0.046	0.697	0.003	14590182.75	19.50
1572.097	1572.061	0.036	0.688	0.012	1369618.66	2.11
1863.306	1863.313	-0.007	0.638	0.062	2213323.69	-0.15
2037.431	2037.469	-0.038	0.730	-0.030	2320109.80	-8.08
2211.557	2211.512	0.045	0.769	-0.069	1360537.80	6.08

Original Calibration Table

Mass Dac

Acq. Date: n/a
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44.998	832
585.385	11121
933.636	17757
1165.804	22180
1572.097	29917
1863.306	35460
2037.431	38774
2211.557	42091

New Calibration Table

Mass	Dac
44.998	832
585.385	11121
933.636	17757
1165.804	22179
1572.097	29916
1863.306	35460
2037.431	38775
2211.557	42090

File path of acquired data for resolution optimization :
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Acq. Date: n/a
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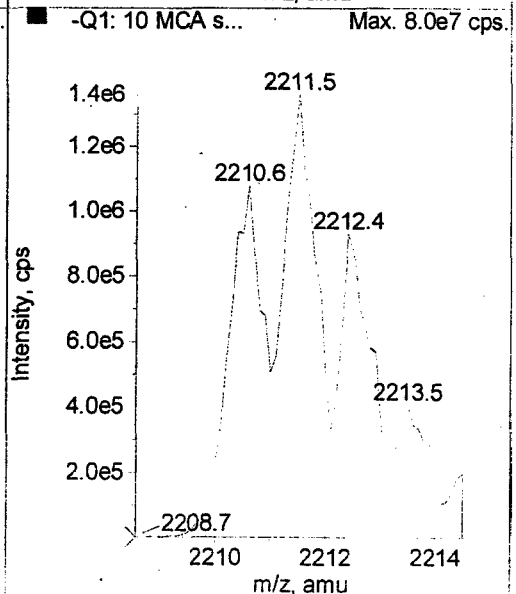
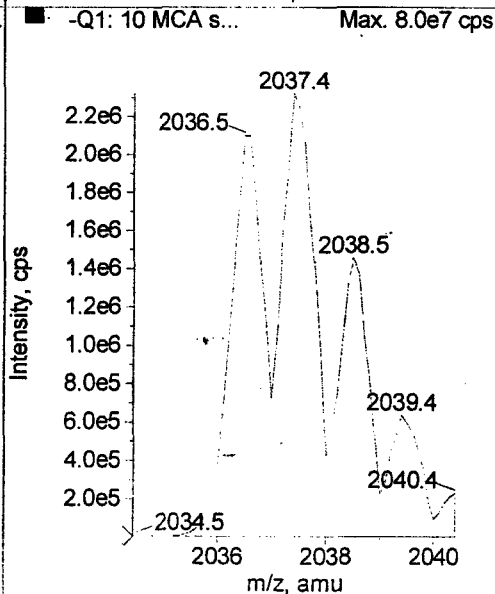
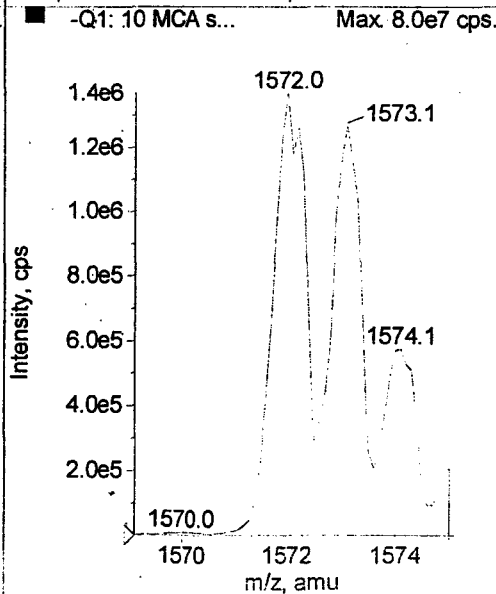
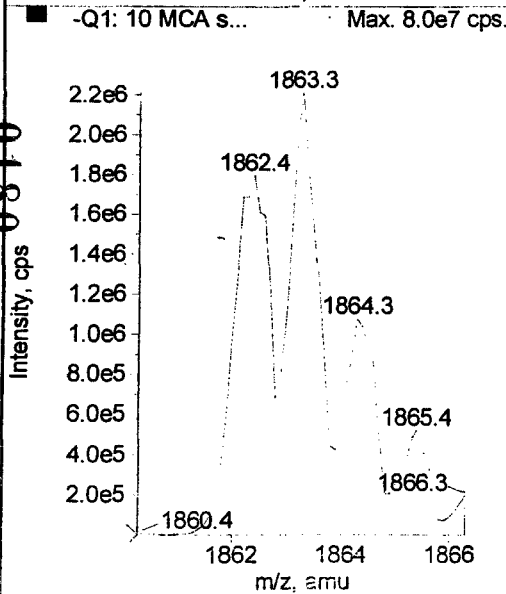
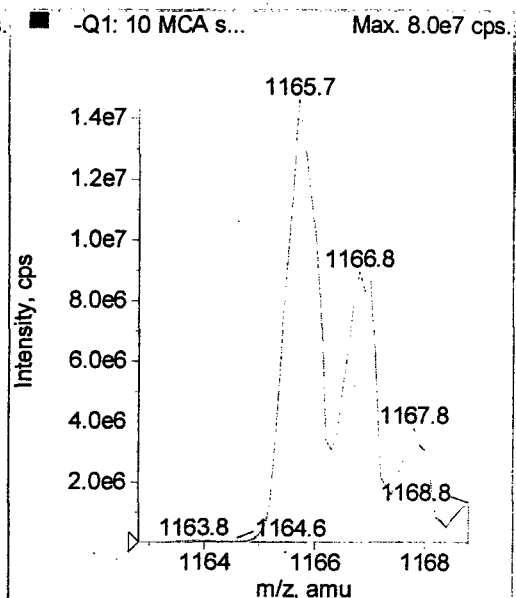
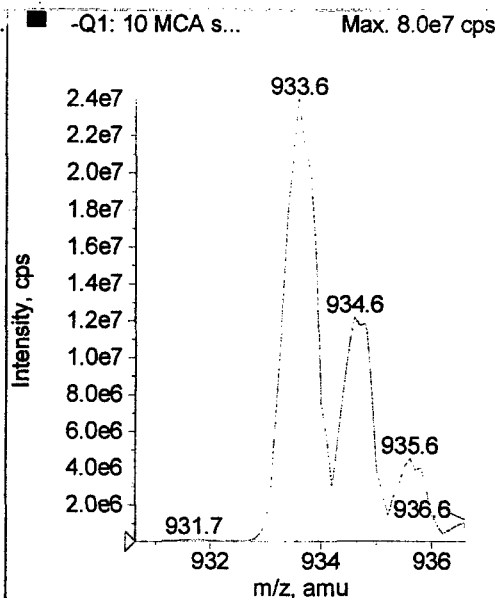
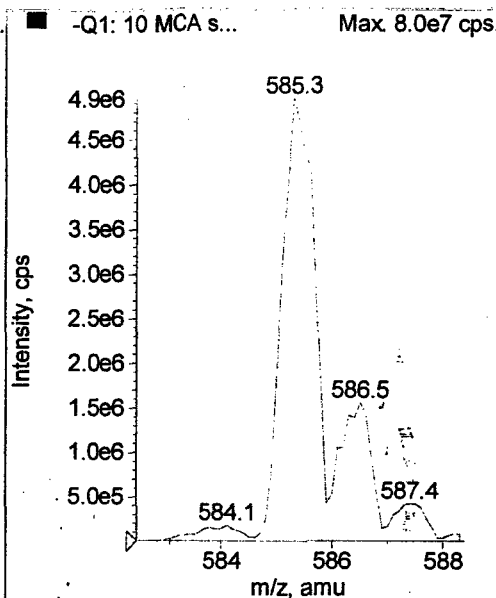
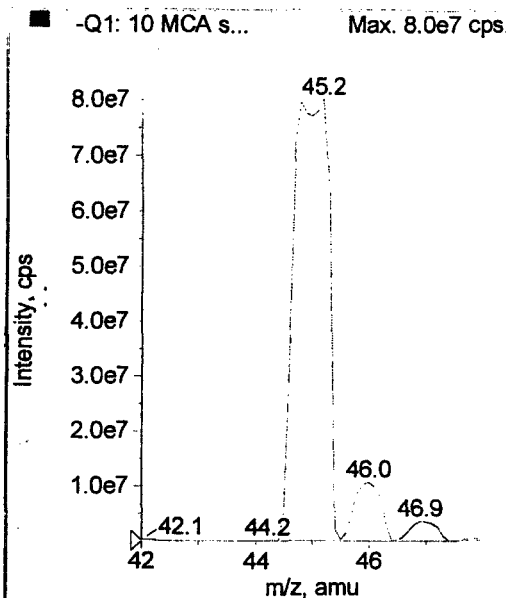
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0179

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Performed By: AMB 06/24/05

Printing Date: Friday, June 24, 2005



0810

Acq. Date: Friday, June 24, 2005
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Unit Resolution Optimization for Quad 3, Negative

Friday, June 24, 2005, 16:02

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q3 NEG PFG 081803.dam

Threshold: 200 cps
Search Range: 5.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
44.998	0.050
585.385	0.290
933.636	0.435
1165.804	0.537
1572.097	0.707
1863.306	0.831
2211.557	0.992

First Resolution Table

Mass	Offset
44.998	0.050
585.385	0.290
933.636	0.435
1165.804	0.537
1223.845	0.561
1572.097	0.707

Iteration 0

Mass	Offset	Found At	Intensity	Width
44.998	0.050	44.998	88717888	0.784
585.385	0.290	585.407	2473143	0.766
933.636	0.435	933.648	16372704	0.761
1165.804	0.537	1165.847	8892008	0.694
1223.845	0.561	1223.868	5561614	0.755
1572.097	0.707	1572.125	944368	0.701

Successful resolution optimization.

New Resolution Table

Mass	Offset
44.998	0.050
585.385	0.290
933.636	0.435
1165.804	0.537
1223.845	0.561
1572.097	0.707

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	PW Shift	Intensity	Intensity Diff
44.998	44.998	0.000	0.784	-0.084	88717888.00	-0.12
585.385	585.407	-0.022	0.766	-0.066	2473143.23	4.52
933.636	933.648	-0.012	0.761	-0.061	16372704.25	0.93
1165.804	1165.847	-0.043	0.694	0.006	8892008.94	6.22
1223.845	1223.868	-0.023	0.755	-0.055	5561614.06	6.22
1572.097	1572.125	-0.028	0.701	-0.001	944368.08	8.97

Original Calibration Table

Mass	Dac
44.998	833
585.385	11132
933.636	17772
1165.804	22199
1572.097	29943
1863.306	35475
2211.557	42129

New Calibration Table

Mass	Dac
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Acq. Date: n/a
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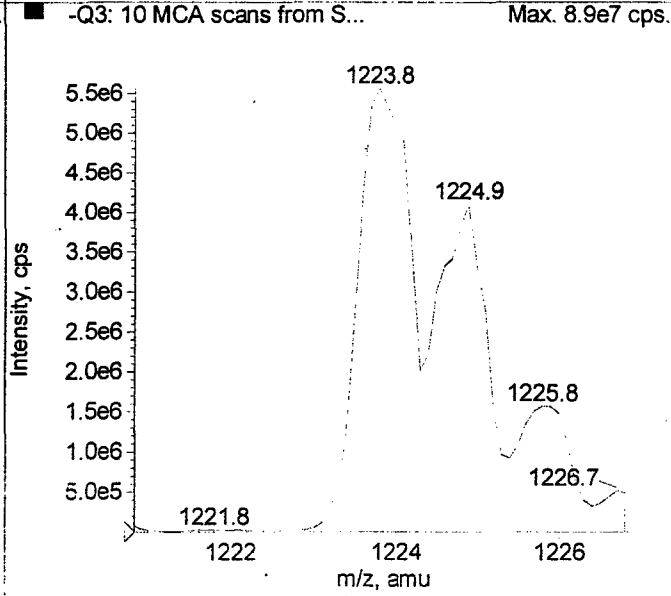
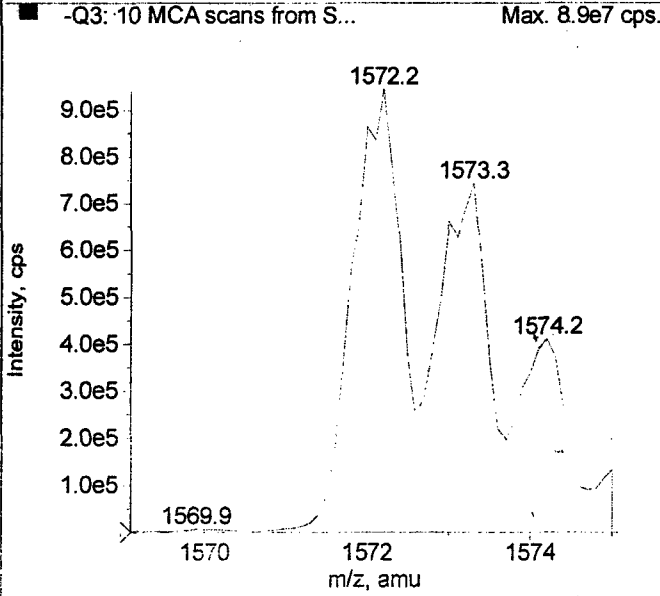
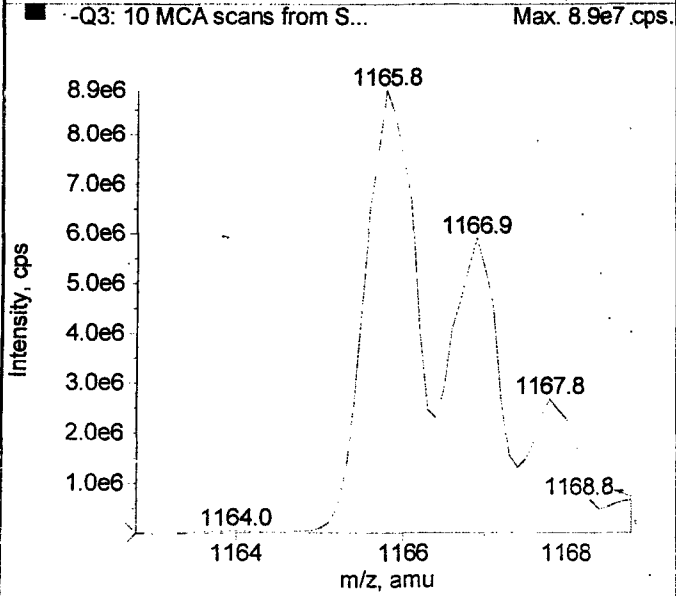
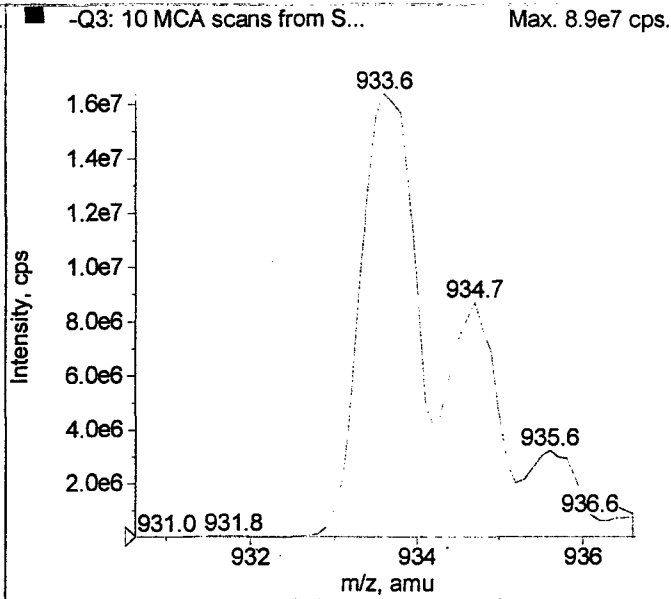
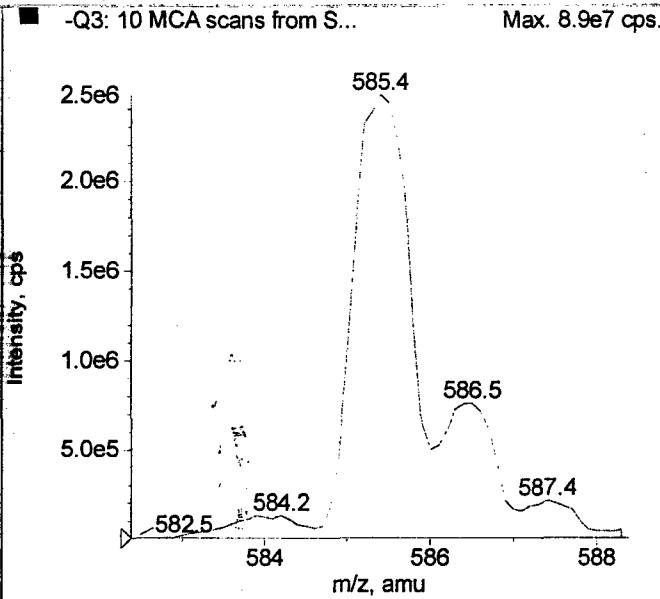
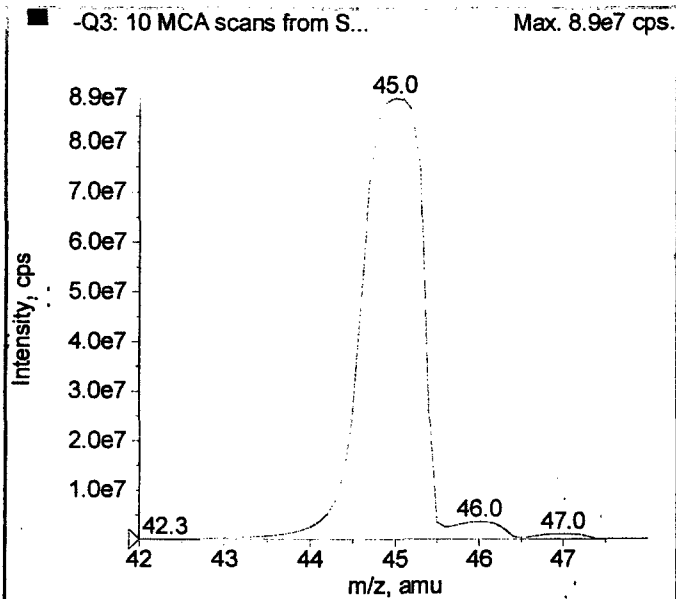
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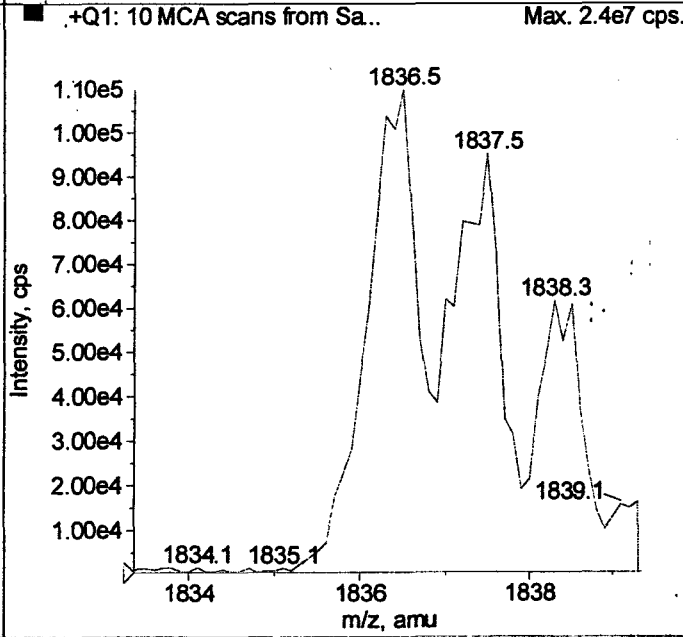
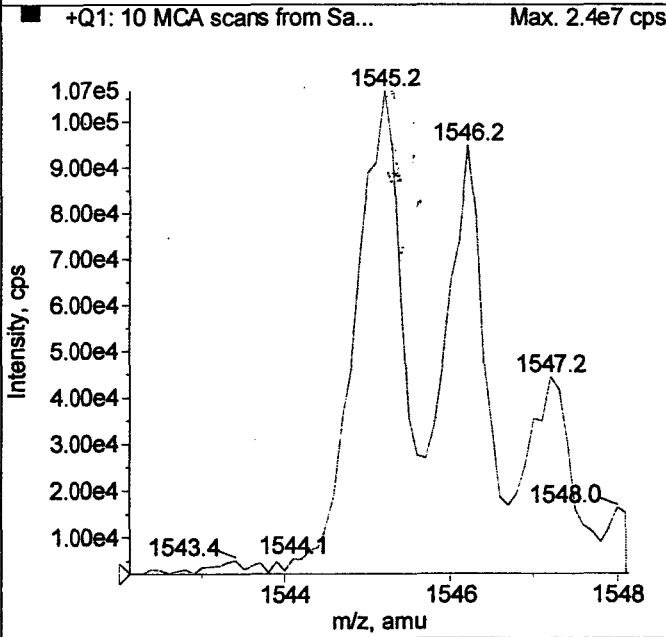
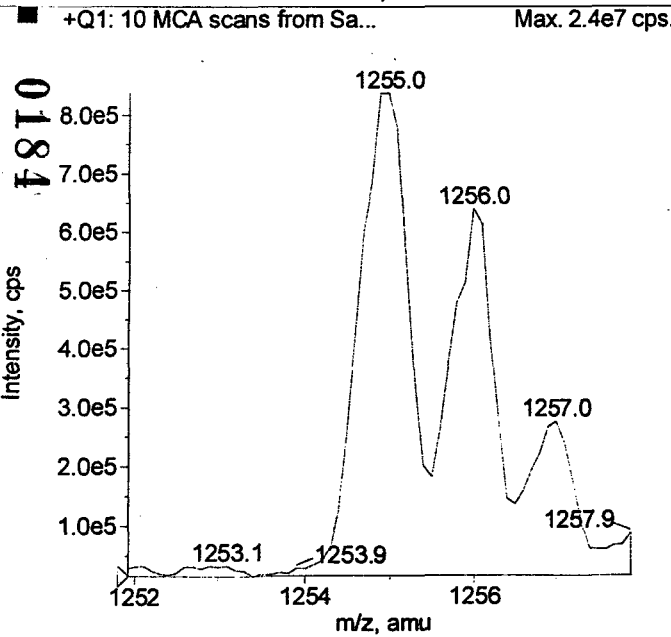
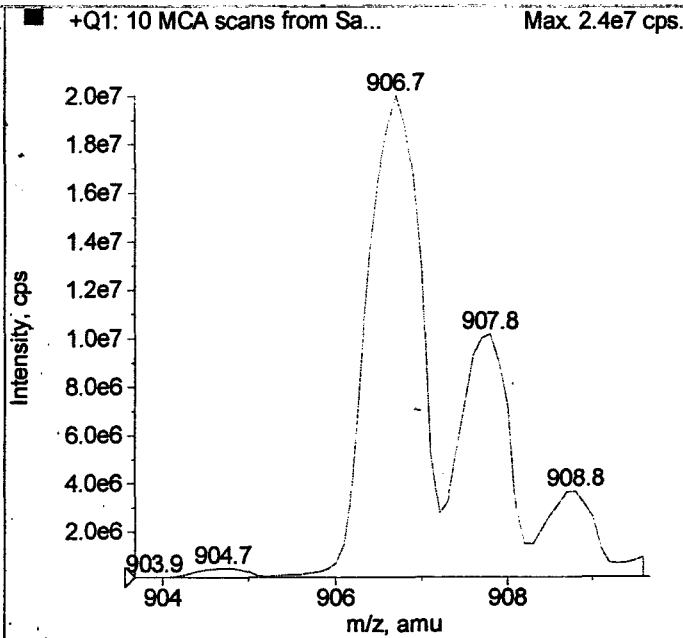
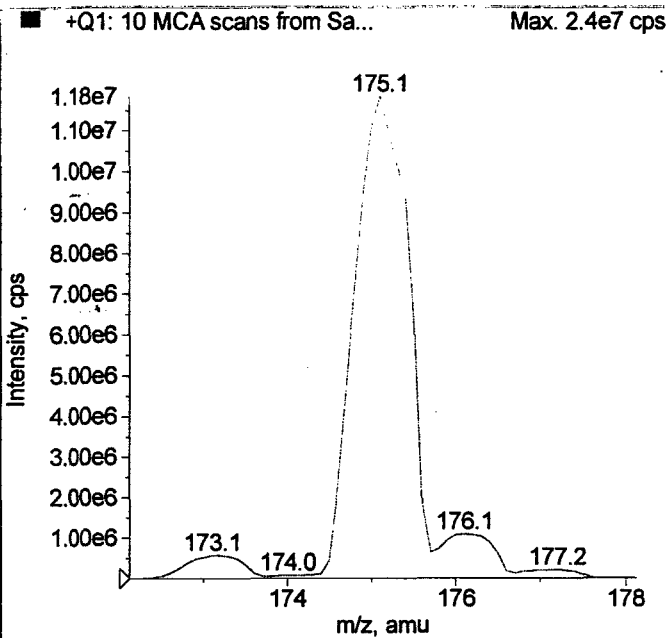
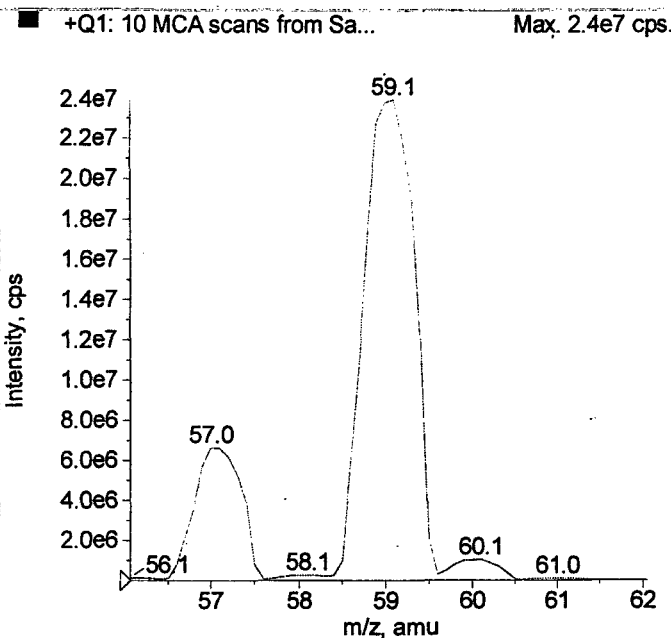
BY BKAC DATE 08/08/05

44.998	833
585.385	11132
933.636	17772
1165.804	22200
1223.845	23306
1572.097	29944

File path of acquired data for resolution optimization :
D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.06.24.16.02.05



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Oxygen STUDY NO. P760

BY AMS DATE 08/05/05

Resolution Optimization for Quad 1, Positive

Friday, August 05, 2005, 13:10

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q1 PFG Positive02.dam

Threshold: 200 cps.

Search Range: 3.0 amu

Width At (percent max): 50.0

Target Peak Width: 0.70

Peak Width Tolerance: 0.100

Max Iterations: 20

Original Resolution Table

Mass	Offset
59.050	0.070
175.133	0.115
906.673	0.398
1254.925	0.525
1545.134	0.627
1836.344	0.729

First Resolution Table

Mass	Offset
59.050	0.070
175.133	0.115
906.673	0.398
1254.925	0.525
1545.134	0.627
1836.344	0.729

The masses selected in the reference table do not match the masses in the acquisition method.

Iteration 0

Mass	Offset	Found At	Intensity	Width
59.050	0.070	59.053	23909661	0.686
175.133	0.115	175.131	11836541	0.763
906.673	0.398	906.695	20024774	0.718
1254.925	0.525	1254.948	839364	0.669
1545.134	0.627	1545.146	106780	0.603
1836.344	0.729	1836.397	109871	0.620

Successful resolution optimization.

New Resolution Table

Mass	Offset
59.050	0.070
175.133	0.115
906.673	0.398
1254.925	0.525
1545.134	0.627
1836.344	0.729

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
59.050	59.053	-0.003	0.686	0.014	23909661.00	3.86
175.133	175.131	0.002	0.763	-0.063	11836541.25	-0.71
906.673	906.695	-0.022	0.718	-0.018	20024774.00	0.14
1254.925	1254.948	-0.023	0.669	0.031	839364.80	-17.76
1545.134	1545.146	-0.012	0.603	0.097	106780.36	-14.83
1836.344	1836.397	-0.053	0.620	0.080	109871.35	-15.81

Original Calibration Table

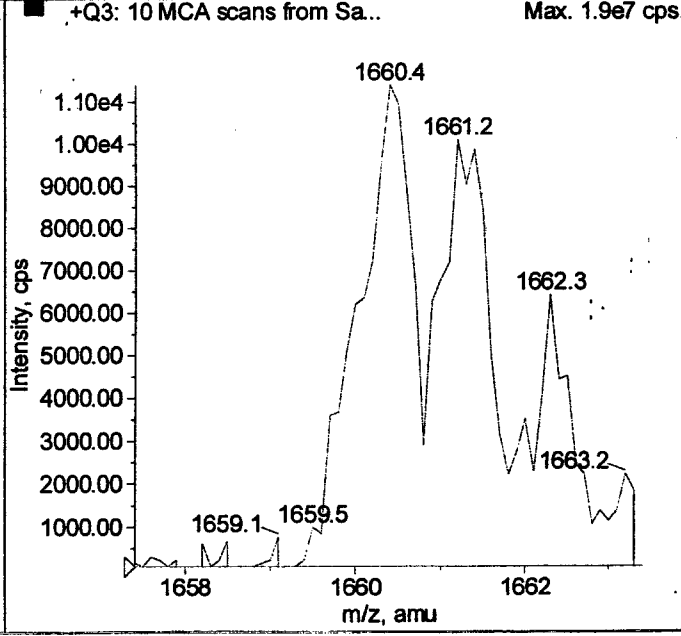
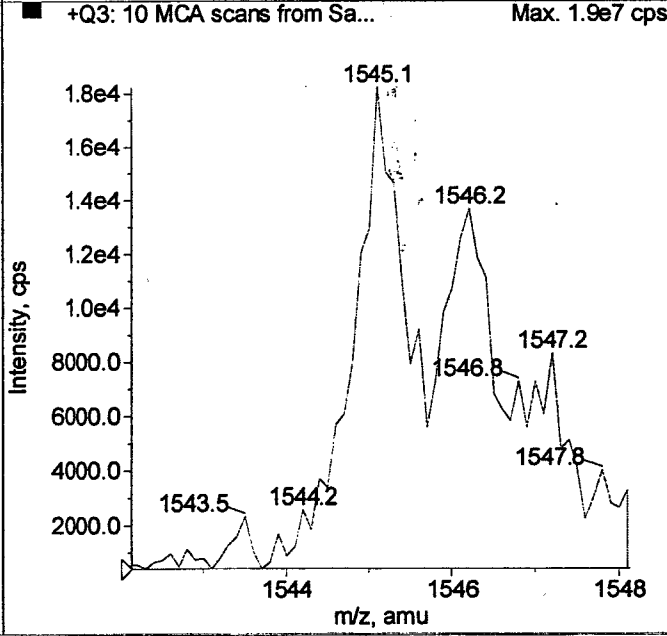
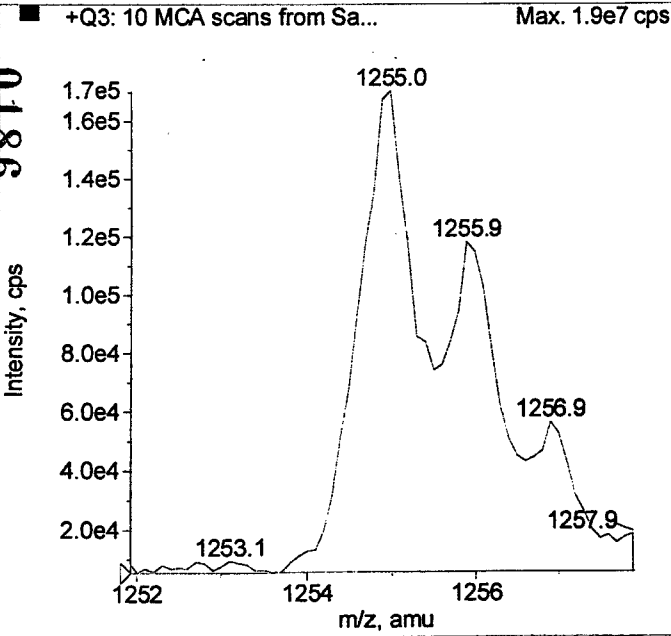
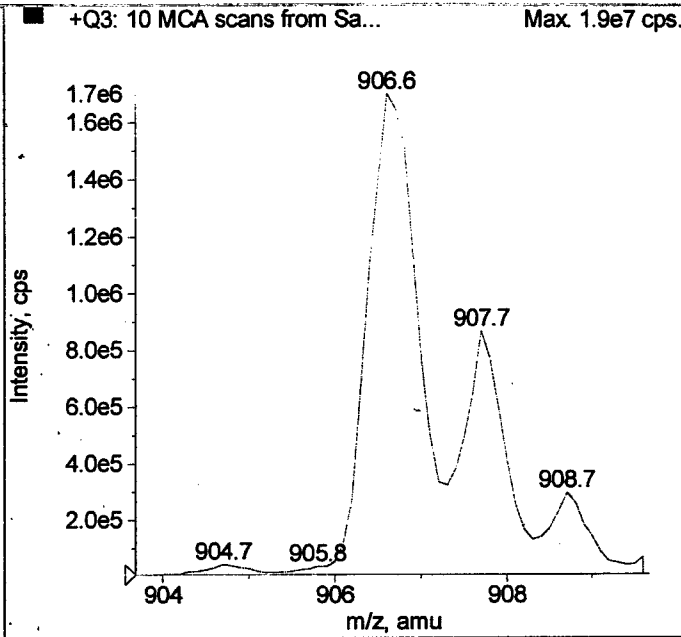
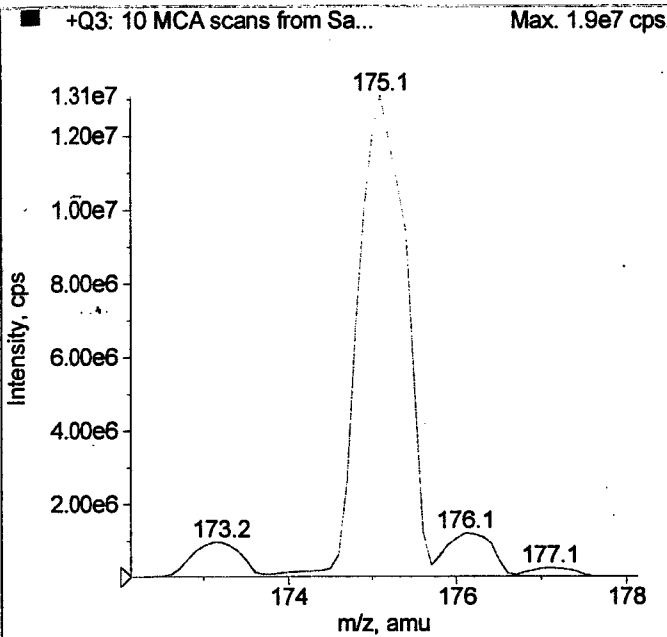
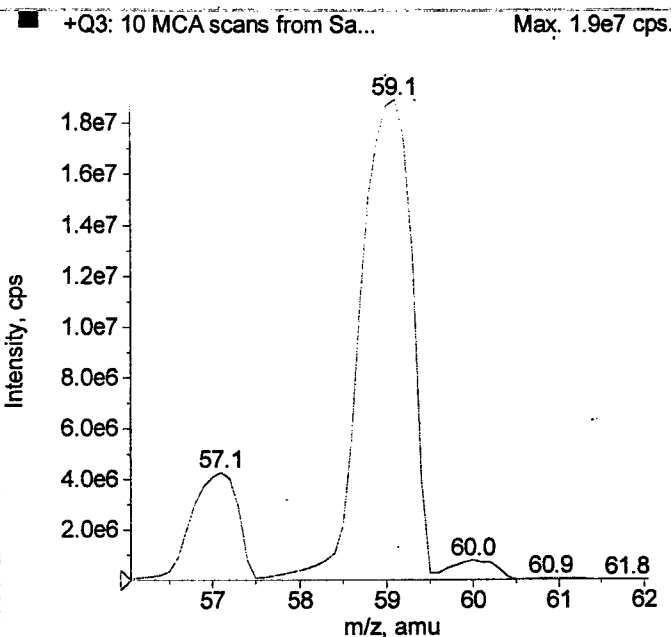
Mass	Dac
59.050	1098
175.133	3307
906.673	17245
1254.925	23881
1545.134	29413
1836.344	34945

New Calibration Table

Mass	Dac
59.050	1098
175.133	3307
906.673	17245
1254.925	23881
1545.134	29413
1836.344	34946

File path of acquired data for resolution optimization :

D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.08.05.13.10.07



9870

Oxygen Research

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Oxygen STUDY NO. P760

Resolution Optimization for Quad 3, Positive

Friday, August 05, 2005, 13:35

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q3+ MCRrev1.dam

Threshold: 200 cps
Search Range: 3.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
59.050	0.075
175.133	0.145
906.673	0.556
1254.925	0.733
1545.134	0.896
1660.400	0.921

First Resolution Table

Mass	Offset
59.050	0.075
175.133	0.145
906.673	0.556
1254.925	0.733
1545.134	0.896
1660.400	0.921

The masses selected in the reference table do not match the masses in the acquisition method.

Iteration 0

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.028	19091067	0.670
175.133	0.145	175.134	13168679	0.670
906.673	0.556	906.656	1715421	0.640
1254.925	0.733	1254.957	207493	0.809
1545.134	0.896	1545.146	16796	0.612
1660.400	0.921	1660.380	20659	0.608

Iteration 1

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.029	18976489	0.669
175.133	0.145	175.132	13213717	0.665
906.673	0.556	906.660	1721521	0.622
1254.925	0.743	1254.978	120992	0.576
1545.134	0.896	1545.247	16956	0.945
1660.400	0.921	1660.304	16756	0.781

Iteration 2

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.030	19066464	0.669
175.133	0.145	175.132	12943487	0.677
906.673	0.556	906.659	1701594	0.642
1254.925	0.738	1254.943	180044	0.731
1545.134	0.906	1545.189	11117	0.695
1660.400	0.921	1660.347	16680	0.807

Iteration 3

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.023	18927994	0.671
175.133	0.145	175.128	13064979	0.677
906.673	0.556	906.651	1709423	0.637
1254.925	0.738	1254.960	187809	0.638
1545.134	0.906	1545.251	9358	0.597
1660.400	0.931	1661.317	5815	0.345

Iteration 4

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.025	18897595	0.668
175.133	0.145	175.131	13085516	0.675
906.673	0.556	906.657	1699053	0.638
1254.925	0.738	1254.944	170563	0.749
1545.134	0.896	1545.145	18236	0.634
1660.400	0.926	1660.416	11400	0.772

Successful resolution optimization.

New Resolution Table

Mass	Offset
59.050	0.075
175.133	0.145
906.673	0.556
1254.925	0.738
1545.134	0.896
1660.400	0.926

Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
59.050	59.025	0.025	0.668	0.032	18897595.88	-0.52

Printing Date: Fri August 5, 05
Performed by: AMBS 08/05/05 Instrument Calibration

API 4000
LC/MS/MS 8

175.133	175.131	0.002	0.675	0.025	13085516.25	-1.02
906.673	906.657	0.016	0.638	0.062	1699053.27	3.71
1254.925	1254.944	-0.019	0.749	-0.049	170563.70	-23.93
1545.134	1545.145	-0.011	0.634	0.066	18236.65	-0.44
1660.400	1660.416	-0.016	0.772	-0.072	11400.87	-27.52

Original Calibration Table

Mass	Dac
59.050	1096
175.133	3304
906.673	17225
1254.925	23851
1545.134	29372
1660.400	31584

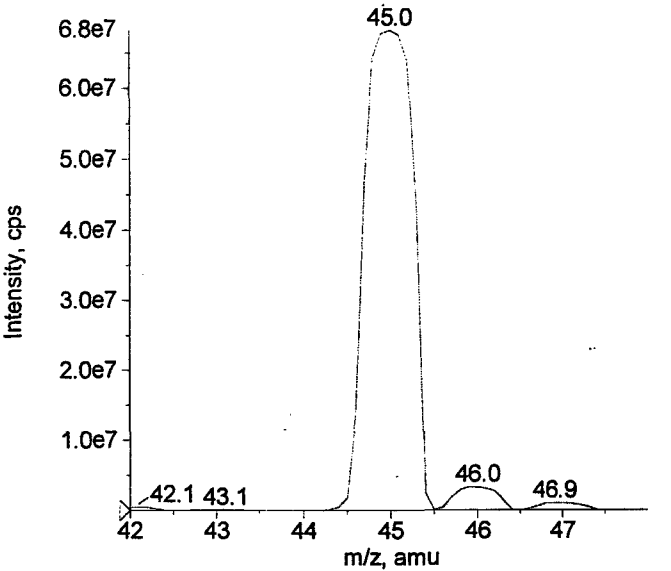
New Calibration Table

Mass	Dac
59.050	1096
175.133	3304
906.673	17225
1254.925	23851
1545.134	29372
1660.400	31584

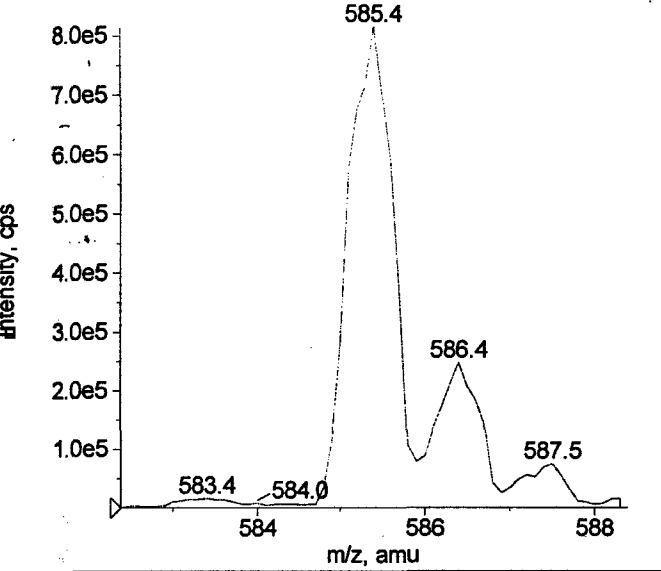
File path of acquired data for resolution optimization :

D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.08.05.13.35.12

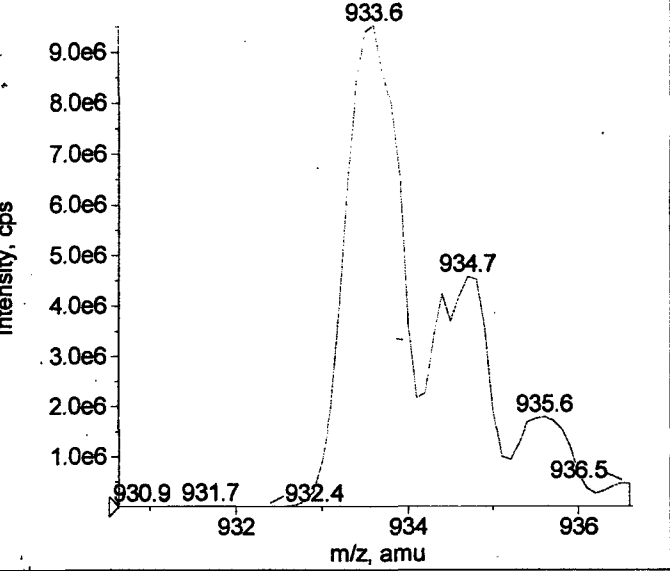
-Q1: 10 MCA scans from Sa... Max. 6.8e7 cps.



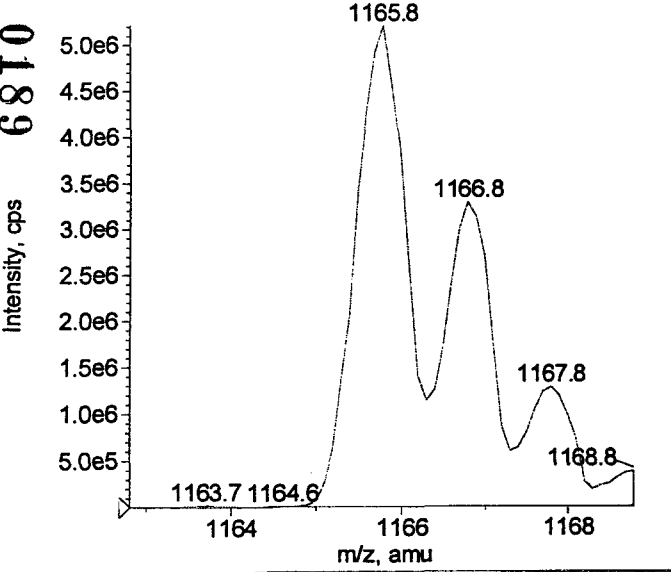
-Q1: 10 MCA scans from Sa... Max. 6.8e7 cps.



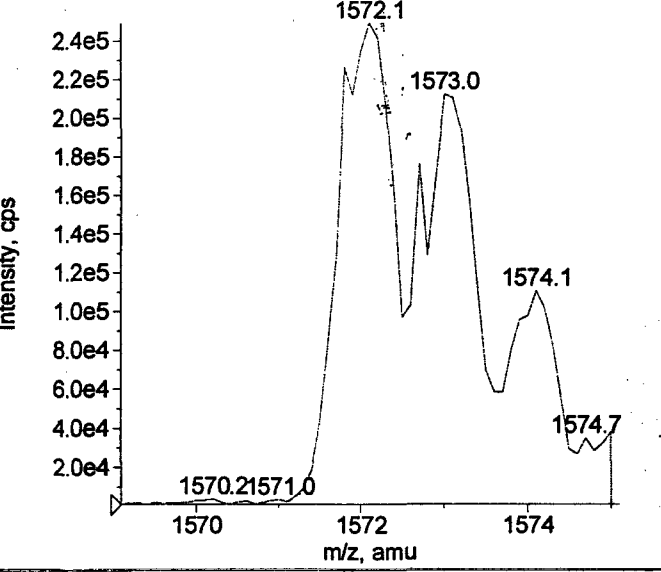
-Q1: 10 MCA scans from Sa... Max. 6.8e7 cps.



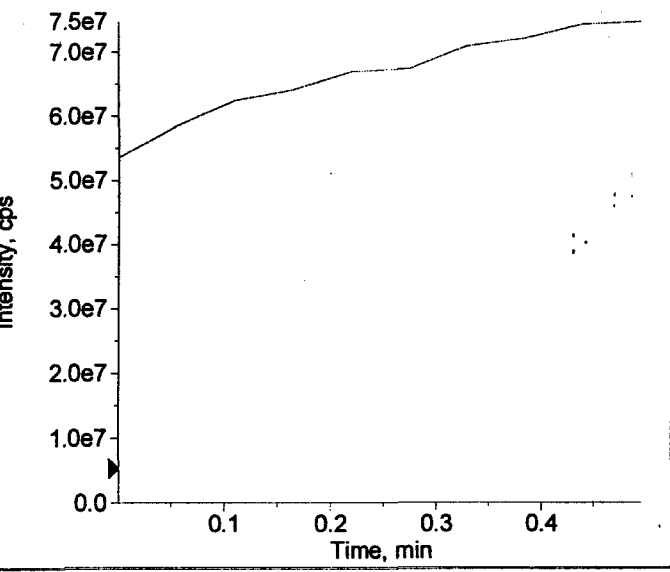
-Q1: 10 MCA scans from Sa... Max. 6.8e7 cps.



-Q1: 10 MCA scans from Sa... Max. 6.8e7 cps.



TIC of -Q1: from Sample 1 of ... Max. 7.5e7 cps.



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Oxygen STUDY NO: P760

Resolution Optimization for Quad 1, Negative

Fri, August 05, 2005, 13:43

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q1 neg ppg pc01.dam

Threshold: 200 cps
Search Range: 3.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
44.998	0.055
585.385	0.269
933.636	0.392
1165.804	0.482
1572.097	0.630

First Resolution Table

Mass	Offset
44.998	0.055
585.385	0.269
933.636	0.392
1165.804	0.482
1572.097	0.630

Iteration 0

Mass	Offset	Found At	Intensity	Width
44.998	0.055	44.993	68270600	0.660
585.385	0.269	585.357	815100	0.641
933.636	0.392	933.592	9517100	0.735
1165.804	0.482	1165.766	5217578	0.661
1572.097	0.630	1572.066	249270	0.767

Successful resolution optimization.

New Resolution Table

Mass	Offset
44.998	0.055
585.385	0.269
933.636	0.392
1165.804	0.482
1572.097	0.630

Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
44.998	44.993	0.005	0.660	0.040	68270600.00	-3.22
585.385	585.357	0.028	0.641	0.059	815100.00	-143.49
933.636	933.592	0.044	0.735	-0.035	9517100.00	-122.86
1165.804	1165.766	0.038	0.661	0.039	5217578.59	-85.10
1572.097	1572.066	0.031	0.767	-0.067	249270.46	-21.67

Original Calibration Table

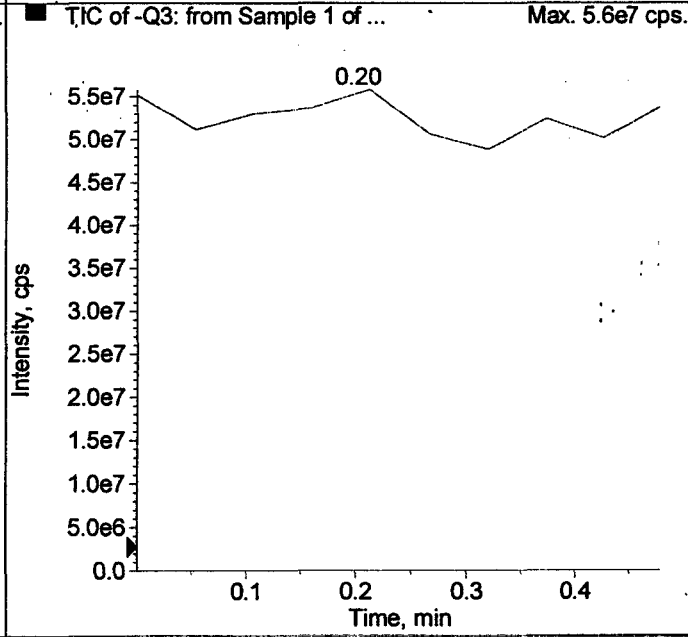
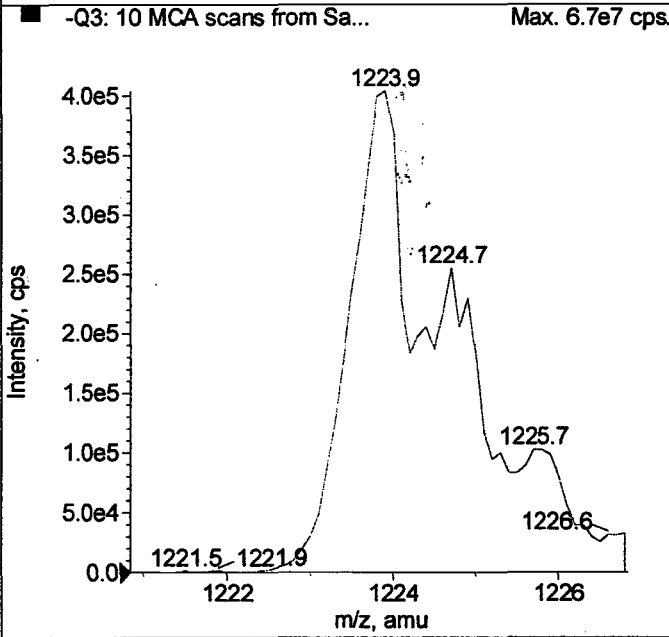
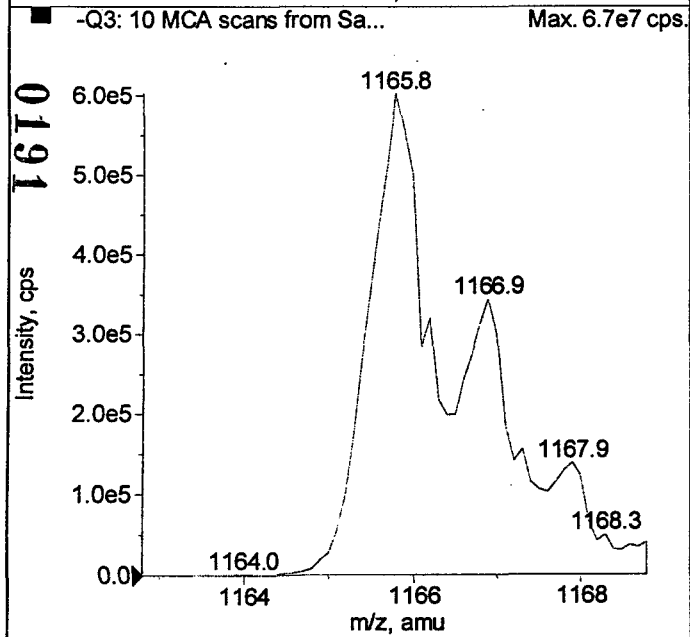
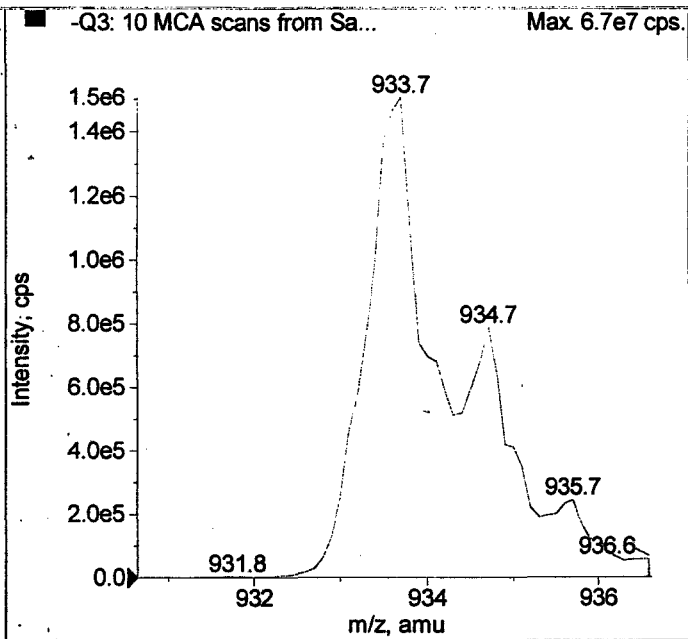
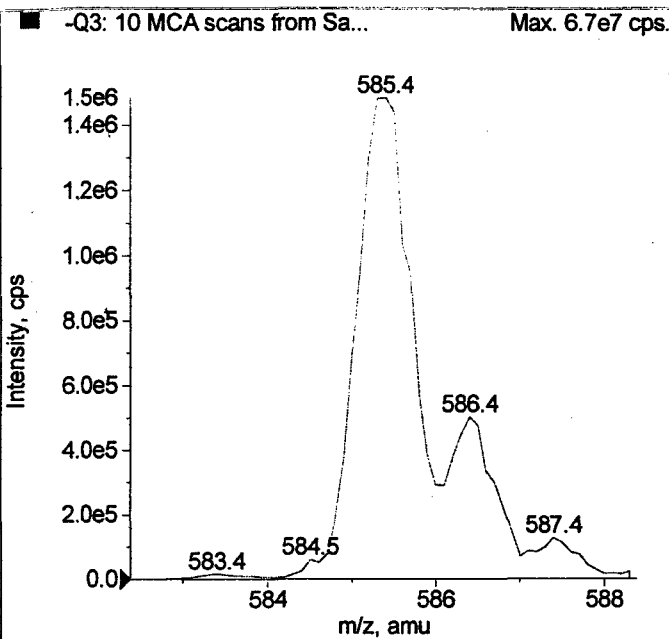
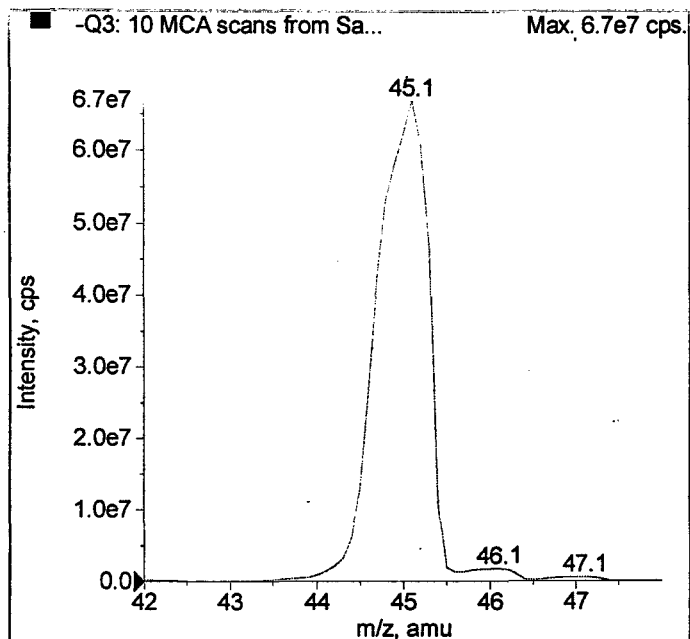
Mass	Dac
44.998	832
585.385	11123
933.636	17759
1165.804	22184
1572.097	29928

New Calibration Table

Mass	Dac
44.998	832
585.385	11122
933.636	17758
1165.804	22183
1572.097	29927

File path of acquired data for resolution optimization :

D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.08.05.13.43.13



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Oxygen STUDY NO: P760

Resolution Optimization for Quad 3, Negative

Fri, August 05, 2005, 14:21

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q3- MDN.dam

Threshold: 200 cps
Search Range: 3.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
44.998	0.067
585.385	0.347
933.636	0.519
1165.804	0.636
1572.097	0.865

First Resolution Table

Mass	Offset
44.998	0.067
585.385	0.347
933.636	0.519
1165.804	0.636
1223.845	0.669

The masses selected in the reference table do not match the masses in the acquisition method.

Iteration 0

Mass	Offset	Found At	Intensity	Width
44.998	0.067	45.024	63408800	0.706
585.385	0.347	585.340	1949400	0.628
933.636	0.519	933.642	1485000	0.741
1165.804	0.636	1165.800	535970	0.740
1223.845	0.669	1223.834	323930	0.456

Iteration 1

Mass	Offset	Found At	Intensity	Width
44.998	0.067	45.022	66829000	0.697
585.385	0.347	585.383	1483400	0.740
933.636	0.519	933.612	1506000	0.606
1165.804	0.636	1165.807	602828	0.656
1223.845	0.659	1223.838	404170	0.718

Useful resolution optimization.

New Resolution Table

Mass	Offset
44.998	0.067
585.385	0.347
933.636	0.519
1165.804	0.636
1223.845	0.659

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
44.998	45.022	-0.024	0.697	0.003	66829000.00	2.69
585.385	585.383	0.002	0.740	-0.040	1483400.00	-4.52
933.636	933.612	0.024	0.606	0.094	1506000.00	9.48
1165.804	1165.807	-0.003	0.656	0.044	602828.54	18.70
1223.845	1223.838	0.007	0.718	-0.018	404170.48	18.70

Original Calibration Table

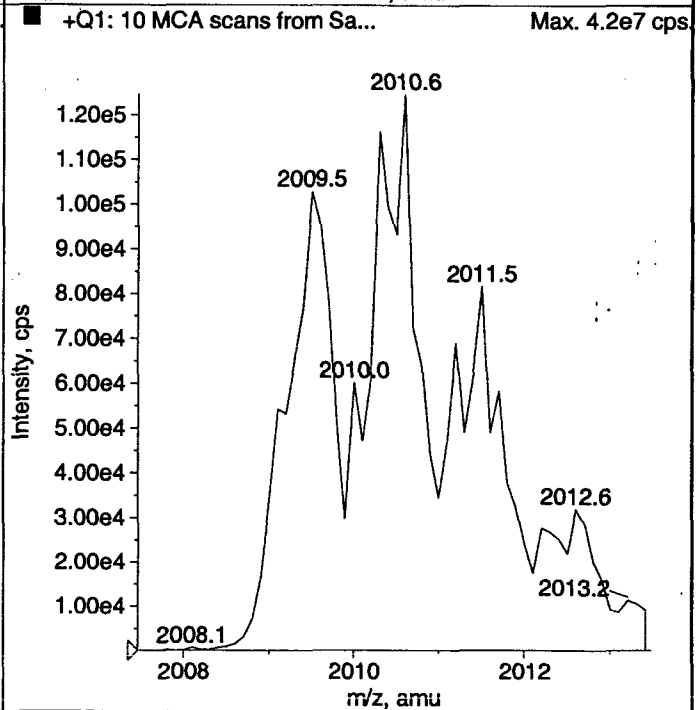
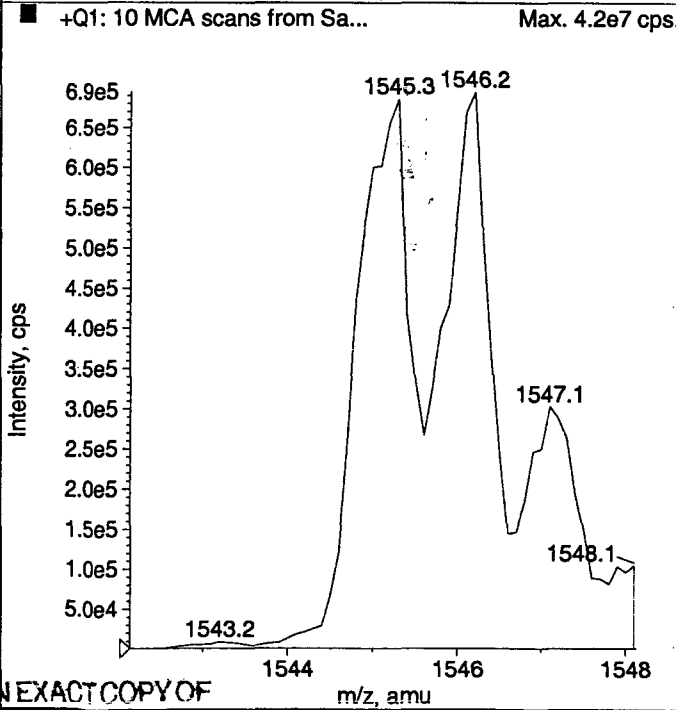
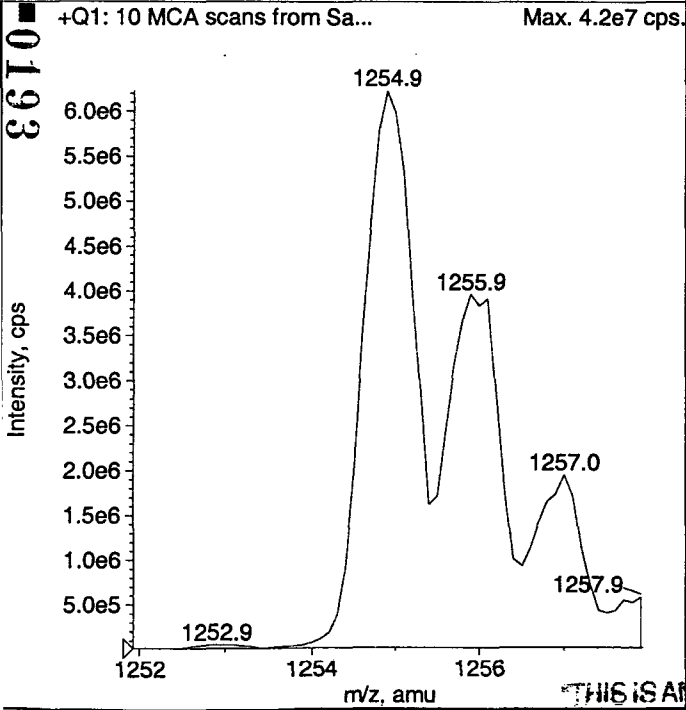
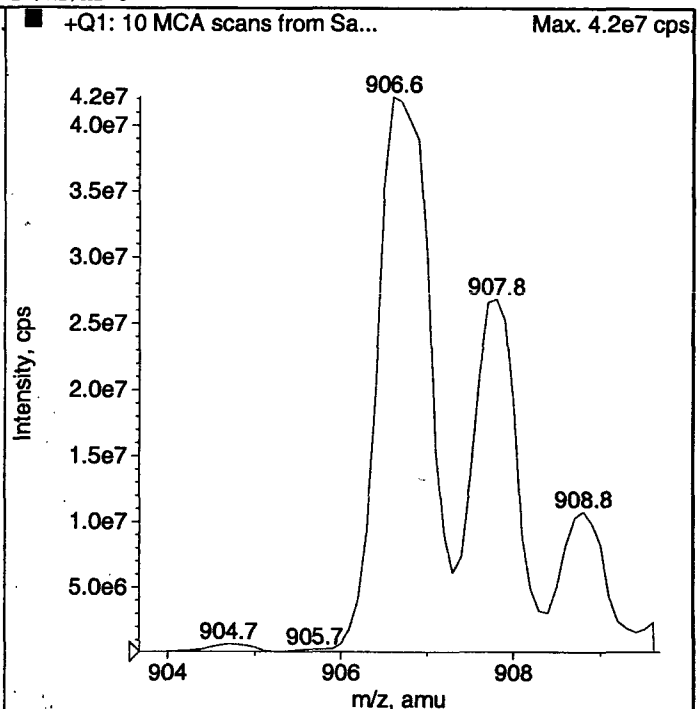
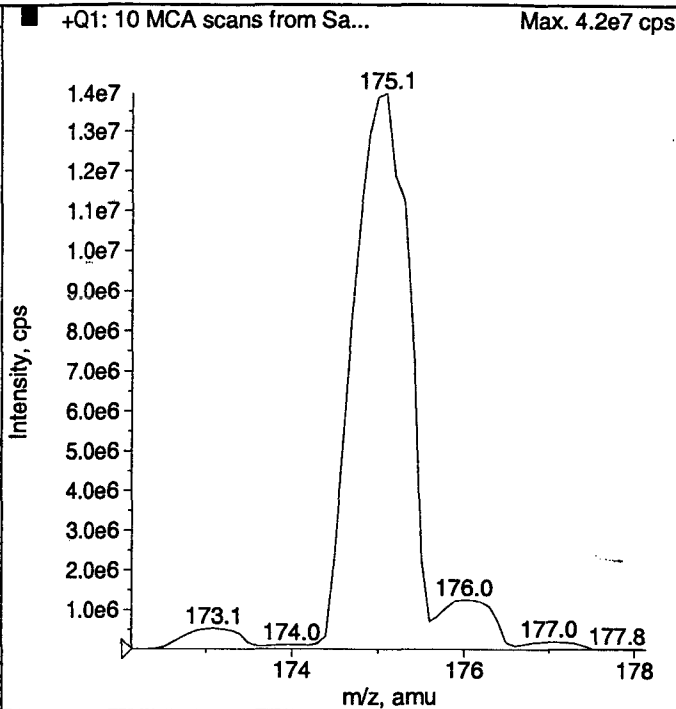
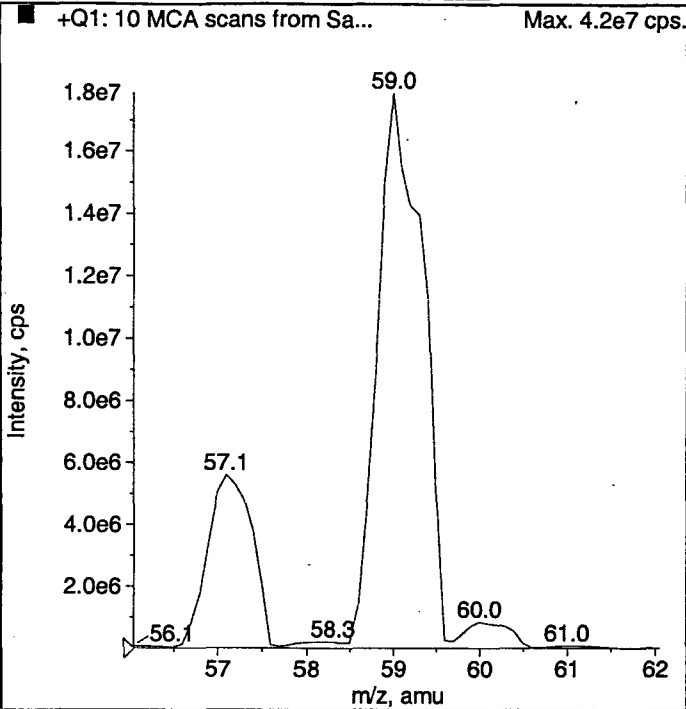
Mass	Dac
44.998	830
585.385	11109
933.636	17735
1165.804	22152
1572.097	29885

New Calibration Table

Mass	Dac
44.998	830
585.385	11109
933.636	17735
1165.804	22152
1223.845	23257

File path of acquired data for resolution optimization :

D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.08.05.14.21.17



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BY BML DATE 08/08/05 Oxygen STUDY NO: P760

Unit Resolution Optimization for Quad 1, Positive

Wednesday, July 06, 2005, 09:07

Method File:

Analyst Data\Projects\API Instrument\Acquisition Methods\Q1 PFG Positive02.dam

Threshold: 200 cps
Search Range: 3.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
59.050	0.070
175.133	0.125
906.673	0.398
1254.925	0.525
1545.134	0.627
2010.469	0.796

First Resolution Table

Mass	Offset
59.050	0.070
175.133	0.125
906.673	0.398
1254.925	0.525
1545.134	0.627
2010.469	0.796

Iteration 0

Mass	Offset	Found At	Intensity	Width
59.050	0.070	59.118	21885172	0.660
175.133	0.125	175.091	11684854	0.602
906.673	0.398	906.689	54335849	0.804
1254.925	0.525	1254.931	6043227	0.717
1545.134	0.627	1545.112	677955	0.710
2010.469	0.796	2010.526	129404	0.659

Iteration 1

Mass	Offset	Found At	Intensity	Width
59.050	0.070	59.123	21355387	0.650
175.133	0.125	175.106	10476343	0.612
906.673	0.408	906.799	40326793	0.560
1254.925	0.525	1254.921	6533429	0.706
1545.134	0.627	1545.122	743543	0.702
2010.469	0.796	2010.508	103712	0.669

Iteration 2

Mass	Offset	Found At	Intensity	Width
59.050	0.070	59.124	21251077	0.656
175.133	0.125	175.097	11943698	0.592
906.673	0.403	906.747	46695295	0.648
1254.925	0.525	1254.922	7266428	0.684
1545.134	0.627	1545.188	772018	0.761
2010.469	0.796	2010.481	153067	0.560

Iteration 3

Mass	Offset	Found At	Intensity	Width
59.050	0.070	59.128	21253314	0.638
175.133	0.115	175.062	13369064	0.754
906.673	0.403	906.743	45978240	0.655
1254.925	0.525	1254.911	6419930	0.701
1545.134	0.627	1546.133	674595	0.701
2010.469	0.786	2010.549	137585	1.100

Iteration 4

Mass	Offset	Found At	Intensity	Width
59.050	0.070	59.103	17871271	0.641
175.133	0.115	175.044	13953241	0.768
906.673	0.403	906.732	42165943	0.653
1254.925	0.525	1254.922	6226432	0.705
1545.134	0.627	1546.117	694672	0.689
2010.469	0.791	2010.471	124604	0.608

Successful resolution optimization.

New Resolution Table

Mass	Offset
59.050	0.070
175.133	0.115
906.673	0.403
1254.925	0.525
1545.134	0.627
2010.469	0.791

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
59.050	59.103	-0.053	0.641	0.059	17871271.00	-35.53
175.133	175.044	0.089	0.768	-0.068	13953241.13	19.82
906.673	906.732	-0.059	0.653	0.047	42165943.44	-32.79
1254.925	1254.922	0.003	0.705	-0.005	6226432.63	0.38
1545.134	1546.117	-0.983	0.689	0.011	694672.25	6.34
2010.469	2010.471	-0.002	0.608	0.092	124604.22	18.01

Original Calibration Table

Mass	Dec
59.050	1097

Oxygen STUDY NO. P760

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BY BRK DATE 08/08/05

175.133	3309
906.673	17245
1254.925	23882
1545.134	29413
2010.469	38284

Calibration Table

Mass	Dac
59.050	1098
175.133	3307
906.673	17246
1254.925	23882
1545.134	29432
2010.469	38284

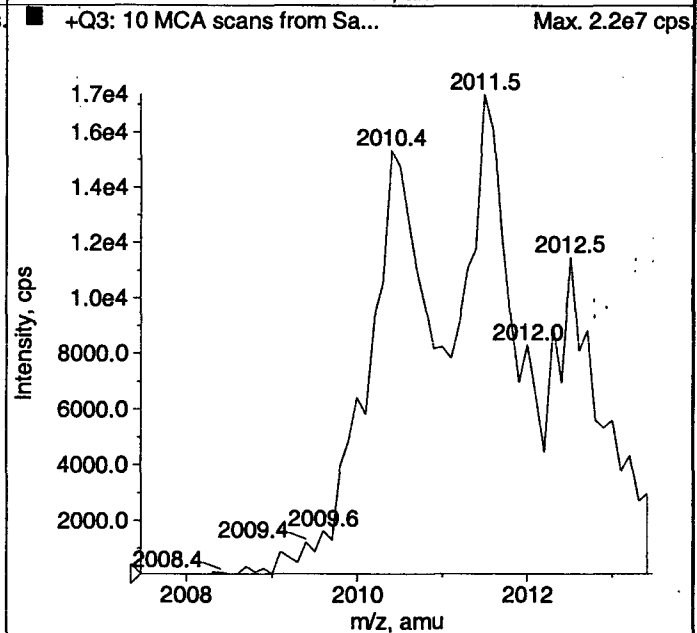
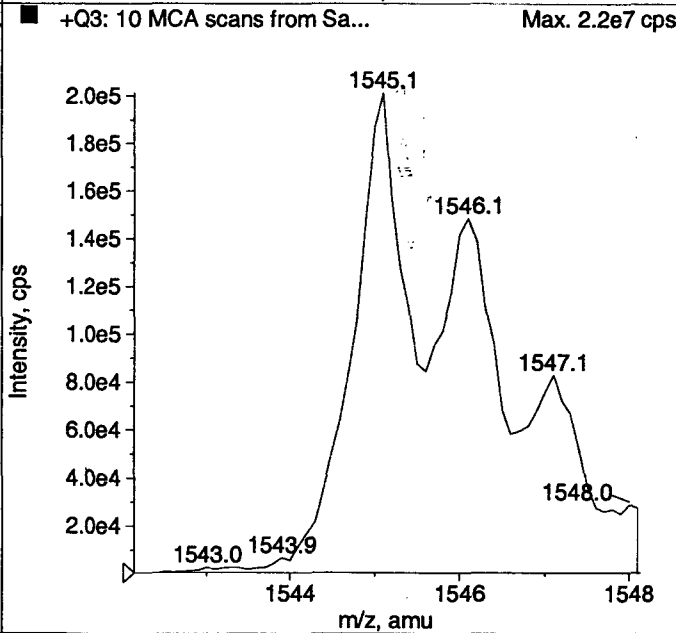
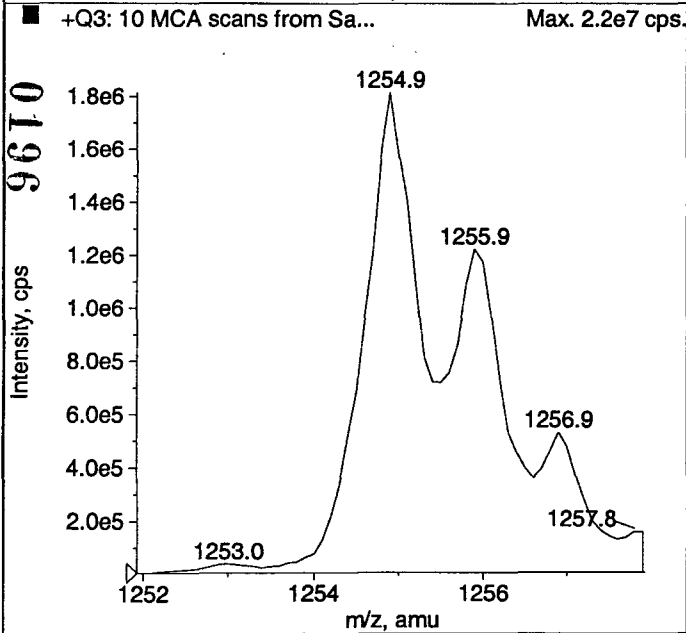
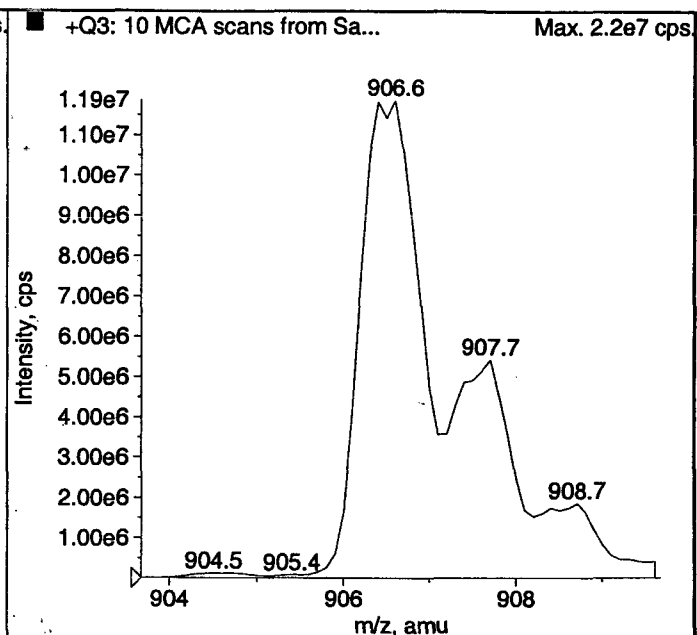
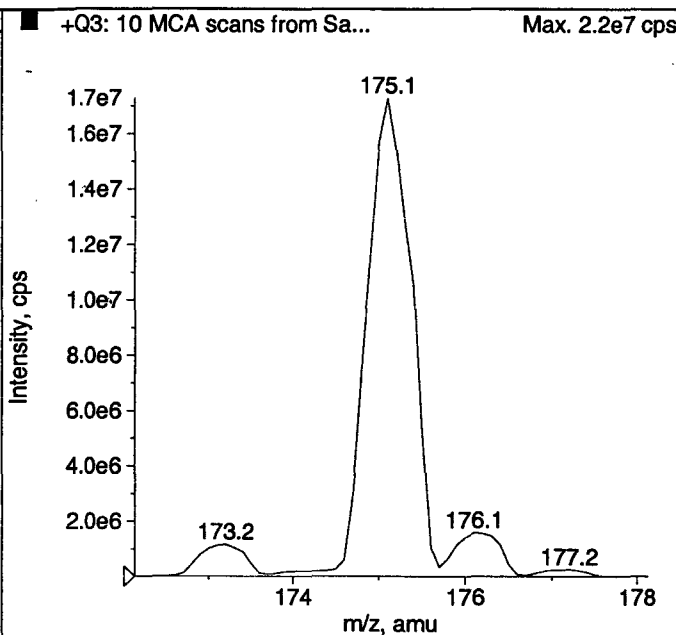
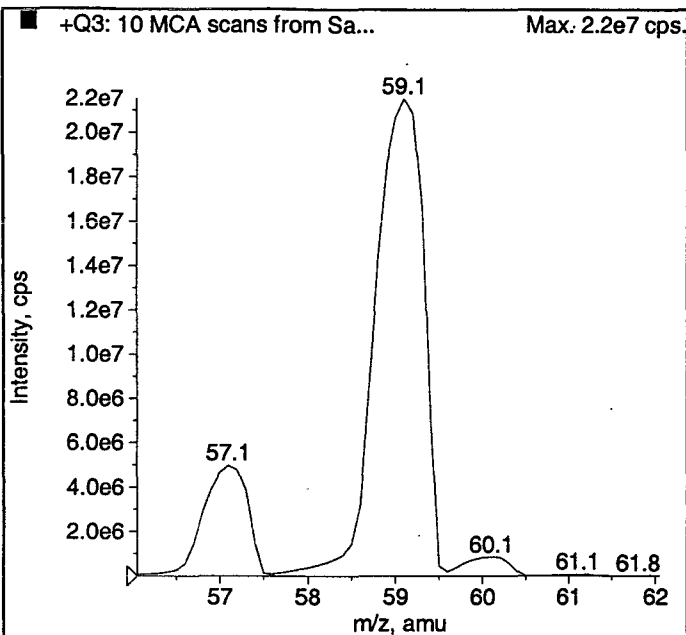
File path of acquired data for resolution optimization :

D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.07.06.09.07.36

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BY BKK DATE 08/08/05

Exygen STUDY NO. P760

Resolution Optimization for Quad 3, Positive

Wednesday, July 06, 2005, 09:34

Method File:

D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q3+ MDNrev1.dam

Threshold: 200 cps
Search Range: 3.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
59.050	0.075
175.133	0.145
906.673	0.556
1254.925	0.733
1545.134	0.896
2010.469	1.174

First Resolution Table

Mass	Offset
59.050	0.075
175.133	0.145
906.673	0.556
1254.925	0.733
1545.134	0.896
2010.469	1.174

Iteration 0

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.069	20566759	0.632
175.133	0.145	175.133	14344251	0.676
906.673	0.556	906.643	6796721	0.499
1254.925	0.733	1254.928	2211097	0.655
1545.134	0.896	1545.095	196123	0.669
2010.469	1.174	2011.495	20282	0.859

Iteration 1

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.069	21780963	0.630
175.133	0.145	175.127	17376357	0.646
906.673	0.546	906.500	11791706	0.765
1254.925	0.733	1254.932	1895884	0.649
1545.134	0.896	1545.092	168529	0.549
2010.469	1.184	2010.654	9668	0.557

Iteration 2

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.060	21244672	0.618
175.133	0.145	175.128	17251612	0.649
906.673	0.546	906.515	11794349	0.773
1254.925	0.733	1254.909	1814349	0.679
1545.134	0.886	1545.085	279468	0.906
2010.469	1.179	2011.575	9600	0.718

Iteration 3

Mass	Offset	Found At	Intensity	Width
59.050	0.075	59.070	21633445	0.626
175.133	0.145	175.130	17317187	0.645
906.673	0.546	906.513	11952339	0.774
1254.925	0.733	1254.916	1741031	0.726
1545.134	0.891	1545.084	200203	0.717
2010.469	1.179	2011.583	11628	0.580

Iteration 4

Mass	Offset	Found At	Intensity	Width
59.050	-0.075	59.069	21515919	0.628
175.133	0.145	175.122	17294312	0.615
906.673	0.546	906.523	11869888	0.784
1254.925	0.733	1254.924	1814801	0.688
1545.134	0.891	1545.080	201402	0.682
2010.469	1.174	2011.527	17375	0.661

Successful resolution optimization.

New Resolution Table

Mass	Offset
59.050	0.075
175.133	0.145
906.673	0.546
1254.925	0.733
1545.134	0.891
2010.469	1.174

Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
59.050	59.069	-0.019	0.628	0.072	21515919.25	-3.65
175.133	175.122	0.011	0.615	0.085	17294312.50	-4.22

906.673	906.523	0.150	0.784	-0.084	11869888.00	54.29
1254.925	1254.924	0.001	0.688	0.012	1814801.31	1.63
1545.134	1545.080	0.054	0.682	0.018	201402.96	20.21
2010.469	2011.527	-1.058	0.661	0.039	17375.63	7.78

Original Calibration Table

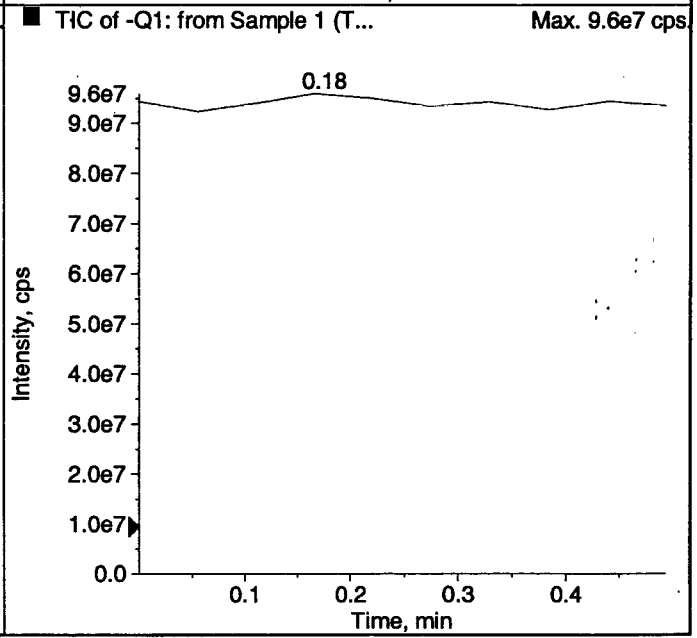
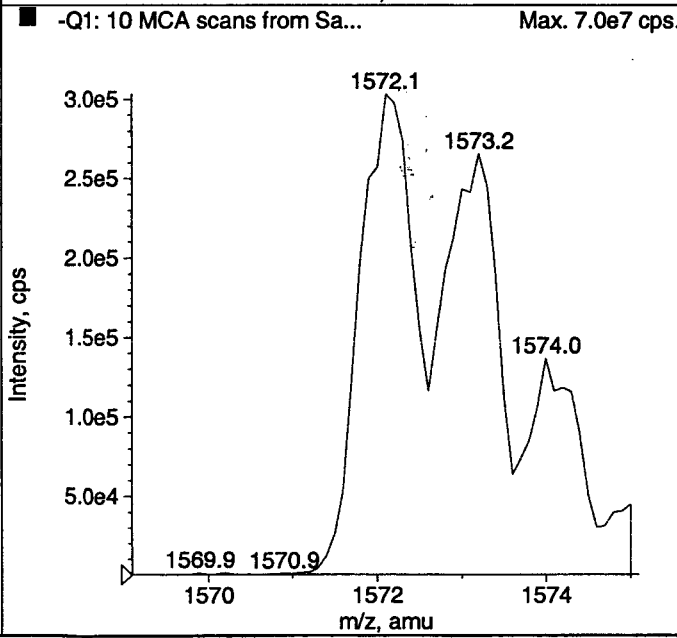
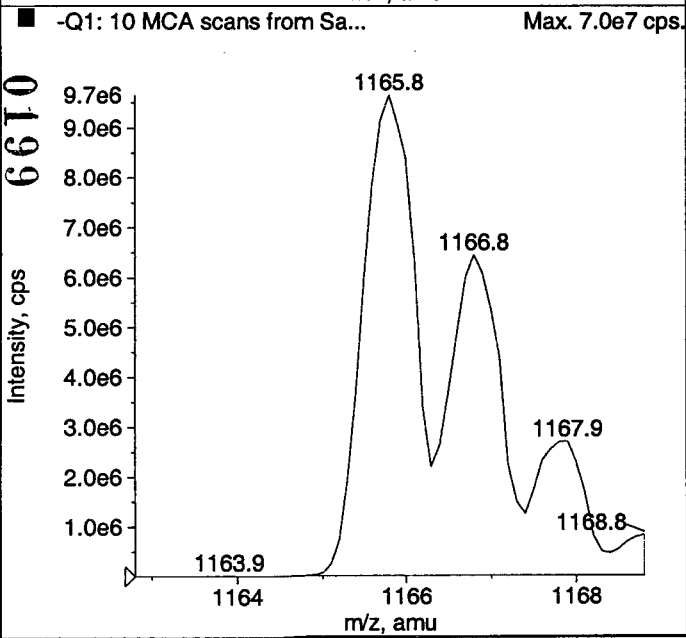
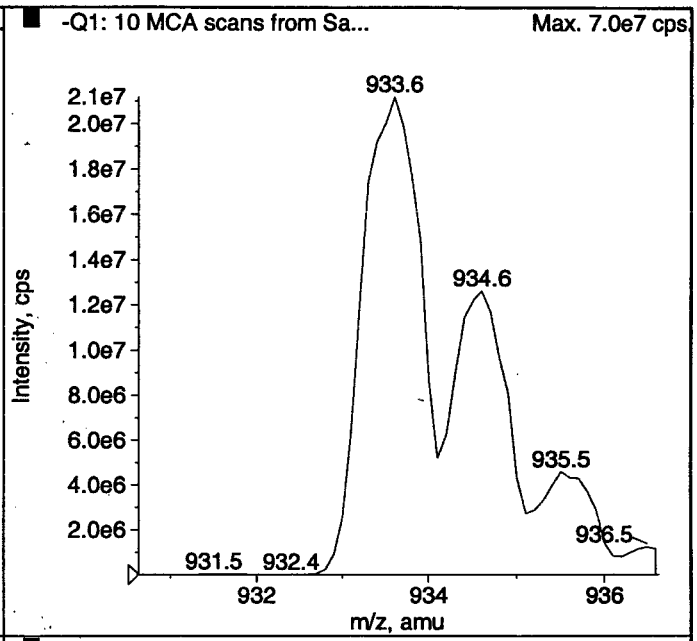
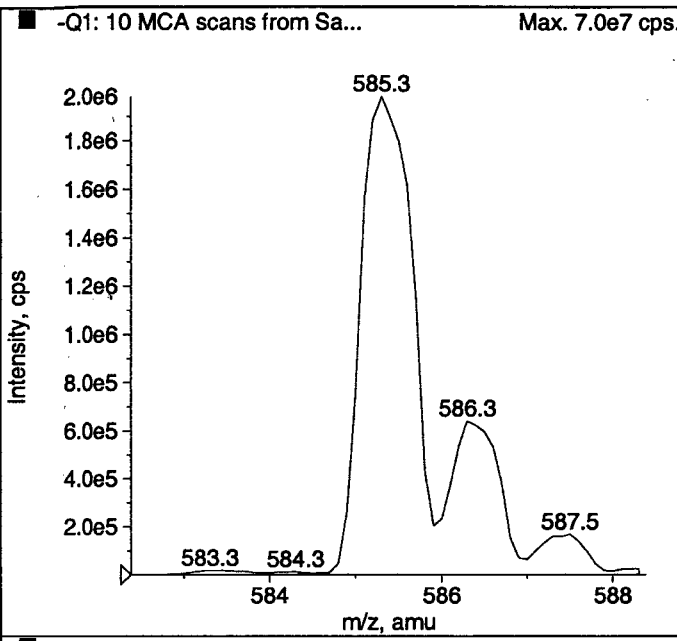
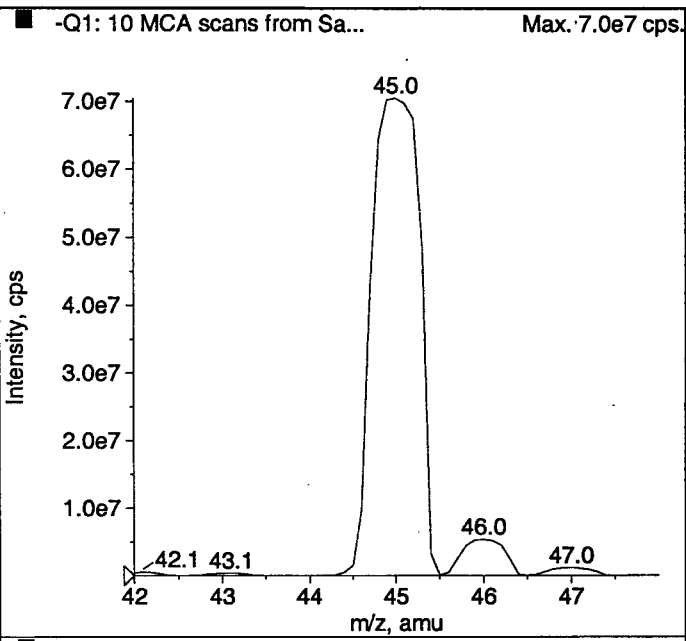
Mass	Dac
59.050	1096
175.133	3305
906.673	17227
1254.925	23853
1545.134	29376
2010.469	38216

New Calibration Table

Mass	Dac
59.050	1096
175.133	3305
906.673	17224
1254.925	23853
1545.134	29375
2010.469	38236

File path of acquired data for resolution optimization :

D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.07.06.09.34.12



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BY BMC DATE 08/08/05

Oxygen STUDY NO: P766

Resolution Optimization for Quad 1, Negative

Wednesday, July 06, 2005, 10:14

Method File:
D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q1 neg ppg pc01.dam
Threshold: 200 cps
Search Range: 3.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
44.998	0.055
179.092	0.108
295.176	0.154
585.385	0.269
933.636	0.392
1223.845	0.505

First Resolution Table

Mass	Offset
44.998	0.055
585.385	0.269
933.636	0.392
1165.804	0.482
1572.097	0.640

The masses selected in the reference table do not match the masses in the acquisition method.

Iteration 0

Mass	Offset	Found At	Intensity	Width
44.998	0.055	45.011	70204800	0.650
585.385	0.269	585.363	2021600	0.676
933.636	0.392	933.564	21174300	0.772
1165.804	0.482	1165.797	9796079	0.701
1572.097	0.640	1572.181	180063	0.542

Iteration 1

Mass	Offset	Found At	Intensity	Width
44.998	0.055	45.011	70470700	0.652
585.385	0.269	585.360	1984700	0.694
933.636	0.392	933.572	21210000	0.792
1165.804	0.482	1165.809	9657925	0.701
1572.097	0.630	1572.115	303297	0.769

Successful resolution optimization.

New Resolution Table

Mass	Offset
44.998	0.055
585.385	0.269
933.636	0.392
1165.804	0.482
1572.097	0.630

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
44.998	45.011	-0.013	0.652	0.048	70470700.00	0.02
585.385	585.360	0.025	0.694	0.006	1984700.00	-2.82
933.636	933.572	0.064	0.792	-0.092	21210000.00	1.18
1165.804	1165.809	-0.005	0.701	-0.001	9657925.25	1.18
1572.097	1572.115	-0.018	0.769	-0.069	303297.22	1.18

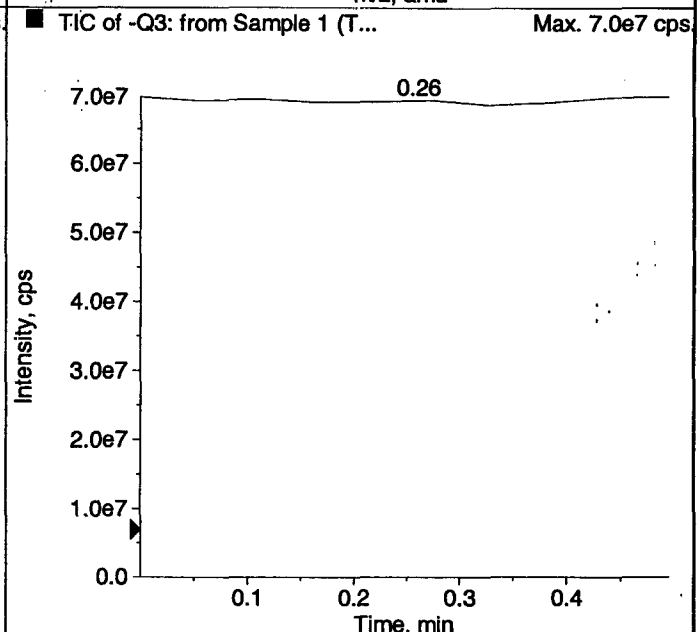
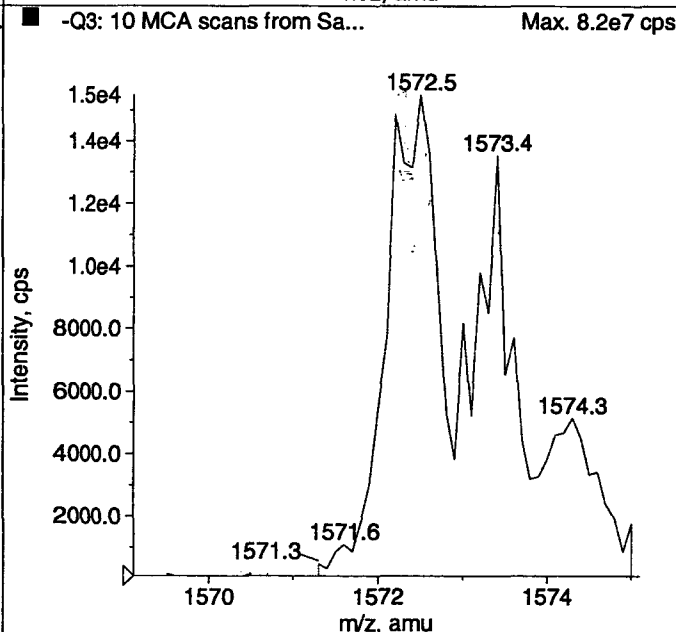
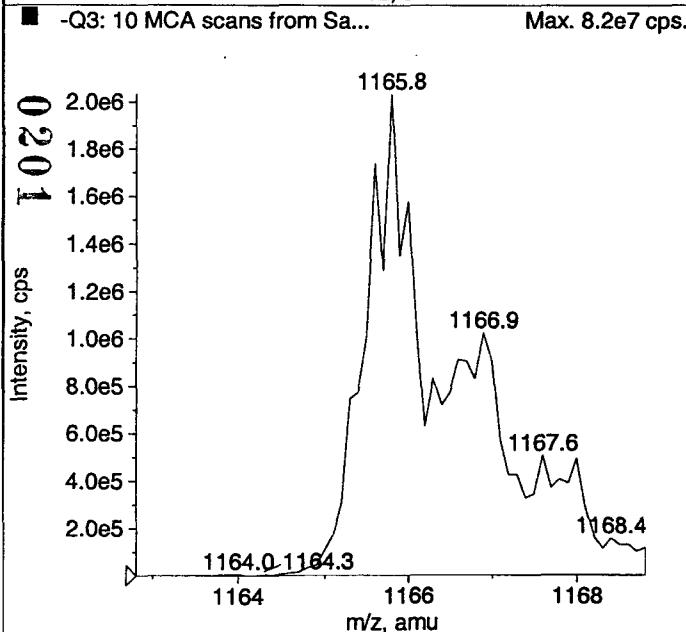
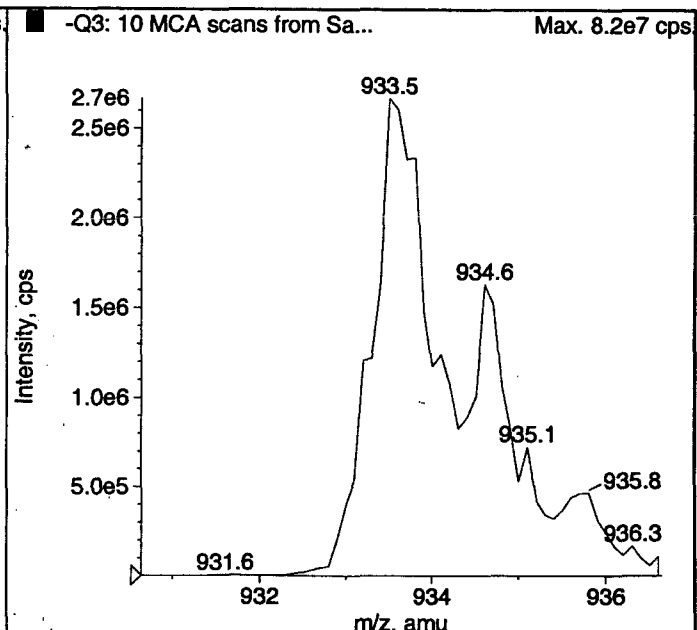
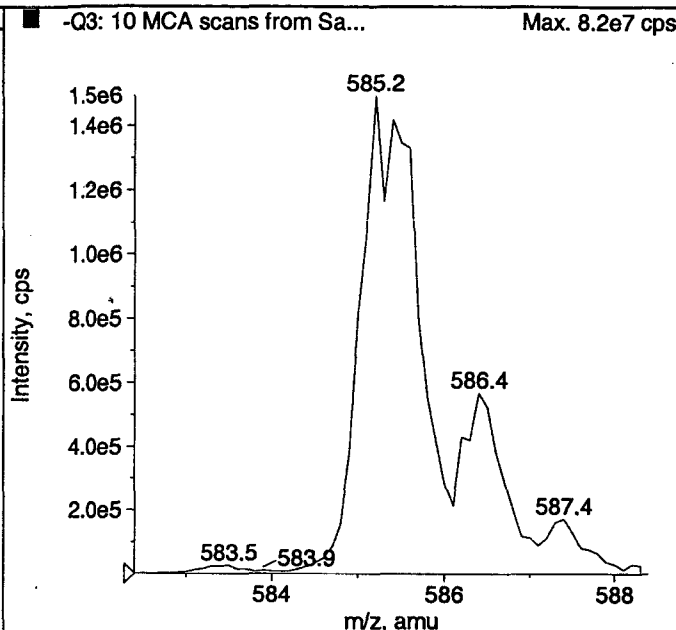
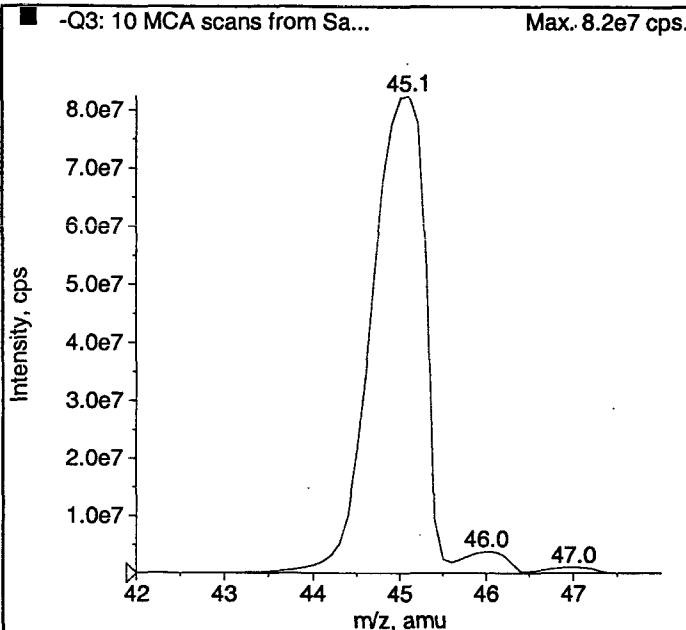
Original Calibration Table

Mass	Dac
44.998	832
585.385	11123
933.636	17760
1165.804	22184
1572.097	29928

New Calibration Table

Mass	Dac
44.998	832
585.385	11123
933.636	17759
1165.804	22184
1572.097	29928

File path of acquired data for resolution optimization :
D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.07.06.10.14.03



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BY BAL DATE 08/08/05

Exygen STUDY NO: P760

Resolution Optimization for Quad 3, Negative

Wednesday, July 06, 2005, 10:22

Method File:
D:\Analyst Data\Projects\API Instrument\Acquisition Methods\Q3- MDN.dam

Threshold: 200 cps
Search Range: 3.0 amu
Width At (percent max): 50.0
Target Peak Width: 0.70
Peak Width Tolerance: 0.100
Max Iterations: 20

Original Resolution Table

Mass	Offset
44.998	0.067
585.385	0.347
933.636	0.519
1165.804	0.631
1223.845	0.666

First Resolution Table

Mass	Offset
44.998	0.067
585.385	0.347
933.636	0.519
1165.804	0.631
1572.097	0.875

The masses selected in the reference table do not match the masses in the acquisition method.

Iteration 0

Mass	Offset	Found At	Intensity	Width
44.998	0.067	45.014	82496500	0.687
585.385	0.347	585.362	1495600	0.739
933.636	0.519	933.630	2670300	0.620
1165.804	0.631	1165.792	2033008	0.601
1572.097	0.875	1572.412	15491	0.638

Successful resolution optimization.

New Resolution Table

Mass	Offset
44.998	0.067
585.385	0.347
933.636	0.519
1165.804	0.631
1572.097	0.875

Mass Calibration Results

Mass	Found At	Mass Shift	Peak Width	FW Shift	Intensity	Intensity Diff
44.998	45.014	-0.016	0.687	0.013	82496500.00	16.30
585.385	585.362	0.023	0.739	-0.039	1495600.00	19.30
933.636	933.630	0.006	0.620	0.080	2670300.00	58.05
1165.804	1165.792	0.012	0.601	0.099	2033008.95	58.05
1572.097	1572.412	-0.315	0.638	0.062	15491.82	58.05

Original Calibration Table

Mass	Dac
44.998	831
585.385	11110
933.636	17736
1165.804	22153
1572.097	29882

New Calibration Table

Mass	Dac
44.998	831
585.385	11110
933.636	17736
1165.804	22153
1572.097	29888

File path of acquired data for resolution optimization :
D:\Analyst Data\Projects\API Instrument\Data\AutoRes.2005.07.06.10.22.13

Note To File

NOTE TO FILE

Date: 07/28/05

Exygen Research Project/Study #: P760 and P1131

Protocol #: P0000760 and P0001131

NOTE:

Vegetation samples C0049876 – C0049885 and C0073483 were pulled from Freezer 34 (IN0000713) at 0829 on 07/06/05. Samples were weighed on Balance 20 and capped tightly. After weighing, the samples to be used for the extraction were transferred to Freezer 34 at 1225.

The samples remained in the freezer until the day of extraction, 07/12/05.

Vegetation samples C0049886 – C0049890 and C0073483 were pulled from Freezer 34 (IN0000713) at 0829 on 07/06/05. Samples were weighed on Balance 20 and capped tightly. After weighing, the samples to be used for the extraction were transferred to Freezer 34 at 1225.

The samples remained in the freezer until the day of extraction, 07/13/05.

Vegetation samples C0049891 – C0049895 and C0073483 were pulled from Freezer 34 (IN0000713) at 1341 on 07/06/05. Samples were weighed on Balance 20 and capped tightly. After weighing, the samples to be used for the extraction were transferred to Freezer 34 at 1553.

The samples remained in the freezer until the day of extraction, 07/14/05.

Vegetation samples C0049896 – C0049897 and C0073483 were pulled from Freezer 34 (IN0000713) at 1341 on 07/06/05. Samples were weighed on Balance 20 and capped tightly. After weighing, the samples to be used for the extraction were transferred to Freezer 34 at 1553.

The samples remained in the freezer until the day of extraction, 07/15/05.


Vegetation samples C0050021 – C0050029 and C0073483 were pulled from Freezer 34 (IN0000713) at 1423 on 07/27/05. Samples were weighed on Balance 20 and capped tightly. After weighing, the samples to be used for the extraction were transferred to Freezer 34 at 1610.

The samples remained in the freezer until the day of extraction, 07/28/05.

NOTE TO FILE

Samples C0049876 – C0049897 were weighed by Carlyle Horrell and verified by Brittany Kravets.

All spiking was performed on the day of extraction after the samples were pulled from the freezer.

Signature: 

Date: 07/28/05

NOTE TO FILE

Date: 08/16/05

Exygen Research Project/Study #: P760 and P1131

Protocol #: P0000760 and P0001131

NOTE:

The method for extraction of fish (V0001783) and the method for extraction of vegetation (V0001784) require samples to be evaporated on a Rotovap. Procedure indicates that, when using a Rotovap, the temperature of the water bath should not exceed 40°C. Because temperature settings on the water baths are not accurate, water baths were left off during evaporation and the water was at room temperature for all fish and vegetation samples.

Signature: _____



Date: _____

08/16/05

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BY BAK DATE 08/17/05



3058 Research Drive
State College, PA 16801

Phone: 814-272-1039
Fax: 814-231-1580

NOTE TO FILE

Date: 09/21/05

Exygen Research Project/Study #: P760 and P1131

Protocol #: P0000760 and P0001131

NOTE:

The extraction sheets show that vegetation samples C0049876 – C0049890 were transferred to freezer 34 at 1225 on 07/06/05. ExyLIMS shows that these samples were transferred to freezer 30. The extraction sheets are correct. The samples were, in actuality, transferred to freezer 34.

Signature: 

Date: 09/21/05

Overall Summary

Summary of PFOA in Vegetation Samples

Exygen ID	Client Sample ID	C8 Acid PFOA Perfluorooctanoic Acid	
		Analyte Found (ppb, ng/g) Wet Weight	Assessed Accuracy (+/- %)
C0049876	DF06-V01-AVP001-0-041007	90.0	60
C0049876 Rep	DF06-V01-AVP001-0-041007*	78.4	60
C0049877	DF06-V01-PAP001-0-041007	NR	---
C0049877 Rep	DF06-V01-PAP001-0-041007*	NR	---
C0049878	DF06-V01-RCP001-0-041007	238	30
C0049878 Rep	DF06-V01-RCP001-0-041007*	255	30
C0049879	DF06-V02-AVP001-0-041007	46.8	50
C0049879 Rep	DF06-V02-AVP001-0-041007*	37.7	50
C0049880	DF06-V02-PAP001-0-041007	440	40
C0049880 Rep	DF06-V02-PAP001-0-041007*	472	40
C0049881	DF06-V02-RCP001-0-041007	123	60
C0049881 Rep	DF06-V02-RCP001-0-041007*	138	60
C0049882	DF09-V01-AVP001-0-041007	10.0	50
C0049882 Rep	DF09-V01-AVP001-0-041007*	9.56	50
C0049883	DF09-V01-PAP001-0-041007	62.0	50
C0049883 Rep	DF09-V01-PAP001-0-041007*	64.8	50
C0049884	DF09-V01-RCP001-0-041007	50.4	30
C0049884 Rep	DF09-V01-RCP001-0-041007*	48.8	30
C0049885	DF09-V02-RCP001-0-041007	150	30
C0049885 Rep	DF09-V02-RCP001-0-041007*	165	30
C0049886	DF09-V02-PAP001-0-041007	448	40
C0049886 Rep	DF09-V02-PAP001-0-041007*	464	40
C0049887	DF8b-V01-PAP001-0-041007	912	60
C0049887 Rep	DF8b-V01-PAP001-0-041007*	912	60
C0049888	DF8b-V01-RCP001-0-041007	212	30
C0049888 Rep	DF8b-V01-RCP001-0-041007*	204	30
C0049889	DF8b-V02-PAP001-0-041007	944	60
C0049889 Rep	DF8b-V02-PAP001-0-041007*	904	60
C0049890	DF8b-V02-RCP001-0-041007	236	50
C0049890 Rep	DF8b-V02-RCP001-0-041007*	225	50
C0049891	DF14-V01-AVP001-0-041007	4.76	30
C0049891 Rep	DF14-V01-AVP001-0-041007*	6.04	30
C0049892	DF14-V01-PAP001-0-041007	9.36	60
C0049892 Rep	DF14-V01-PAP001-0-041007*	9.08	60
C0049893	DF14-V02-AVP001-0-041007	5.52	40
C0049893 Rep	DF14-V02-AVP001-0-041007*	5.52	40
C0049894	DF14-V02-PAP001-0-041007	11.3	40
C0049894 Rep	DF14-V02-PAP001-0-041007*	11.4	40
C0049895	DBKG-V01-AVP001-0-041007	3.70	40
C0049895 Rep	DBKG-V01-AVP001-0-041007*	2.85	40
C0049896	DBKG-V01-PAP001-0-041007	3.92	30
C0049896 Rep	DBKG-V01-PAP001-0-041007*	4.68	30
C0049897	DBKG-V01-RCP001-0-041007	2.00	30
C0049897 Rep	DBKG-V01-RCP001-0-041007*	1.97	30

*Laboratory Duplicate

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

NQ = Not quantifiable = Measured concentration between 0.2 ng/g and the Limit of Quantitation (LOQ) which is 0.4 ng/g.

NR = Not reported due to quality control result failures.

Exygen STUDY NO. *AP760*
AMB 09/22/05

Summary of PFOA in Vegetation Samples (continued)

Exygen ID	Client Sample ID	C8 Acid PFOA	
		Analyte Found (ppb, ng/g) Wet Weight	Perfluorooctanoic Acid Assessed Accuracy (+/- %)
C0050021	DF09-V01-SSP001-0-041007	112	50
C0050021 Rep	DF09-V01-SSP001-0-041007*	108	50
C0050022	DF09-V02-UGP001-0-041007	388	30
C0050022 Rep	DF09-V02-UGP001-0-041007*	386	30
C0050023	DF06-V01-SSP001-0-041007	245	30
C0050023 Rep	DF06-V01-SSP001-0-041007*	258	30
C0050024	DF06-V02-SSP001-0-041007	NR	---
C0050024 Rep	DF06-V02-SSP001-0-041007*	NR	---
C0050025	DF8b-V01-UGP001-0-041007	580	50
C0050025 Rep	DF8b-V01-UGP001-0-041007*	632	50
C0050026	DF8b-V02-UGP001-0-041007	369	60
C0050026 Rep	DF8b-V02-UGP001-0-041007*	362	60
C0050027	DF14-V01-SSP001-0-041007	NR	---
C0050027 Rep	DF14-V01-SSP001-0-041007*	NR	---
C0050028	DF14-V02-SSP001-0-041007	NR	---
C0050028 Rep	DF14-V02-SSP001-0-041007*	NR	---
C0050029	DBKG-V01-SSP001-0-041007	2.53	50
C0050029 Rep	DBKG-V01-SSP001-0-041007*	2.51	50

*Laboratory Duplicate

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

NQ = Not quantifiable = Measured concentration between 0.2 ng/g and the Limit of Quantitation (LOQ) which is 0.4 ng/g.

NR = Not reported due to quality control result failures.

Exygen STUDY NO. P760
AMB 09/22/05

Laboratory Matrix Spike Recovery Summary for PFOA in Vegetation Samples

Sample Description	C8 Acid PFOA			
	Amount Spiked (ng/g)	Amt Found in Sample (ng/g)	Amount Recovered (ng/g)	Recovery (%)
DF06-V01-AVP001-0-041007 (C0049876 Spk C, 10 ppb)	10	90.0	98.8	*
DF06-V01-AVP001-0-041007 (C0049876 Spk D, 100 ppb)	100	90.0	129	39
DF06-V01-PAP001-0-41007 (C0049877 Spk E, 10 ppb)	10	NR	NR	NR
DF06-V01-PAP001-0-041007 (C0049877 Spk F, 100 ppb)	100	NR	NR	NR
DF06-V01-RCP001-0-041007 (C0049878 Spk G, 10 ppb)	10	238	265	*
DF06-V01-RCP001-0-041007 (C0049878 Spk H, 100 ppb)	100	238	317	79
DF06-V02-AVP001-0-041007 (C0049879 Spk I, 10 ppb)	10	46.8	49.2	*
DF06-V02-AVP001-0-041007 (C0049879 Spk J, 100 ppb)	100	46.8	98.8	52
DF06-V02-PAP001-0-041007 (C0049880 Spk K, 10 ppb)	10	440	496	*
DF06-V02-PAP001-0-041007 (C0049880 Spk L, 100 ppb)	100	440	540	*
DF06-V02-RCP001-0-041007 (C0049881 Spk C, 10 ppb)	10	123	128	*
DF06-V02-RCP001-0-041007 (C0049881 Spk D, 100 ppb)	100	123	168	45
DF09-V01-AVP001-0-041007 (C0049882 Spk E, 10 ppb Spike)	10	10.0	15.8	58
DF09-V01-AVP001-0-041007 (C0049882 Spk F, 100 ppb Spike)	100	10.0	71.2	61
DF09-V01-PAP001-0-041007 (C0049883 Spk G, 10 ppb Spike)	10	62.0	70	*
DF09-V01-PAP001-0-041007 (C0049883 Spk H, 100 ppb Spike)	100	62.0	116	54
DF09-V01-RCP001-0-041007 (C0049884 Spk I, 10 ppb Spike)	10	50.4	58.0	*
DF09-V01-RCP001-0-041007 (C0049884 Spk J, 100 ppb Spike)	100	50.4	133	83
DF09-V02-RCP001-0-041007 (C0049885 Spk K, 10 ppb Spike)	10	150	165	*
DF09-V02-RCP001-0-041007 (C0049885 Spk L, 100 ppb Spike)	100	150	246	96

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

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

NQ = Not quantifiable = Measured concentration between 0.2 ng/g and the Limit of Quantitation (LOQ) which is 0.4 ng/g.

NR = Not reported due to quality control result failures.

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

Oxygen STUDY NO. P760
AMB 09/22/05

Laboratory Matrix Spike Recovery Summary for PFOA in Vegetation Samples (continued)

Sample Description	C8 Acid PFOA			
	Amount Spiked (ng/g)	Amt Found in Sample (ng/g)	Amount Recovered (ng/g)	Recovery (%)
DF09-V02-PAP001-0-041007 (C0049886 Spk C, 10 ppb Spike)	10	448	416	*
DF09-V02-PAP001-0-041007 (C0049886 Spk D, 100 ppb Spike)	100	448	540	*
DF8b-V01-PAP001-0-041007 (C0049887 Spk E, 10 ppb Spike)	10	912	996	*
DF8b-V01-PAP001-0-041007 (C0049887 Spk F, 100 ppb Spike)	100	912	1060	*
DF8b-V01-RCP001-0-041007 (C0049888 Spk G, 10 ppb Spike)	10	212	212	*
DF8b-V01-RCP001-0-041007 (C0049888 Spk H, 100 ppb Spike)	100	212	298	86
DF8b-V02-PAP001-0-041007 (C0049889 Spk I, 10 ppb Spike)	10	944	948	*
DF8b-V02-PAP001-0-041007 (C0049889 Spk J, 100 ppb Spike)	100	944	1044	*
DF8b-V02-RCP001-0-041007 (C0049890 Spk K, 10 ppb Spike)	10	236	236	*
DF8b-V02-RCP001-0-041007 (C0049890 Spk L, 100 ppb Spike)	100	236	384	148
DF14-V01-AVP001-0-041007 (C0049891 Spk C, 10 ppb Spike)	10	4.76	12.6	78
DF14-V01-AVP001-0-041007 (C0049891 Spk D, 100 ppb Spike)	100	4.76	72.0	67
DF14-V01-PAP001-0-041007 (C0049892 Spk E, 10 ppb Spike)	10	9.36	13.8	44
DF14-V01-PAP001-0-041007 (C0049892 Spk F, 100 ppb Spike)	100	9.36	93.6	84
DF14-V02-AVP001-0-041007 (C0049893 Spk G, 10 ppb Spike)	10	5.52	12.2	67
DF14-V02-AVP001-0-041007 (C0049893 Spk H, 100 ppb Spike)	100	5.52	79.2	74
DF14-V02-PAP001-0-041007 (C0049894 Spk I, 10 ppb Spike)	10	11.3	18.2	69
DF14-V02-PAP001-0-041007 (C0049894 Spk J, 100 ppb Spike)	100	11.3	95.2	84
DBKG-V01-AVP001-0-041007 (C0049895 Spk K, 10 ppb Spike)	10	3.70	10.4	67
DBKG-V01-AVP001-0-041007 (C0049895 Spk L, 100 ppb Spike)	100	3.70	84.8	81

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

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

NQ = Not quantifiable = Measured concentration between 0.2 ng/g and the Limit of Quantitation (LOQ) which is 0.4 ng/g.

NR = Not reported due to quality control result failures.

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

Laboratory Matrix Spike Recovery Summary for PFOA in Vegetation Samples (continued)

Sample Description	C8 Acid PFOA			
	Amount Spiked (ng/g)	Amt Found in Sample (ng/g)	Amount Recovered (ng/g)	Recovery (%)
DBKG-V01-PAP001-0-041007 (C0049896 Spk C, 10 ppb Spike)	10	3.92	11.5	76
DBKG-V01-PAP001-0-041007 (C0049896 Spk D, 100 ppb Spike)	100	3.92	80.0	76
DBKG-V01-RCP001-0-041007 (C0049897 Spk E, 10 ppb Spike)	10	2.00	9.04	70
DBKG-V01-RCP001-0-041007 (C0049897 Spk F, 100 ppb Spike)	100	2.00	78.0	76
DF09-V01-SSP001-0-041007 (C0050021 Spk C, 10 ppb)	10	112	116	*
DF09-V01-SSP001-0-041007 (C0050021 Spk D, 100 ppb)	100	112	163	51
DF09-V02-UGP001-0-041007 (C0050022 Spk E, 10 ppb)	10	388	366	*
DF09-V02-UGP001-0-041007 (C0050022 Spk F, 100 ppb)	100	388	464	76
DF06-V01-SSP001-0-041007 (C0050023 Spk G, 10 ppb)	10	245	260	*
DF06-V01-SSP001-0-041007 (C0050023 Spk H, 100 ppb)	100	245	315	70
DF06-V02-SSP001-0-041007 (C0050024 Spk I, 10 ppb)	10	NR	NR	NR
DF06-V02-SSP001-0-041007 (C0050024 Spk J, 100 ppb)	100	NR	NR	NR
DF8b-V01-UGP001-0-041007 (C0050025 Spk K, 10 ppb)	10	580	824	*
DF8b-V01-UGP001-0-041007 (C0050025 Spk L, 100 ppb)	100	580	848	*
DF8b-V02-UGP001-0-041007 (C0050026 Spk C, 10 ppb)	10	369	364	*
DF8b-V02-UGP001-0-041007 (C0050026 Spk D, 100 ppb)	100	369	416	47
DF14-V01-SSP001-0-041007 (C0050027 Spk E, 10 ppb)	10	NR	NR	NR
DF14-V01-SSP001-0-041007 (C0050027 Spk F, 100 ppb)	100	NR	NR	NR
DF14-V02-SSP001-0-041007 (C0050028 Spk G, 10 ppb)	10	NR	NR	NR
DF14-V02-SSP001-0-041007 (C0050028 Spk H, 100 ppb)	100	NR	NR	NR

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

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

NQ = Not quantifiable = Measured concentration between 0.2 ng/g and the Limit of Quantitation (LOQ) which is 0.4 ng/g.

NR = Not reported due to quality control result failures.

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

Laboratory Matrix Spike Recovery Summary for PFOA in Vegetation Samples (continued)

C8 Acid PFOA				
Sample Description	Amount Spiked (ng/g)	Amt Found in Sample (ng/g)	Amount Recovered (ng/g)	Recovery (%)
DBKG-V01-SSP001-0-041007 (C0050029 Spk I, 10 ppb)	10	2.53	7.92	54
DBKG-V01-SSP001-0-041007 (C0050029 Spk J, 100 ppb)	100	2.53	85.2	83
			Average:	71
			Standard Deviation:	20

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

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

NQ = Not quantifiable = Measured concentration between 0.2 ng/g and the Limit of Quantitation (LOQ) which is 0.4 ng/g.

NR = Not reported due to quality control result failures.

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

Oxygen STUDY NO. P760

AMB 09/22/05

Surrogate Spike Recovery Summary for ¹³C-PFOA in Vegetation Samples

Exygen ID	Sample Description	¹³ C-PFOA		Recovery (%)
		Amount Spiked (ng/g)	Amount Recovered (ng/g)	
C0049876	DF06-V01-AVP001-0-041007	10	4.16	42
C0049876 Rep	DF06-V01-AVP001-0-041007	10	4.16	42
C0049876 Spk C	DF06-V01-AVP001-0-041007	10	4.72	47
C0049876 Spk D	DF06-V01-AVP001-0-041007	100	64.4	64
C0049877	DF06-V01-PAP001-0-041007	10	NR	NR
C0049877 Rep	DF06-V01-PAP001-0-041007	10	NR	NR
C0049877 Spk E	DF06-V01-PAP001-0-041007	10	NR	NR
C0049877 Spk F	DF06-V01-PAP001-0-041007	100	NR	NR
C0049878	DF06-V01-RCP001-0-041007	10	4.04	40
C0049878 Rep	DF06-V01-RCP001-0-041007	10	3.82	38
C0049878 Spk G	DF06-V01-RCP001-0-041007	10	4.40	44
C0049878 Spk H	DF06-V01-RCP001-0-041007	100	70.4	70
C0049879	DF06-V02-AVP001-0-041007	10	4.44	44
C0049879 Rep	DF06-V02-AVP001-0-041007	10	4.56	46
C0049879 Spk I	DF06-V02-AVP001-0-041007	10	5.20	52
C0049879 Spk J	DF06-V02-AVP001-0-041007	100	58.4	58
C0049880	DF06-V02-PAP001-0-041007	10	2.70	27
C0049880 Rep	DF06-V02-PAP001-0-041007	10	2.78	28
C0049880 Spk K	DF06-V02-PAP001-0-041007	10	3.40	34
C0049880 Spk L	DF06-V02-PAP001-0-041007	100	65.2	65
C0049881	DF06-V02-RCP001-0-041007	10	4.76	48
C0049881 Rep	DF06-V02-RCP001-0-041007	10	4.72	47
C0049881 Spk C	DF06-V02-RCP001-0-041007	10	5.20	52
C0049881 Spk D	DF06-V02-RCP001-0-041007	100	73.2	73
C0049882	DF09-V01-AVP001-0-041007	10	4.92	49
C0049882 Rep	DF09-V01-AVP001-0-041007	10	5.64	56
C0049882 Spk E	DF09-V01-AVP001-0-041007	10	6.64	66
C0049882 Spk F	DF09-V01-AVP001-0-041007	100	61.2	61
C0049883	DF09-V01-PAP001-0-041007	10	4.64	46
C0049883 Rep	DF09-V01-PAP001-0-041007	10	5.24	52
C0049883 Spk G	DF09-V01-PAP001-0-041007	10	6.12	61
C0049883 Spk H	DF09-V01-PAP001-0-041007	100	66.8	67

NR = Not reported due to quality control result failures.

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

Exygen STUDY NO. P760
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Surrogate Spike Recovery Summary for ¹³C-PFOA in Vegetation Samples (continued)

Exygen ID	Sample Description	Amount Spiked (ng/g)	¹³ C-PFOA	
			Amount Recovered (ng/g)	Recovery (%)
C0049884	DF09-V01-RCP001-0-041007	10	5.88	59
C0049884 Rep	DF09-V01-RCP001-0-041007	10	5.68	57
C0049884 Spk I	DF09-V01-RCP001-0-041007	10	6.56	66
C0049884 Spk J	DF09-V01-RCP001-0-041007	100	80.0	80
C0049885	DF09-V02-RCP001-0-041007	10	4.68	47
C0049885 Rep	DF09-V02-RCP001-0-041007	10	4.20	42
C0049885 Spk K	DF09-V02-RCP001-0-041007	10	4.60	46
C0049885 Spk L	DF09-V02-RCP001-0-041007	100	73.6	74
C0049886	DF09-V02-PAP001-0-041007	10	2.76	28
C0049886 Rep	DF09-V02-PAP001-0-041007	10	2.60	26
C0049886 Spk C	DF09-V02-PAP001-0-041007	10	3.28	33
C0049886 Spk D	DF09-V02-PAP001-0-041007	100	65.2	65
C0049887	DF8b-V01-PAP001-0-041007	10	1.67	17
C0049887 Rep	DF8b-V01-PAP001-0-041007	10	1.62	16
C0049887 Spk E	DF8b-V01-PAP001-0-041007	10	1.92	19
C0049887 Spk F	DF8b-V01-PAP001-0-041007	100	48.0	48
C049888	DF8b-V01-RCP001-0-041007	10	3.54	35
C0049888 Rep	DF8b-V01-RCP001-0-041007	10	3.58	36
C0049888 Spk G	DF8b-V01-RCP001-0-041007	10	3.97	40
C0049888 Spk H	DF8b-V01-RCP001-0-041007	100	71.6	72
C0049889	DF8b-V02-PAP001-0-041007	10	1.48	15
C0049889 Rep	DF8b-V02-PAP001-0-041007	10	1.43	14
C0049889 Spk I	DF8b-V02-PAP001-0-041007	10	1.76	18
C0049889 Spk J	DF8b-V02-PAP001-0-041007	100	46.8	47
C0049890	DF8b-V02-RCP001-0-041007	10	3.58	36
C0049890 Rep	DF8b-V02-RCP001-0-041007	10	3.06	31
C0049890 Spk K	DF8b-V02-RCP001-0-041007	10	3.84	38
C0049890 Spk L	DF8b-V02-RCP001-0-041007	100	73.2	73
C0049891	DF14-V01-AVP001-0-041007	10	6.24	62
C0049891 Rep	DF14-V01-AVP001-0-041007	10	6.20	62
C0049891 Spk C	DF14-V01-AVP001-0-041007	10	7.16	72
C0049891 Spk D	DF14-V01-AVP001-0-041007	100	67.6	68

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

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Surrogate Spike Recovery Summary for ¹³C-PFOA in Vegetation Samples (continued)

Exygen ID	Sample Description	Amount Spiked (ng/g)	¹³ C-PFOA	
			Amount Recovered (ng/g)	Recovery (%)
C0049892	DF14-V01-PAP001-0-041007	10	6.32	63
C0049892 Rep	DF14-V01-PAP001-0-041007	10	6.36	64
C0049892 Spk E	DF14-V01-PAP001-0-041007	10	6.96	70
C0049892 Spk F	DF14-V01-PAP001-0-041007	100	81.2	81
C0049893	DF14-V02-AVP001-0-041007	10	6.52	65
C0049893 Rep	DF14-V02-AVP001-0-041007	10	6.44	64
C0049893 Spk G	DF14-V02-AVP001-0-041007	10	6.92	69
C0049896 Spk H	DF14-V02-AVP001-0-041007	100	74.0	74
C0049894	DF14-V02-PAP001-0-041007	10	6.88	69
C0049894 Rep	DF14-V02-PAP001-0-041007	10	6.36	64
C0049894 Spk I	DF14-V02-PAP001-0-041007	10	7.08	71
C0049894 Spk J	DF14-V02-PAP001-0-041007	100	81.2	81
C0049895	DBKG-V01-AVP001-0-041007	10	6.84	68
C0049895 Rep	DBKG-V01-AVP001-0-041007	10	6.64	66
C0049895 Spk K	DBKG-V01-AVP001-0-041007	10	7.80	78
C0049895 Spk L	DBKG-V01-AVP001-0-041007	100	80.8	81
C0049896	DBKG-V01-PAP001-0-041007	10	6.08	61
C0049896 Rep	DBKG-V01-PAP001-0-041007	10	6.36	64
C0049896 Spk C	DBKG-V01-PAP001-0-041007	10	7.48	75
C0049896 Spk D	DBKG-V01-PAP001-0-041007	100	76.0	76
C0049897	DBKG-V01-RCP001-0-041007	10	6.68	67
C0049897 Rep	DBKG-V01-RCP001-0-041007	10	6.44	64
C0049897 Spk E	DBKG-V01-RCP001-0-041007	10	7.16	72
C0049897 Spk F	DBKG-V01-RCP001-0-041007	100	79.2	79
C0050021	DF09-V01-SSP001-0-041007	10	1.25	13
C0050021 Rep	DF09-V01-SSP001-0-041007	10	1.31	13
C0050021 Spk C	DF09-V01-SSP001-0-041007	10	1.67	17
C0050021 Spk D	DF09-V01-SSP001-0-041007	100	54.8	55
C0050022	DF09-V02-UGP001-0-041007	10	2.70	27
C0050022 Rep	DF09-V02-UGP001-0-041007	10	2.51	25
C0050022 Spk E	DF09-V02-UGP001-0-041007	10	3.00	30
C0050022 Spk F	DF09-V02-UGP001-0-041007	100	60.0	60

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

Exygen STUDY NO. D760
 AMB 09/22/05

Surrogate Spike Recovery Summary for ¹³C-PFOA in Vegetation Samples (continued)

Exygen ID	Sample Description	Amount Spiked (ng/g)	¹³ C-PFOA	
			Amount Recovered (ng/g)	Recovery (%)
C0050023	DF06-V01-SSP001-0-041007	10	1.06	11
C0050023 Rep	DF06-V01-SSP001-0-041007	10	1.22	12
C0050023 Spk G	DF06-V01-SSP001-0-041007	10	1.24	12
C0050023 Spk H	DF06-V01-SSP001-0-041007	100	50.0	50
C0050024	DF06-V02-SSP001-0-041007	10	NR	NR
C0050024 Rep	DF06-V02-SSP001-0-041007	10	NR	NR
C0050024 Spk I	DF06-V02-SSP001-0-041007	10	NR	NR
C0050024 Spk J	DF06-V02-SSP001-0-041007	100	NR	NR
C0050025	DF8b-V01-UGP001-0-041007	10	1.88	19
C0050025 Rep	DF8b-V01-UGP001-0-041007	10	1.75	18
C0050025 Spk K	DF8b-V01-UGP001-0-041007	10	2.05	21
C0050025 Spk L	DF8b-V01-UGP001-0-041007	100	54.0	54
C0050026	DF8b-V02-UGP001-0-041007	10	3.97	40
C0050026 Rep	DF8b-V02-UGP001-0-041007	10	3.82	38
C0050026 Spk C	DF8b-V02-UGP001-0-041007	10	4.68	47
C0050026 Spk D	DF8b-V02-UGP001-0-041007	100	64.4	64
C0050027	DF14-V01-SSP001-0-041007	10	NR	NR
C0050027 Rep	DF14-V01-SSP001-0-041007	10	NR	NR
C0050027 Spk E	DF14-V01-SSP001-0-041007	10	NR	NR
C0050027 Spk F	DF14-V01-SSP001-0-041007	100	NR	NR
C0050028	DF14-V02-SSP001-0-041007	10	NR	NR
C0050028 Rep	DF14-V02-SSP001-0-041007	10	NR	NR
C0050028 Spk G	DF14-V02-SSP001-0-041007	10	NR	NR
C0050028 Spk H	DF14-V02-SSP001-0-041007	100	NR	NR
C0050029	DBKG-V01-SSP001-0-041007	10	4.40	44
C0050029 Rep	DBKG-V01-SSP001-0-041007	10	4.48	45
C0050029 Spk I	DBKG-V01-SSP001-0-041007	10	5.40	54
C0050029 Spk J	DBKG-V01-SSP001-0-041007	100	80.4	80
			Average:	50
			Standard Deviation:	20

NR = Not reported due to quality control result failures.

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

Exygen STUDY NO. P760
AMB 09/22/05

	A	B	C	D
1	Summary of PFOA in Vegetation Samples			
2				
3				C8 Acid PFOA
4		Client		Perfluorooctanoic Acid
5	Exygen ID	Sample ID	ound (ppb, ng/g) W	Assessed Accuracy (+/- %)
7	C0049876	DF06-V01-AVP001-0-041007	90	60
8	C0049876 Rep	DF06-V01-AVP001-0-041007*	78.4	60
10	C0049877	DF06-V01-PAP001-0-041007	NR	----
11	C0049877 Rep	DF06-V01-PAP001-0-041007*	NR	----
13	C0049878	DF06-V01-RCP001-0-041007	238	30
14	C0049878 Rep	DF06-V01-RCP001-0-041007*	255	30
16	C0049879	DF06-V02-AVP001-0-041007	46.8	50
17	C0049879 Rep	DF06-V02-AVP001-0-041007*	37.7	50
19	C0049880	DF06-V02-PAP001-0-041007	440	40
20	C0049880 Rep	DF06-V02-PAP001-0-041007*	472	40
22	C0049881	DF06-V02-RCP001-0-041007	123	60
23	C0049881 Rep	DF06-V02-RCP001-0-041007*	138	60
25	C0049882	DF09-V01-AVP001-0-041007	10	50
26	C0049882 Rep	DF09-V01-AVP001-0-041007*	9.56	50
28	C0049883	DF09-V01-PAP001-0-041007	62	50
29	C0049883 Rep	DF09-V01-PAP001-0-041007*	64.8	50
31	C0049884	DF09-V01-RCP001-0-041007	50.4	30
32	C0049884 Rep	DF09-V01-RCP001-0-041007*	48.8	30
34	C0049885	DF09-V02-RCP001-0-041007	150	30
35	C0049885 Rep	DF09-V02-RCP001-0-041007*	165	30
37	C0049886	DF09-V02-PAP001-0-041007	448	40
38	C0049886 Rep	DF09-V02-PAP001-0-041007*	464	40
40	C0049887	DF8b-V01-PAP001-0-041007	912	60
41	C0049887 Rep	DF8b-V01-PAP001-0-041007*	912	60
43	C0049888	DF8b-V01-RCP001-0-041007	212	30
44	C0049888 Rep	DF8b-V01-RCP001-0-041007*	204	30
46	C0049889	DF8b-V02-PAP001-0-041007	944	60
47	C0049889 Rep	DF8b-V02-PAP001-0-041007*	904	60
49	C0049890	DF8b-V02-RCP001-0-041007	236	50
50	C0049890 Rep	DF8b-V02-RCP001-0-041007*	225	50
52	C0049891	DF14-V01-AVP001-0-041007	4.76	30
53	C0049891 Rep	DF14-V01-AVP001-0-041007*	6.04	30
55	C0049892	DF14-V01-PAP001-0-041007	9.36	60
56	C0049892 Rep	DF14-V01-PAP001-0-041007*	9.08	60
58	C0049893	DF14-V02-AVP001-0-041007	5.52	40
59	C0049893 Rep	DF14-V02-AVP001-0-041007*	5.52	40
61	C0049894	DF14-V02-PAP001-0-041007	11.3	40
62	C0049894 Rep	DF14-V02-PAP001-0-041007*	11.4	40
64	C0049895	DBKG-V01-AVP001-0-041007	3.7	40
65	C0049895 Rep	DBKG-V01-AVP001-0-041007*	2.85	40
67	C0049896	DBKG-V01-PAP001-0-041007	3.92	30
68	C0049896 Rep	DBKG-V01-PAP001-0-041007*	4.68	30
70	C0049897	DBKG-V01-RCP001-0-041007	2	30
71	C0049897 Rep	DBKG-V01-RCP001-0-041007*	1.97	30
73				
74	*Laboratory Duplicate			
75	ND = Not detected at or abc			
76	NQ = Not quantifiable = Me:			
77	NR = Not reported due to q			
78				

	A	B	C	D
79				
80	Summary of PFOA in Vegetation Samples (continued)			
81				
82				C8 Acid PFOA
83		Client		Perfluorooctanoic Acid
84	Exygen ID	Sample ID	ound (ppb, ng/g) W	Assessed Accuracy (+/- %)
86	C0050021	DF09-V01-SSP001-0-041007	112	50
87	C0050021 Rep	DF09-V01-SSP001-0-041007*	108	50
89	C0050022	DF09-V02-UGP001-0-041007	388	30
90	C0050022 Rep	DF09-V02-UGP001-0-041007*	386	30
92	C0050023	DF06-V01-SSP001-0-041007	245	30
93	C0050023 Rep	DF06-V01-SSP001-0-041007*	258	30
95	C0050024	DF06-V02-SSP001-0-041007	NR	----
96	C0050024 Rep	DF06-V02-SSP001-0-041007*	NR	----
98	C0050025	DF8b-V01-UGP001-0-041007	580	50
99	C0050025 Rep	DF8b-V01-UGP001-0-041007*	632	50
101	C0050026	DF8b-V02-UGP001-0-041007	369	60
102	C0050026 Rep	DF8b-V02-UGP001-0-041007*	362	60
104	C0050027	DF14-V01-SSP001-0-041007	NR	----
105	C0050027 Rep	DF14-V01-SSP001-0-041007*	NR	----
107	C0050028	DF14-V02-SSP001-0-041007	NR	----
108	C0050028 Rep	DF14-V02-SSP001-0-041007*	NR	----
110	C0050029	DBKG-V01-SSP001-0-041007	2.53	50
111	C0050029 Rep	DBKG-V01-SSP001-0-041007*	2.51	50
113				
114	*Laboratory Duplicate			
115	ND = Not detected at or above			
116	NQ = Not quantifiable = Me			
117	NR = Not reported due to q			

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	A	B	C	D	E
1	Laboratory Matrix Spike Recovery Summary for PFOA in Vegetation Samples				
2					
3	C8 Acid PFOA				
4		Amount	Amt Found	Amount	
5	Sample	Spiked	in Sample	Recovered	Recovery
6	Description	(ng/g)	(ng/g)	(ng/g)	(%)
8	DF06-V01-AVP001-0-041007				
9	(C0049876 Spk C, 10 ppb)	10	90	98.8	*
11	DF06-V01-AVP001-0-041007				
12	(C0049876 Spk D, 100 ppb)	100	90	129	$=((D12-C12)/B12)*100$
14	DF06-V01-PAP001-0-41007				
15	(C0049877 Spk E, 10 ppb)	10	NR	NR	NR
17	DF06-V01-PAP001-0-041007				
18	(C0049877 Spk F, 100 ppb)	100	NR	NR	NR
20	DF06-V01-RCP001-0-041007				
21	(C0049878 Spk G, 10 ppb)	10	238	265	*
23	DF06-V01-RCP001-0-041007				
24	(C0049878 Spk H, 100 ppb)	100	238	317	$=((D24-C24)/B24)*100$
26	DF06-V02-AVP001-0-041007				
27	(C0049879 Spk I, 10 ppb)	10	46.8	49.2	*
29	DF06-V02-AVP001-0-041007				
30	(C0049879 Spk J, 100 ppb)	100	46.8	98.8	$=((D30-C30)/B30)*100$
32	DF06-V02-PAP001-0-041007				
33	(C0049880 Spk K, 10 ppb)	10	440	496	*
35	DF06-V02-PAP001-0-041007				
36	(C0049880 Spk L, 100 ppb)	100	440	540	*
38	DF06-V02-RCP001-0-041007				
39	(C0049881 Spk C, 10 ppb)	10	123	128	*
41	DF06-V02-RCP001-0-041007				
42	(C0049881 Spk D, 100 ppb)	100	123	168	$=((D42-C42)/B42)*100$
44	DF09-V01-AVP001-0-041007				
45	(C0049882 Spk E, 10 ppb Spike)	10	10	15.8	$=((D45-C45)/B45)*100$
47	DF09-V01-AVP001-0-041007				
48	(C0049882 Spk F, 100 ppb Spike)	100	10	71.2	$=((D48-C48)/B48)*100$
50	DF09-V01-PAP001-0-041007				
51	(C0049883 Spk G, 10 ppb Spike)	10	62	70	*
53	DF09-V01-PAP001-0-041007				
54	(C0049883 Spk H, 100 ppb Spike)	100	62	116	$=((D54-C54)/B54)*100$
56	DF09-V01-RCP001-0-041007				
57	(C0049884 Spk I, 10 ppb Spike)	10	50.4	58	*
59	DF09-V01-RCP001-0-041007				
60	(C0049884 Spk J, 100 ppb Spike)	100	50.4	133	$=((D60-C60)/B60)*100$
62	DF09-V02-RCP001-0-041007				
63	(C0049885 Spk K, 10 ppb Spike)	10	150	165	*
65	DF09-V02-RCP001-0-041007				
66	(C0049885 Spk L, 100 ppb Spike)	100	150	246	$=((D66-C66)/B66)*100$
68					
69	*Sample residue exceeds the spiking level				
70	ND = Not detected at or above 0.2 ng/g				
71	NQ = Not quantifiable = Measured concentration				
72	NR = Not reported due to quality control				
73	Note: Since this summary table shows				
74					
75					
76					
77					

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	A	B	C	D	E
78					
79	atory Matrix Spike Recovery Summary for PFOA in Vegetation Samples (cont				
80					
81	C8 Acid PFOA				
82		Amount	Amt Found	Amount	
83	Sample	Spiked	in Sample	Recovered	Recovery
84	Description	(ng/g)	(ng/g)	(ng/g)	(%)
86	DF09-V02-PAP001-0-041007				
87	(C0049886 Spk C, 10 ppb Spike)	10	448	416	*
89	DF09-V02-PAP001-0-041007				
90	(C0049886 Spk D, 100 ppb Spike)	100	448	540	*
91					
92	DF8b-V01-PAP001-0-041007				
93	(C0049887 Spk E, 10 ppb Spike)	10	912	996	*
95	DF8b-V01-PAP001-0-041007				
96	(C0049887 Spk F, 100 ppb Spike)	100	912	1060	*
97					
98	DF8b-V01-RCP001-0-041007				
99	(C0049888 Spk G, 10 ppb Spike)	10	212	212	*
101	DF8b-V01-RCP001-0-041007				
102	(C0049888 Spk H, 100 ppb Spike)	100	212	298	$=((D102-C102)/B102)*100$
103					
104	DF8b-V02-PAP001-0-041007				
105	(C0049889 Spk I, 10 ppb Spike)	10	944	948	*
107	DF8b-V02-PAP001-0-041007				
108	(C0049889 Spk J, 100 ppb Spike)	100	944	1044	*
109					
110	DF8b-V02-RCP001-0-041007				
111	(C0049890 Spk K, 10 ppb Spike)	10	236	236	*
113	DF8b-V02-RCP001-0-041007				
114	(C0049890 Spk L, 100 ppb Spike)	100	236	384	$=((D114-C114)/B114)*100$
115					
116	DF14-V01-AVP001-0-041007				
117	(C0049891 Spk C, 10 ppb Spike)	10	4.76	12.6	$=((D117-C117)/B117)*100$
119	DF14-V01-AVP001-0-041007				
120	(C0049891 Spk D, 100 ppb Spike)	100	4.76	72	$=((D120-C120)/B120)*100$
121					
122	DF14-V01-PAP001-0-041007				
123	(C0049892 Spk E, 10 ppb Spike)	10	9.36	13.8	$=((D123-C123)/B123)*100$
125	DF14-V01-PAP001-0-041007				
126	(C0049892 Spk F, 100 ppb Spike)	100	9.36	93.6	$=((D126-C126)/B126)*100$
127					
128	DF14-V02-AVP001-0-041007				
129	(C0049893 Spk G, 10 ppb Spike)	10	5.52	12.2	$=((D129-C129)/B129)*100$
131	DF14-V02-AVP001-0-041007				
132	(C0049893 Spk H, 100 ppb Spike)	100	5.52	79.2	$=((D132-C132)/B132)*100$
133					
134	DF14-V02-PAP001-0-041007				
135	(C0049894 Spk I, 10 ppb Spike)	10	11.3	18.2	$=((D135-C135)/B135)*100$
137	DF14-V02-PAP001-0-041007				
138	(C0049894 Spk J, 100 ppb Spike)	100	11.3	95.2	$=((D138-C138)/B138)*100$
139					
140	DBKG-V01-AVP001-0-041007				
141	(C0049895 Spk K, 10 ppb Spike)	10	3.7	10.4	$=((D141-C141)/B141)*100$
143	DBKG-V01-AVP001-0-041007				
144	(C0049895 Spk L, 100 ppb Spike)	100	3.7	84.8	$=((D144-C144)/B144)*100$
146					
147	*Sample residue exceeds the spiking level				
148	ND = Not detected at or above 0.2 ng/g				
149	NQ = Not quantifiable = Measured concentration				
150	NR = Not reported due to quality control				
151	Note: Since this summary table shows				
152					
153					
154					

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	A	B	C	D	E
155					
156					
157	atory Matrix Spike Recovery Summary for PFOA in Vegetation Samples (cont				
158					
159					C8 Acid PFOA
160	Sample	Amount Spiked (ng/g)	Amt Found in Sample (ng/g)	Amount Recovered (ng/g)	Recovery (%)
161	Description				
162					
164	DBKG-V01-PAP001-0-041007				
165	(C0049896 Spk C, 10 ppb Spike)	10	3.92	11.5	$=((D165-C165)/B165)*100$
167	DBKG-V01-PAP001-0-041007				
168	(C0049896 Spk D, 100 ppb Spike)	100	3.92	80	$=((D168-C168)/B168)*100$
169					
170	DBKG-V01-RCP001-0-041007				
171	(C0049897 Spk E, 10 ppb Spike)	10	2	9.04	$=((D171-C171)/B171)*100$
173	DBKG-V01-RCP001-0-041007				
174	(C0049897 Spk F, 100 ppb Spike)	100	2	78	$=((D174-C174)/B174)*100$
175					
176	DF09-V01-SSP001-0-041007				
177	(C0050021 Spk C, 10 ppb)	10	112	116	*
179	DF09-V01-SSP001-0-041007				
180	(C0050021 Spk D, 100 ppb)	100	112	163	$=((D180-C180)/B180)*100$
181					
182	DF09-V02-UGP001-0-041007				
183	(C0050022 Spk E, 10 ppb)	10	388	366	*
185	DF09-V02-UGP001-0-041007				
186	(C0050022 Spk F, 100 ppb)	100	388	464	$=((D186-C186)/B186)*100$
187					
188	DF06-V01-SSP001-0-041007				
189	(C0050023 Spk G, 10 ppb)	10	245	260	*
191	DF06-V01-SSP001-0-041007				
192	(C0050023 Spk H, 100 ppb)	100	245	315	$=((D192-C192)/B192)*100$
193					
194	DF06-V02-SSP001-0-041007				
195	(C0050024 Spk I, 10 ppb)	10	NR	NR	NR
197	DF06-V02-SSP001-0-041007				
198	(C0050024 Spk J, 100 ppb)	100	NR	NR	NR
199					
200	DF8b-V01-UGP001-0-041007				
201	(C0050025 Spk K, 10 ppb)	10	580	824	*
203	DF8b-V01-UGP001-0-041007				
204	(C0050025 Spk L, 100 ppb)	100	580	848	*
205					
206	DF8b-V02-UGP001-0-041007				
207	(C0050026 Spk C, 10 ppb)	10	369	364	*
209	DF8b-V02-UGP001-0-041007				
210	(C0050026 Spk D, 100 ppb)	100	369	416	$=((D210-C210)/B210)*100$
211					
212	DF14-V01-SSP001-0-041007				
213	(C0050027 Spk E, 10 ppb)	10	NR	NR	NR
215	DF14-V01-SSP001-0-041007				
216	(C0050027 Spk F, 100 ppb)	100	NR	NR	NR
217					
218	DF14-V02-SSP001-0-041007				
219	(C0050028 Spk G, 10 ppb)	10	NR	NR	NR
221	DF14-V02-SSP001-0-041007				
222	(C0050028 Spk H, 100 ppb)	100	NR	NR	NR
224					
225	*Sample residue exceeds the spiking l				
226	ND = Not detected at or above 0.2 ng/				
227	NQ = Not quantifiable = Measured cor				
228	NR = Not reported due to quality contr				
229	Note: Since this summary table shows:				
230					
231					

	A	B	C	D	E
232					
233					
234					
235	Laboratory Matrix Spike Recovery Summary for PFOA in Vegetation Samples (continued)				
236					
237					C8 Acid PFOA
238		Amount	Amt Found	Amount	
239	Sample	Spiked	in Sample	Recovered	Recovery
240	Description	(ng/g)	(ng/g)	(ng/g)	(%)
242	DBKG-V01-SSP001-0-041007				
243	(C0050029 Spk I, 10 ppb)	10	2.53	7.92	$=((D243-C243)/B243)*100$
245	DBKG-V01-SSP001-0-041007				
246	(C0050029 Spk J, 100 ppb)	100	2.53	85.2	$=((D246-C246)/B246)*100$
249					Average: $=AVERAGE(E243:E246,E165:E222,E87:E144,E9:E66)$
250					Std Deviation: $=STDEV(E243:E246,E165:E222,E87:E144,E9:E66)$
251					
252	*Sample residue exceeds the spiking level				
253	ND = Not detected at or above 0.2 ng/g				
254	NQ = Not quantifiable = Measured concentration				
255	NR = Not reported due to quality control				
256	Note: Since this summary table shows				

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	A	B	C	D	E
1	Surrogate Spike Recovery Summary for ¹³C-PFOA in Vegetation Samples				
2					
3					¹³ C-PFOA
4			Amount	Amount	
5	Oxygen	Sample	Spike	Recovered	Recovery
6	ID	Description	(ng/g)	(ng/g)	(%)
7					
8					
9	C0049876	DF06-V01-AVP001-0-041007	10	4.16	=(D9)/C9*100
10	C0049876 Rep	DF06-V01-AVP001-0-041007	10	4.16	=(D10)/C10*100
11	C0049876 Spk C	DF06-V01-AVP001-0-041007	10	4.72	=(D11)/C11*100
12	C0049876 Spk D	DF06-V01-AVP001-0-041007	100	64.4	=(D12)/C12*100
13					
14	C0049877	DF06-V01-PAP001-0-041007	10	NR	NR
15	C0049877 Rep	DF06-V01-PAP001-0-041007	10	NR	NR
16	C0049877 Spk E	DF06-V01-PAP001-0-041007	10	NR	NR
17	C0049877 Spk F	DF06-V01-PAP001-0-041007	100	NR	NR
18					
19	C0049878	DF06-V01-RCP001-0-041007	10	4.04	=(D19)/C19*100
20	C0049878 Rep	DF06-V01-RCP001-0-041007	10	3.82	=(D20)/C20*100
21	C0049878 Spk G	DF06-V01-RCP001-0-041007	10	4.4	=(D21)/C21*100
22	C0049878 Spk H	DF06-V01-RCP001-0-041007	100	70.4	=(D22)/C22*100
23					
24	C0049879	DF06-V02-AVP001-0-041007	10	4.44	=(D24)/C24*100
25	C0049879 Rep	DF06-V02-AVP001-0-041007	10	4.56	=(D25)/C25*100
26	C0049879 Spk I	DF06-V02-AVP001-0-041007	10	5.2	=(D26)/C26*100
27	C0049879 Spk J	DF06-V02-AVP001-0-041007	100	58.4	=(D27)/C27*100
28					
29	C0049880	DF06-V02-PAP001-0-041007	10	2.7	=(D29)/C29*100
30	C0049880 Rep	DF06-V02-PAP001-0-041007	10	2.78	=(D30)/C30*100
31	C0049880 Spk K	DF06-V02-PAP001-0-041007	10	3.4	=(D31)/C31*100
32	C0049880 Spk L	DF06-V02-PAP001-0-041007	100	65.2	=(D32)/C32*100
33					
34	C0049881	DF06-V02-RCP001-0-041007	10	4.76	=(D34)/C34*100
35	C0049881 Rep	DF06-V02-RCP001-0-041007	10	4.72	=(D35)/C35*100
36	C0049881 Spk C	DF06-V02-RCP001-0-041007	10	5.2	=(D36)/C36*100
37	C0049881 Spk D	DF06-V02-RCP001-0-041007	100	73.2	=(D37)/C37*100
38					
39	C0049882	DF09-V01-AVP001-0-041007	10	4.92	=(D39)/C39*100
40	C0049882 Rep	DF09-V01-AVP001-0-041007	10	5.64	=(D40)/C40*100
41	C0049882 Spk E	DF09-V01-AVP001-0-041007	10	6.64	=(D41)/C41*100
42	C0049882 Spk F	DF09-V01-AVP001-0-041007	100	61.2	=(D42)/C42*100
43					
44	C0049883	DF09-V01-PAP001-0-041007	10	4.64	=(D44)/C44*100
45	C0049883 Rep	DF09-V01-PAP001-0-041007	10	5.24	=(D45)/C45*100
46	C0049883 Spk G	DF09-V01-PAP001-0-041007	10	6.12	=(D46)/C46*100
47	C0049883 Spk H	DF09-V01-PAP001-0-041007	100	66.8	=(D47)/C47*100
48					
49					
50	NR = Not reported d				
51	Note: Since this sum				
52					

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	A	B	C	D	E
53	Surrogate Spike Recovery Summary for ¹³C-PFOA in Vegetation Samples (continued)				
54					
55					¹³ C-PFOA
56			Amount	Amount	
57	Oxygen	Sample	Spike	Recovery	Recovery
58	ID	Description	(ng/g)	(ng/g)	(%)
59					
60					
61	C0049884	DF09-V01-RCP001-0-041007	10	5.88	=(D61)/C61*100
62	C0049884 Rep	DF09-V01-RCP001-0-041007	10	5.68	=(D62)/C62*100
63	C0049884 Spk I	DF09-V01-RCP001-0-041007	10	6.56	=(D63)/C63*100
64	C0049884 Spk J	DF09-V01-RCP001-0-041007	100	80	=(D64)/C64*100
65					
66	C0049885	DF09-V02-RCP001-0-041007	10	4.68	=(D66)/C66*100
67	C0049885 Rep	DF09-V02-RCP001-0-041007	10	4.2	=(D67)/C67*100
68	C0049885 Spk K	DF09-V02-RCP001-0-041007	10	4.6	=(D68)/C68*100
69	C0049885 Spk L	DF09-V02-RCP001-0-041007	100	73.6	=(D69)/C69*100
70					
71	C0049886	DF09-V02-PAP001-0-041007	10	2.76	=(D71)/C71*100
72	C0049886 Rep	DF09-V02-PAP001-0-041007	10	2.6	=(D72)/C72*100
73	C0049886 Spk C	DF09-V02-PAP001-0-041007	10	3.28	=(D73)/C73*100
74	C0049886 Spk D	DF09-V02-PAP001-0-041007	100	65.2	=(D74)/C74*100
75					
76	C0049887	DF8b-V01-PAP001-0-041007	10	1.67	=(D76)/C76*100
77	C0049887 Rep	DF8b-V01-PAP001-0-041007	10	1.62	=(D77)/C77*100
78	C0049887 Spk E	DF8b-V01-PAP001-0-041007	10	1.92	=(D78)/C78*100
79	C0049887 Spk F	DF8b-V01-PAP001-0-041007	100	48	=(D79)/C79*100
80					
81	C049888	DF8b-V01-RCP001-0-041007	10	3.54	=(D81)/C81*100
82	C0049888 Rep	DF8b-V01-RCP001-0-041007	10	3.58	=(D82)/C82*100
83	C0049888 Spk G	DF8b-V01-RCP001-0-041007	10	3.97	=(D83)/C83*100
84	C0049888 Spk H	DF8b-V01-RCP001-0-041007	100	71.6	=(D84)/C84*100
85					
86	C0049889	DF8b-V02-PAP001-0-041007	10	1.48	=(D86)/C86*100
87	C0049889 Rep	DF8b-V02-PAP001-0-041007	10	1.43	=(D87)/C87*100
88	C0049889 Spk I	DF8b-V02-PAP001-0-041007	10	1.76	=(D88)/C88*100
89	C0049889 Spk J	DF8b-V02-PAP001-0-041007	100	46.8	=(D89)/C89*100
90					
91	C0049890	DF8b-V02-RCP001-0-041007	10	3.58	=(D91)/C91*100
92	C0049890 Rep	DF8b-V02-RCP001-0-041007	10	3.06	=(D92)/C92*100
93	C0049890 Spk K	DF8b-V02-RCP001-0-041007	10	3.84	=(D93)/C93*100
94	C0049890 Spk L	DF8b-V02-RCP001-0-041007	100	73.2	=(D94)/C94*100
95					
96	C0049891	DF14-V01-AVP001-0-041007	10	6.24	=(D96)/C96*100
97	C0049891 Rep	DF14-V01-AVP001-0-041007	10	6.2	=(D97)/C97*100
98	C0049891 Spk C	DF14-V01-AVP001-0-041007	10	7.16	=(D98)/C98*100
99	C0049891 Spk D	DF14-V01-AVP001-0-041007	100	67.6	=(D99)/C99*100
100					
101					
102	Note: Since this surr				
103					

	A	B	C	D	E
104	Surrogate Spike Recovery Summary for ¹³C-PFOA in Vegetation Samples (continued)				
105					
106					¹³ C-PFOA
107			Amount	Amount	
108	Exygen	Sample	Spike	Recoverec	Recovery
109	ID	Description	(ng/g)	(ng/g)	(%)
110					
111					
112	C0049892	DF14-V01-PAP001-0-041007	10	6.32	=(D112)/C112*100
113	C0049892 Rep	DF14-V01-PAP001-0-041007	10	6.36	=(D113)/C113*100
114	C0049892 Spk E	DF14-V01-PAP001-0-041007	10	6.96	=(D114)/C114*100
115	C0049892 Spk F	DF14-V01-PAP001-0-041007	100	81.2	=(D115)/C115*100
116					
117	C0049893	DF14-V02-AVP001-0-041007	10	6.52	=(D117)/C117*100
118	C0049893 Rep	DF14-V02-AVP001-0-041007	10	6.44	=(D118)/C118*100
119	C0049893 Spk G	DF14-V02-AVP001-0-041007	10	6.92	=(D119)/C119*100
120	C0049896 Spk H	DF14-V02-AVP001-0-041007	100	74	=(D120)/C120*100
121					
122	C0049894	DF14-V02-PAP001-0-041007	10	6.88	=(D122)/C122*100
123	C0049894 Rep	DF14-V02-PAP001-0-041007	10	6.36	=(D123)/C123*100
124	C0049894 Spk I	DF14-V02-PAP001-0-041007	10	7.08	=(D124)/C124*100
125	C0049894 Spk J	DF14-V02-PAP001-0-041007	100	81.2	=(D125)/C125*100
126					
127	C0049895	DBKG-V01-AVP001-0-041007	10	6.84	=(D127)/C127*100
128	C0049895 Rep	DBKG-V01-AVP001-0-041007	10	6.64	=(D128)/C128*100
129	C0049895 Spk K	DBKG-V01-AVP001-0-041007	10	7.8	=(D129)/C129*100
130	C0049895 Spk L	DBKG-V01-AVP001-0-041007	100	80.8	=(D130)/C130*100
131					
132	C0049896	DBKG-V01-PAP001-0-041007	10	6.08	=(D132)/C132*100
133	C0049896 Rep	DBKG-V01-PAP001-0-041007	10	6.36	=(D133)/C133*100
134	C0049896 Spk C	DBKG-V01-PAP001-0-041007	10	7.48	=(D134)/C134*100
135	C0049896 Spk D	DBKG-V01-PAP001-0-041007	100	76	=(D135)/C135*100
136					
137	C0049897	DBKG-V01-RCP001-0-041007	10	6.68	=(D137)/C137*100
138	C0049897 Rep	DBKG-V01-RCP001-0-041007	10	6.44	=(D138)/C138*100
139	C0049897 Spk E	DBKG-V01-RCP001-0-041007	10	7.16	=(D139)/C139*100
140	C0049897 Spk F	DBKG-V01-RCP001-0-041007	100	79.2	=(D140)/C140*100
141					
142	C0050021	DF09-V01-SSP001-0-041007	10	1.25	=(D142)/C142*100
143	C0050021 Rep	DF09-V01-SSP001-0-041007	10	1.31	=(D143)/C143*100
144	C0050021 Spk C	DF09-V01-SSP001-0-041007	10	1.67	=(D144)/C144*100
145	C0050021 Spk D	DF09-V01-SSP001-0-041007	100	54.8	=(D145)/C145*100
146					
147	C0050022	DF09-V02-UGP001-0-041007	10	2.7	=(D147)/C147*100
148	C0050022 Rep	DF09-V02-UGP001-0-041007	10	2.51	=(D148)/C148*100
149	C0050022 Spk E	DF09-V02-UGP001-0-041007	10	3	=(D149)/C149*100
150	C0050022 Spk F	DF09-V02-UGP001-0-041007	100	60	=(D150)/C150*100
151					
152					
153	Note: Since this surr				
154					

0227 Exygen STUDY NO. P760
AMB 09/22/05

	A	B	C	D	E
155	Surrogate Spike Recovery Summary for ¹³C-PFOA in Vegetation Samples (continued)				
156					
157					¹³ C-PFOA
158			Amount	Amount	
159	Exygen	Sample	Spike	Recovery	Recovery
160	ID	Description	(ng/g)	(ng/g)	(%)
161					
162					
163	C0050023	DF06-V01-SSP001-0-041007	10	1.06	=(D163)/C163*100
164	C0050023 Rep	DF06-V01-SSP001-0-041007	10	1.22	=(D164)/C164*100
165	C0050023 Spk G	DF06-V01-SSP001-0-041007	10	1.24	=(D165)/C165*100
166	C0050023 Spk H	DF06-V01-SSP001-0-041007	100	50	=(D166)/C166*100
167					
168	C0050024	DF06-V02-SSP001-0-041007	10	NR	NR
169	C0050024 Rep	DF06-V02-SSP001-0-041007	10	NR	NR
170	C0050024 Spk I	DF06-V02-SSP001-0-041007	10	NR	NR
171	C0050024 Spk J	DF06-V02-SSP001-0-041007	100	NR	NR
172					
173	C0050025	DF8b-V01-UGP001-0-041007	10	1.88	=(D173)/C173*100
174	C0050025 Rep	DF8b-V01-UGP001-0-041007	10	1.75	=(D174)/C174*100
175	C0050025 Spk K	DF8b-V01-UGP001-0-041007	10	2.05	=(D175)/C175*100
176	C0050025 Spk L	DF8b-V01-UGP001-0-041007	100	54	=(D176)/C176*100
177					
178	C0050026	DF8b-V02-UGP001-0-041007	10	3.97	=(D178)/C178*100
179	C0050026 Rep	DF8b-V02-UGP001-0-041007	10	3.82	=(D179)/C179*100
180	C0050026 Spk C	DF8b-V02-UGP001-0-041007	10	4.68	=(D180)/C180*100
181	C0050026 Spk D	DF8b-V02-UGP001-0-041007	100	64.4	=(D181)/C181*100
182					
183	C0050027	DF14-V01-SSP001-0-041007	10	NR	NR
184	C0050027 Rep	DF14-V01-SSP001-0-041007	10	NR	NR
185	C0050027 Spk E	DF14-V01-SSP001-0-041007	10	NR	NR
186	C0050027 Spk F	DF14-V01-SSP001-0-041007	100	NR	NR
187					
188	C0050028	DF14-V02-SSP001-0-041007	10	NR	NR
189	C0050028 Rep	DF14-V02-SSP001-0-041007	10	NR	NR
190	C0050028 Spk G	DF14-V02-SSP001-0-041007	10	NR	NR
191	C0050028 Spk H	DF14-V02-SSP001-0-041007	100	NR	NR
192					
193	C0050029	DBKG-V01-SSP001-0-041007	10	4.4	=(D193)/C193*100
194	C0050029 Rep	DBKG-V01-SSP001-0-041007	10	4.48	=(D194)/C194*100
195	C0050029 Spk I	DBKG-V01-SSP001-0-041007	10	5.4	=(D195)/C195*100
196	C0050029 Spk J	DBKG-V01-SSP001-0-041007	100	80.4	=(D196)/C196*100
197					
198			Average:	=AVERAGE(E163:E196,E112:E150,E61:E99,E9:E47)	
199			Deviation:	=STDEV(E163:E196,E112:E150,E61:E99,E9:E47)	
200					
201	NR = Not reported d				
202	Note: Since this surr				

Exygen STUDY NO. P760

AMB 09/22/05

0228

Vegetation Set #1:

Internal Chain of Custody/Fortification
Sheets

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ : FREEZER 34	Time: 0829	Date: 07/06/05	Initials: <i>CEH</i>

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0073483 Control	5.0	—
—	C0073483 Spike A	5.0	2.5 ^{ng} /g
—	C0073483 Spike B	5.0	10 ^{ng} /g
<i>BAK 07/12/05</i>			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	Micropipet		Initials/Date
			Syringe ①	Volume (μL)	
Spike A	SS0007404 (0.1 ^{ng} /mL)	125	25/50	25/50	<i>BAK 07/12/05</i>
Spike B	SS0007404 (0.1 ^{ng} /mL)	500	100/200	100/200	<i>BAK 07/12/05</i>
Spike —	—	—	—	—	—
Spike —	—	—	—	—	—

All samples were measured on:			
Balance ID: BALANCE 20	Time: 0900	Date: 07/06/05	Initials: <i>CEH</i>

After measuring samples were returned to:			
CED ID ¹ : FREEZER 34	Time: 1235	Date: 07/06/05	Initials: <i>CEH</i>

Comments: ① ② *BAK 07/12/05*

Analysis Summary	Data Set ID:	071205D	Initials/Date:	<i>AMB</i>	1 07/14/05
	Data Set ID:	071205DR	Initials/Date:	<i>AMB</i>	1 07/22/05
	Data Set ID:	—	Initials/Date:	—	1 —

Set extraction/analysis data verified by	Initials/Date:	<i>CEH</i> 10/22/05
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¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used*	ExyLIMS Inventory ID
---	0073483 Control	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	-	-	Methanol	RE0020201
---	0073483 Spk A	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	-	-	Acetonitrile	RE0020106
---	0073483 Spk B	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	-	-	Carbon	RE0019742
															Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019970
															2% Ascorbic Acid in Methanol	SL0014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640
															Toluene	RE0007267
Initials/Date	-	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	---	AMB 07/14/05	-	-	Extraction Items Initials/Date	BAK 07/12/05
			BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05					Analysis Items Initials/Date	* AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.

STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605

STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.

STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/12/05

STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605

STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182

STEP 7 = Repeat steps 5 through 6 again.

STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.

STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.

STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.

STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

→ After this step samples were covered w/ foil and stored in Refrigerator 32 (IN0000708) overnight.
BAK 07/12/05

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0231

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ : FREEZER 34	Time: 0829	Date: 07/06/05	Initials: <i>CLH</i>

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0049876 Spike C	5.0	10 mg/g
—	C0049876 Spike D	5.0	100 mg/g
—	C0049876	5.0	10 mg/g*
—	C0049876 Rep.	5.0	10 mg/g*
BAK 07/06/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	MicroPipet Syringe Volume (μL)	Initials/Date
Spike C	SS0007404 (0.1 μg/mL)	500	100/200	BAK 07/12/05
Spike D	SS0007403 (1.0 μg/mL)	500	100/200	BAK 07/12/05
Spike *	SS0007505 (0.1 μg/mL) ^{13C}	500	100/200	BAK 07/12/05
Spike —	—	—	—	—

All samples were measured on:			
Balance ID: BALANCE 20	Time: 0900	Date: 07/06/05	Initials: <i>CLH</i>

After measuring samples were returned to:			
CED ID ¹ : FREEZER 34	Time: 1225	Date: 07/06/05	Initials: <i>CLH</i>

Comments: ① (CC) BAK 07/12/05

Analysis Summary	Data Set ID:	071205D	Initials/Date:	AMB 10/14/05
	Data Set ID:	071205DR	Initials/Date:	AMB 10/22/05
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:	<i>BAK</i> 10/22/05
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¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used*	ExyLIMS Inventory ID	
---	CO049876 Spk C										---		0.1 1.0 0.1 10		Methanol	RE0020201	
---	CO049876 Spk B										0.1/1.0		0.1 1.0 0.1 10		Acetonitrile	RE0020106	
---	CO049876										---		0.1 1.0 0.1 10		Carbon	RE0019742	
---	CO049876 Kp	✓	✓	✓	✓	✓	✓	✓	✓	✓	---	↓	0.1 1.0 0.1 10	↓	Silica gel	RE0000639	
															Florisil	RE0000641	
															LC-NH2	RE0019970	
															2% Ascorbic Acid in Methanol	SL0014702	
															1-Octanol	RE0016118	
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640	
															Toluene	RE0007267	
Initials/Date	-	See ICOC page.	BWK 07/12/05	BWK 07/15/05	BWK 07/16/05	BWK 07/12/05	BWK 07/12/05	BWK 07/12/05	BWK 07/12/05	BWK 07/13/05	BWK 07/13/05	AMB 07/14/05	AMB 07/14/05	AMB 07/22/05	AMB 07/22/05	Extraction Items Initials/Date	BWK 07/12/05
																Analysis Items Initials/Date	*AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

DF=10,100 AMB 07/22/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: 1N0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: 1N000082 Glassware silanized: initials/Date BWK 07/15/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: 1N0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: 1N000082
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

After this step, samples were covered w/ foil and stored in Refrigerator 32 (1N0000708) overnight.
BWK 07/12/05

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BWK Date: 07/13/05

0233

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ : FREEZER 34	Time: 0829	Date: 07/06/05	Initials: CLK

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0049877 Spike E	5.0	10 ^{ng} /g
—	C0049877 Spike F	5.0	100 ^{ng} /g
—	C0049877	5.0	10 ^{ng} /g*
—	C0049877 Rep.	5.0	10 ^{ng} /g*
BAK 07/06/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	Micropipet	Initials/Date
			Syringe-D Volume (μL)	
Spike E	SS0007404 (0.1 ^{ng} /ml)	500	100/200	BAK 07/12/05
Spike F	SS0007403 (1.0 ^{ng} /ml)	500	100/200	BAK 07/12/05
Spike *	SS0007505 (0.1 ^{ng} /ml)	500	100/200	BAK 07/12/05
Spike —	—	—	—	—

All samples were measured on:			
Balance ID: BALANCE 20	Time: 0900	Date: 07/06/05	Initials: CLK

After measuring samples were returned to:			
CED ID ¹ : FREEZER 34	Time: 1225	Date: 07/06/05	Initials: CLK

Comments: ① (CC) BAK 07/12/05

Analysis Summary	Data Set ID:	07120SD	Initials/Date:	AMB / 07/14/05
	Data Set ID:	07120SDR	Initials/Date:	AMB / 07/22/05
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:	OR / 09/22/05
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¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
—	CO049877 S*F										—		① 0.1 0.1 0.1 1.0 1.0 1.0		Methanol	RE0020201
—	CO049877 S*F										0.1/1.0		② 0.1 0.1 1.0 1.0		Acetonitrile	RE0020106
—	CO049877 S*F										—		① 0.1 0.1 0.1 1.0 1.0 1.0		Carbon	RE0019742
—	CO049877 Rep.	✓	✓	✓	✓	✓	✓	✓	✓	✓	—	↓	① 0.1 0.1 0.1 1.0 1.0 1.0	↓	Silica gel	RE0000639
Florisil																
LC-NH2																
2% Ascorbic Acid in Methanol																
1-Octanol																
30% Dimethyldichlorosilane in Toluene Solution																
Toluene																
Initials/Date	—	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	AMB 07/14/05	AMB 07/14/05	AMB 07/22/05	AMB 07/22/05	Extraction Items Initials/Date	BAK 07/12/05
															Analysis Items Initials/Date	*AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: W0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: 1N000062 Glassware silanized: initials/Date BAK 07/05/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: 1N0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: 1N0000713
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C. After this step, samples were covered w/ foil.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial. and stored in Refrigerator 32 (1N0000708) overnight.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS. BAK 07/12/05
- STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0235

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID: FREEZER 34	Time: 0829	Date: 07/06/05	Initials: CCB

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
_____	C0049878 Spike G	5.0	10 ^{ng} /g
_____	C0049878 Spike H	5.0	100 ^{ng} /g
_____	C0049878	5.0	10 ^{ng} /g*
_____	C0049878 Rep.	5.0	10 ^{ng} /g*
BAK 07/08/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (µL)	Micropipet		Initials/Date
			Syringe ①	Volume (µL)	
Spike G	SS0007404 (0.1 ^{ng} /mL)	500	100/200	100/200	BAK 07/12/05
Spike H	SS0007403 (1.0 ^{ng} /mL)	500	100/200	100/200	BAK 07/12/05
Spike *	SS0007505 (0.1 ^{ng} /mL) ^{13C}	500	100/200	100/200	BAK 07/12/05
Spike _____	_____	_____	_____	_____	_____

All samples were measured on:			
Balance ID: BALANCE 20	Time: 0900	Date: 07/06/05	Initials: CCB

After measuring samples were returned to:			
CED ID: FREEZER 34	Time: 1225	Date: 07/06/05	Initials: CCB

Comments: ① ② BAK 07/12/05

Analysis Summary	Data Set ID:	071205D	Initials/Date:	AMB / 07/14/05
	Data Set ID:	071205DR	Initials/Date:	AMB / 07/22/05
	Data Set ID:	_____	Initials/Date:	_____ / _____

Set extraction/analysis data verified by	Initials/Date:	BAK / 08/22/05
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¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Oxygen Study/Project Number: F760/P1131

Matrix: Vegetation

Client Sample ID	Oxygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID	
---	CO049878 Spk G	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	⓪ $\frac{0.1}{1.0} \frac{0.1}{10}$	↓	Methanol	RE0020201	
---	CO049878 Spk H	↓	↓	↓	↓	↓	↓	↓	↓	↓	0.1/1.0	↓	$\frac{0.1}{1.0} \frac{0.1}{10}$	↓	Acetonitrile	RE0020106	
---	CO049878	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	⓪ $\frac{0.1}{1.0} \frac{0.1}{10}$	↓	Carbon	RE0019742	
---	CO049878 Rep.	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	⓪ $\frac{0.1}{1.0} \frac{0.1}{10}$	↓	Silica gel	RE0000639	
BAK 07/12/05																	
Florisil RE0000641																	
LC-NH2 RE0019970																	
2% Ascorbic Acid in Methanol SL0014702																	
1-Octanol RE0016118																	
30% Dimethyldichlorosilane in Toluene Solution SL0014640																	
Toluene RE0007267																	
Initials/Date	-	See ICOC page.	BAK 07/12/05	BAK 07/15/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/13/05	BAK 07/13/05	LMB 07/14/05	AMB 07/14/05	AMB 07/22/05	AMB 07/22/05	Extraction Items Initials/Date	BAK 07/12/05
																Analysis Items Initials/Date	* AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

⓪ DF = 10,100 AMB 07/22/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/05/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

After this step, samples were covered w/ foil and stored in Refrigerator 32 (IN0000708) overnight.
BAK 07/12/05

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0237

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.	Matrix:
P0000760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID: FREEZER 34	Time: 0829	Date: 07/06/05	Initials: <i>CLB</i>

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0049879 Spike I	5.0	10 ^{ng} /g
—	C0049879 Spike J	5.0	100 ^{ng} /g
—	C0049879	5.0	10 ^{ng} /g *
—	C0049879 Rep.	5.0	10 ^{ng} /g *
<i>BAK</i> 07/06/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	Micropipet		Initials/Date
			Syringe	Volume (μL)	
Spike I	SS0007404 (0.1 ^{ng} /mL)	500	100/200		<i>BAK</i> 07/12/05
Spike J	SS0007403 (1.0 ^{ng} /mL)	500	100/200		<i>BAK</i> 07/12/05
Spike *	SS0007505 (0.1 ^{ng} /mL ¹)	500	100/200		<i>BAK</i> 07/12/05
Spike —	—	—	—	—	—

All samples were measured on:			
Balance ID: BALANCE 20	Time: 0900	Date: 07/06/05	Initials: <i>CLB</i>

After measuring samples were returned to:			
CED ID: FREEZER 34	Time: 1225	Date: 07/06/05	Initials: <i>CLB</i>

Comments: ① ② *BAK* 07/12/05

Analysis Summary	Data Set ID:	071205D	Initials/Date:	<i>AMB</i> / 07/14/05
	Data Set ID:	071205DR	Initials/Date:	<i>AMB</i> / 07/22/05
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:	<i>DR</i> / 08/22/05
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¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID	
---	C0049879 Spt 1	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	0.1 1.0 0.1 10	↓	Methanol	RE0020201	
---	C0049879 Spt 2	↓	↓	↓	↓	↓	↓	↓	↓	↓	0.1/1.0	↓	0.1 1.0 0.1 10	↓	Acetonitrile	RE0020106	
---	C0049879 Spt 3	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	0.1 1.0 0.1 10	↓	Carbon	RE0019742	
---	C0049879 Rep	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	0.1 1.0 0.1 10	↓	Silica gel	RE0000639	
Florisil RE0000641																	
LC-NH2 RE0019970																	
2% Ascorbic Acid in Methanol SL0014762																	
1-Octanol RE0016118																	
30% Dimethyldichlorosilane in Toluene Solution SL0014640																	
Toluene RE0007267																	
Initials/Date	-	See ICOC page.	BAK 07/12/05	BAK 07/15/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/13/05	BAK 07/13/05	AMB 07/14/05	AMB 07/14/05	AMB 07/22/05	AMB 07/22/05	Extraction Items Initials/Date	BAK 07/12/05
																Analysis Items Initials/Date	* AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

DDF=10,100 AMB 07/22/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: W0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: W0000182 Glassware silanized: initials/Date BAK 07/15/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: W0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: W0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C. → After this step, samples were covered w/ foil and stored in Refrigerator 32 (W0000708) overnight.
- STEP 9 = Add 20 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.:	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	0829	07/06/05	AMB

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0049880 Spike K	5.0	10 ^{ng} /g
—	C0049880 Spike L	5.0	100 ^{ng} /g
—	C0049880	5.0	10 ^{ng} /g*
—	C0049880 Rep.	5.0	10 ^{ng} /g*
BAK 07/06/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	Micropipet	Initials/Date
			Syringe (1) Volume (μL)	
Spike K	SS0007404 (0.1 ^{ng} /mL)	500	100/200	BAK 07/12/05
Spike L	SS0007403 (1.0 ^{ng} /mL)	500	100/200	BAK 07/12/05
Spike *	SS0007505 (0.1 ^{ng} /mL ¹ C)	500	100/200	BAK 07/12/05
Spike —	—	—	—	—

All samples were measured on:			
Balance ID:	Time:	Date:	Initials:
BALANCE 20	0900	07/06/05	AMB

After measuring samples were returned to:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1225	07/06/05	AMB

Comments: (1) (2) BAK 07/12/05

Analysis Summary	Data Set ID:	071205D	Initials/Date:	AMB 107/14/05
	Data Set ID:	071205DR	Initials/Date:	AMB 107/22/05
	Data Set ID:	071205DR2	Initials/Date:	AMB 107/28/05

Set extraction/analysis data verified by	Initials/Date:
	AMB 108/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: _____ Vegetation _____

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
—	00049880 Spk K										—		① 0.1 0.1 0.1 1.0 1.0 1.0	②	Methanol	RE0020201
—	00049880 Spk L										0.4/1.0		① 0.1 0.1 0.1 1.0 1.0 1.0	③	Acetonitrile	RE0020106
—	00049880 Spk L										—		① 0.1 0.1 0.1 1.0 1.0 1.0	④	Carbon	RE0019742
—	00049880 Rep.	↓	↓	↓	↓	↓	↓	↓	↓	↓	—	↓	① 0.1 0.1 0.1 1.0 1.0 1.0	⑤ ↓	Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019970
															2% Ascorbic Acid in Methanol	SL0014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640
															Toluene	RE0007267
Initials/Date	—	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/13/05	BAK 07/13/05	AMB 07/14/05	AMB 07/14/05	AMB 07/22/05	AMB 07/22/05	Extraction Items Initials/Date	BAK 07/12/05
															Analysis Items Initials/Date	* AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
 STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605
 STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
 STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/05/05
 STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605
 STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182
 STEP 7 = Repeat steps 5 through 6 again.
 STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
 STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
 STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
 STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

① DF = 10, 100 AMB 07/22/05
 ② DF = 10, 100, 1000; 0.1 0.1 0.1
 ③ DF = 100, 1000; 0.1 0.1 0.1
 > AMB 07/28/05
 After this step, samples were covered w/ foil and stored in Refrigerator 32 (IN0000708) overnight.
 BAK 07/12/05

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0241

Vegetation Set #1

Data Set 071205D

Extracted on 07/12/05

(Analyzed on 07/14-15/05)

AMB 07/14/05

\\Abbeyroad\PE Sciex Data\Projects\P1131 Decatur Monitoring Study Batch:071205D VEG P760 Tab:Sample Set:SET1 AcqMethod:P760-1131 Isomer Separation

Sample Name	Sample ID	Vial Position	Data File	Run #
SS7279	Calibration Standard, 0.5 ng/mL	1	08_071205D\071205D	071205D-101
SS7278	Calibration Standard, 1.0 ng/mL	2	08_071205D\071205D	071205D-102
SS7277	Calibration Standard, 2.5 ng/mL	3	08_071205D\071205D	071205D-103
SS7276	Calibration Standard, 5.0 ng/mL	4	08_071205D\071205D	071205D-104
SS7275	Calibration Standard, 10 ng/mL	5	08_071205D\071205D	071205D-105
SS7274	Calibration Standard, 25 ng/mL	6	08_071205D\071205D	071205D-106
SS7273	Calibration Standard, 50 ng/mL	7	08_071205D\071205D	071205D-107
Methanol Wash	Methanol Wash	92	08_071205D\071205D	071205D-108
Reagent Control	C0054394 Control	11	08_071205D\071205D	071205D-109
Reagent Spk A	C0054394 Spike A, 2.5 ng/g	12	08_071205D\071205D	071205D-110
Reagent Spk B	C0054394 Spike B, 10 ng/g	13	08_071205D\071205D	071205D-111
SS7279	Calibration Standard, 0.5 ng/mL	1	08_071205D\071205D	071205D-112
C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	14	08_071205D\071205D	071205D-113
C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	15	08_071205D\071205D	071205D-114
C0049876	DF06V01AVP0010041007	16	08_071205D\071205D	071205D-115
C0049876 Rep	DF06V01AVP0010041007	17	08_071205D\071205D	071205D-116
SS7278	Calibration Standard, 1.0 ng/mL	2	08_071205D\071205D	071205D-117
C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	18	08_071205D\071205D	071205D-118
C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	19	08_071205D\071205D	071205D-119
C0049877	DF06V01PAP0010041007	20	08_071205D\071205D	071205D-120
C0049877 Rep	DF06V01PAP0010041007	21	08_071205D\071205D	071205D-121
SS7277	Calibration Standard, 2.5 ng/mL	3	08_071205D\071205D	071205D-122
C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	22	08_071205D\071205D	071205D-123
C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	23	08_071205D\071205D	071205D-124
C0049878	DF06V01RCP0010041007	24	08_071205D\071205D	071205D-125
C0049878 Rep	DF06V01RCP0010041007	25	08_071205D\071205D	071205D-126
SS7276	Calibration Standard, 5.0 ng/mL	4	08_071205D\071205D	071205D-127
C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	26	08_071205D\071205D	071205D-128
C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	27	08_071205D\071205D	071205D-129
C0049879	DF06V02AVP0010041007	28	08_071205D\071205D	071205D-130
C0049879 Rep	DF06V02AVP0010041007	29	08_071205D\071205D	071205D-131
SS7275	Calibration Standard, 10 ng/mL	5	08_071205D\071205D	071205D-132
C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g	30	08_071205D\071205D	071205D-133
C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=10	31	08_071205D\071205D	071205D-134
C0049880	DF06V02PVP0010041007	32	08_071205D\071205D	071205D-135
C0049880 Rep	DF06V02PVP0010041007	33	08_071205D\071205D	071205D-136
SS7274	Calibration Standard, 25 ng/mL	6	08_071205D\071205D	071205D-137
SS7273	Calibration Standard, 50 ng/mL	7	08_071205D\071205D	071205D-138

① RE 73483 MLC 9/21/05
 ② RE PAP MLC 9/21/05

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Study No: NA

Exygen Study No: P760/P1131

Instrument: API 4000 LC/MS/MS System, (LC/MS/MS #8)
Turbo Ion Spray Liquid Introduction Interface

Computer: DELL OptiPlex GX400

Software: Windows NT, Analyst 1.4

HPLC Equipment: 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 (Exygen ID MA0018925)

Column Temperature: 35°C

Mobile Phase (A) : 2 mM ammonium acetate in water (SL0014984)

Mobile Phase (B) : Methanol (RE0020196)

<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

Injected Volume: 15 µL

Ions monitored:

<u>Analyte</u>	<u>Mode</u>	<u>Transition Monitored</u>
PFBS	negative	299 → 99
PFHS	negative	399 → 80
PFOS	negative	499 → 80
PFOA	negative	413 → 369
PFOA Confirm Ion	negative	413 → 219
¹³ C PFOA	negative	415 → 370

Analyst: Amy Sheehan *AMB 07/14/05*
Exygen Research
3058 Research Drive, State College, PA 16801
Phone : (814) 272-1039 FAX : (814) 231-1580

NOTE: ALL HANDWRITTEN PEAK IDENTIFICATIONS BY *AMB / 07/19/05*

CEE 07/28/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
1 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	157152	0.500	0.288	071205D-101
2 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	293199	1.00	0.906	071205D-102
3 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	697320	2.50	2.74	071205D-103
4 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1444221	5.00	6.13	071205D-104
5 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2725320	10.0	11.9	071205D-105
6 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5908891	25.0	26.4	071205D-106
7 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10588859	50.0	47.6	071205D-107
8 Methanol Wash	Methanol Wash	Unknown	1	PFOA	33187	N/A	< 0	071205D-108
9 Reagent Control	C0073483 Control	Unknown	1	PFOA	55009	N/A	< 0	071205D-109
10 Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFOA	1565685	6.25	6.68	071205D-110
11 Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFOA	6278495	25.0	28.1	071205D-111
12 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	182338	0.500	0.403	071205D-112
13 C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	Quality Control	1	PFOA	28926901	① 25.0	131.	071205D-113
14 C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	7195952	250.	322.	071205D-114
15 C0049876	DF06V01AVP0010041007	Unknown	1	PFOA	27411925	① N/A	124.	071205D-115
16 C0049876 Rep	DF06V01AVP0010041007	Unknown	1	PFOA	24894626	① N/A	113.	071205D-116
17 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	279854	1.00	0.845	071205D-117
18 C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	Quality Control	1	PFOA	111947693	① 25.0	508.	071205D-118
19 C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	38860553	① 250.	1760.	071205D-119
20 C0049877	DF06V01PAP0010041007	Unknown	1	PFOA	111415202	① N/A	505.	071205D-120
21 C0049877 Rep	DF06V01PAP0010041007	Unknown	1	PFOA	109850324	① N/A	498.	071205D-121
22 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	795631	2.50	3.19	071205D-122
23 C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	Quality Control	1	PFOA	64230832	① 25.0	291.	071205D-123
24 C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	14499202	① 250.	654.	071205D-124
25 C0049878	DF06V01RCP0010041007	Unknown	1	PFOA	60234325	① N/A	273.	071205D-125
26 C0049878 Rep	DF06V01RCP0010041007	Unknown	1	PFOA	61401925	① N/A	278.	071205D-126
27 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1234165	5.00	5.18	071205D-127
28 C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	Quality Control	1	PFOA	19433745	① 25.0	87.8	071205D-128
29 C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	5527940	250.	247.	071205D-129
30 C0049879	DF06V02AVP0010041007	Unknown	1	PFOA	18689056	① N/A	84.4	071205D-130
31 C0049879 Rep	DF06V02AVP0010041007	Unknown	1	PFOA	16169588	① N/A	73.0	071205D-131
32 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2542658	10.0	11.1	071205D-132
33 C0049880 Spike K	DF06V02AVP0010041007, 10 ng/g	Quality Control	1	PFOA	79506341	① 25.0	361.	071205D-133
34 C0049880 Spike L	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	21180098	① 250.	957.	071205D-134
35 C0049880	DF06V02AVP0010041007	Unknown	1	PFOA	76873462	① N/A	348.	071205D-135
36 C0049880 Rep	DF06V02AVP0010041007	Unknown	1	PFOA	76925150	① N/A	349.	071205D-136
37 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5535612	25.0	24.7	071205D-137
38 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10338038	50.0	46.5	071205D-138

① Requires dilution, see data set 071205DR. BAK 09/20/05

② RE 73483 m/c 9/2/05

Vegetation Conversion

Compound: PFOA

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049876 Spk D	DF06-V01-AVP001-0-041007	322	129
C0049879 Spk J	DF06-V02-AVP001-0-041007	247	98.8

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected ~~Peak Area 0~~ at or above 0.2 ng/g. ①

NQ = Not quantifiable = Measured concentration below Limit of Quantitation (LOQ) which is 0.7 ng/g.

^
between 0.2 ng/g and ①

4①

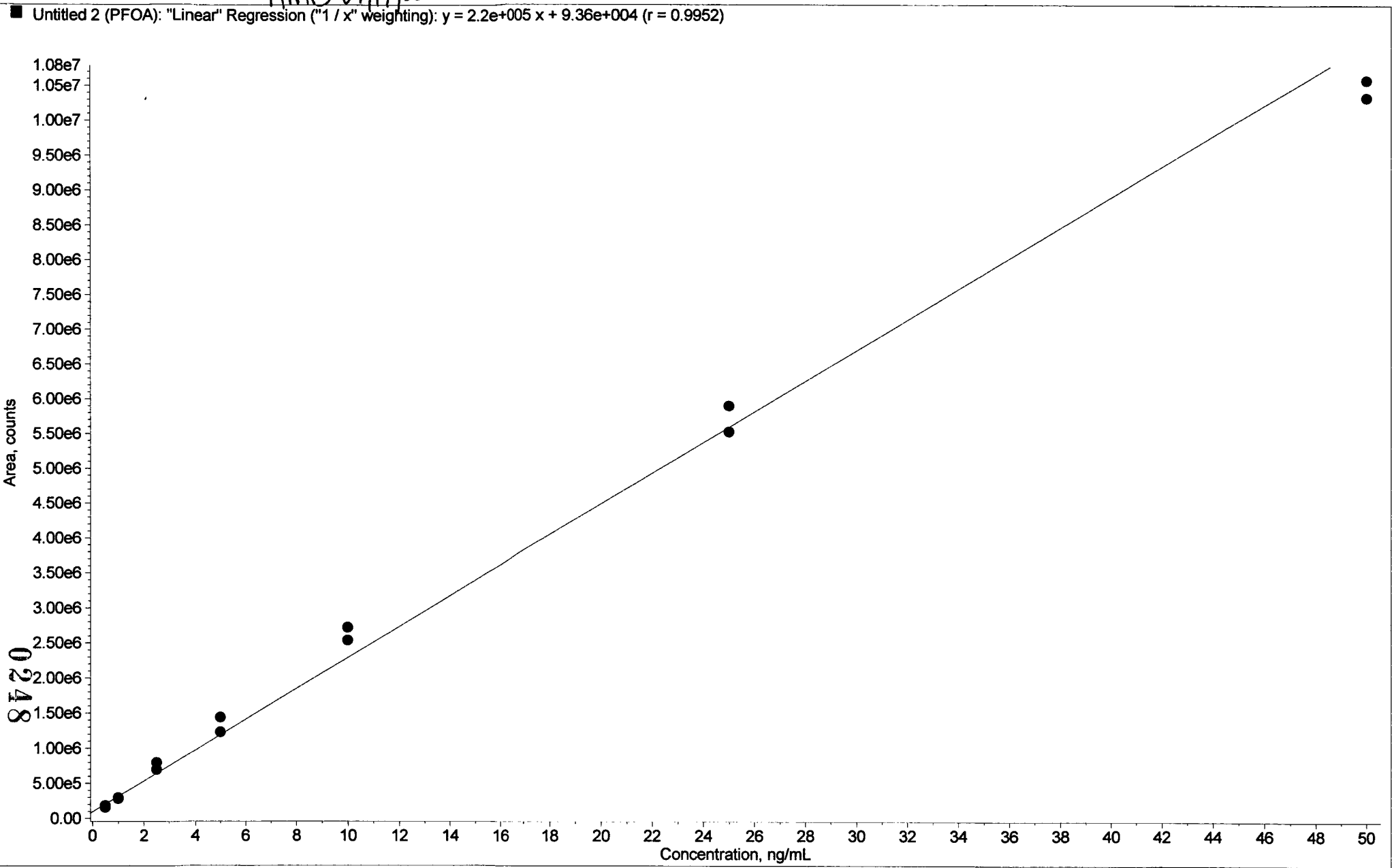
① (RE) BAK 09/20/05

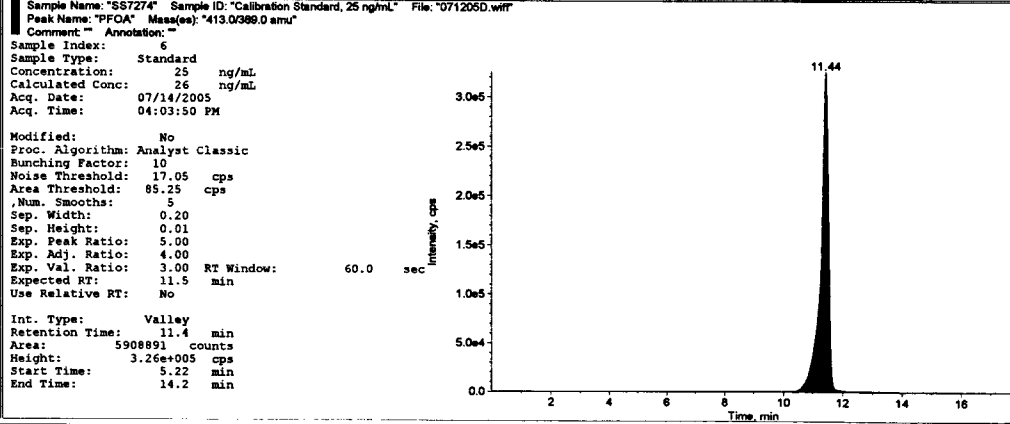
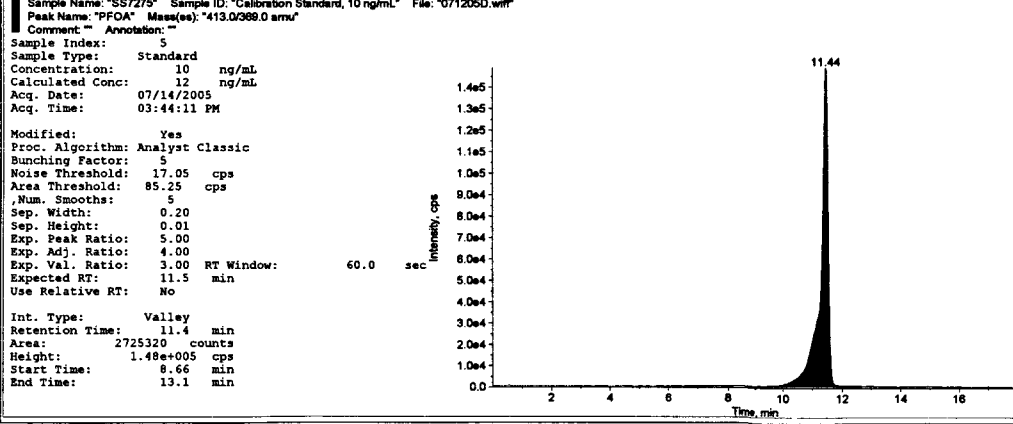
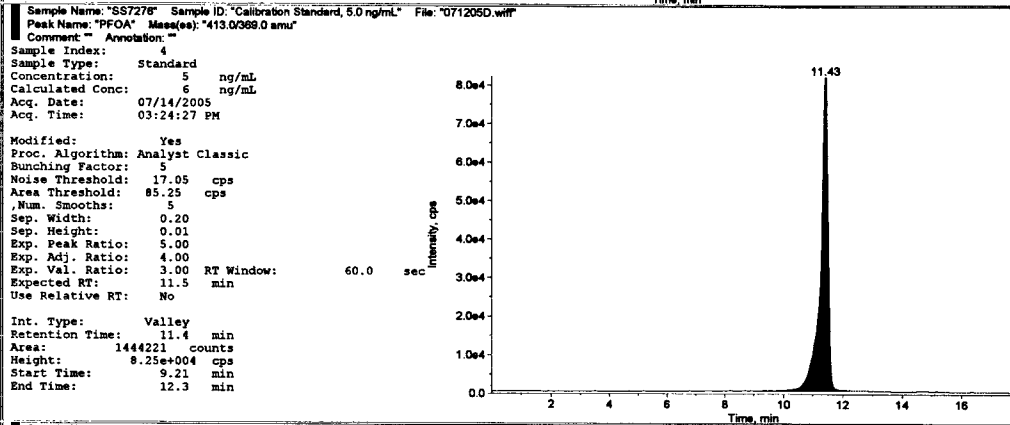
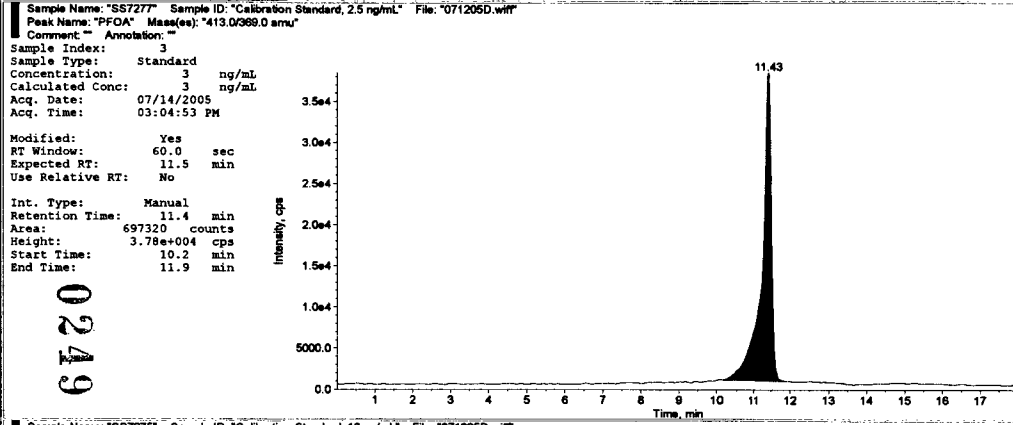
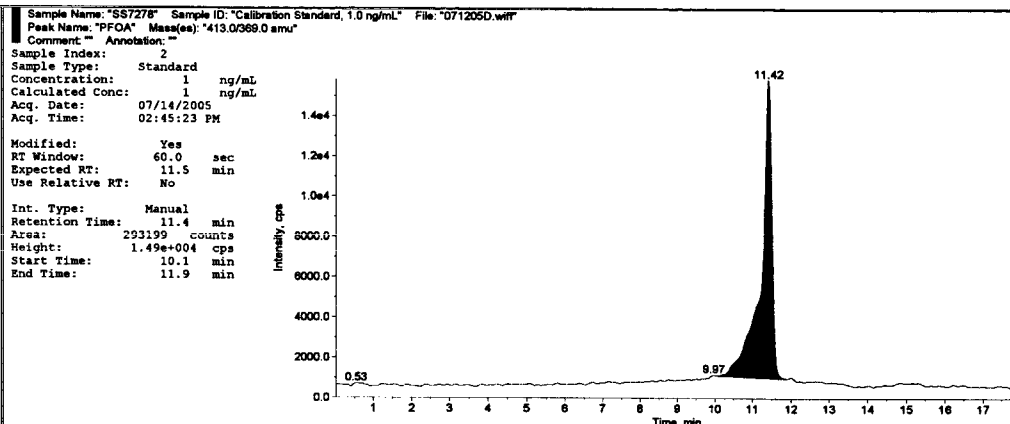
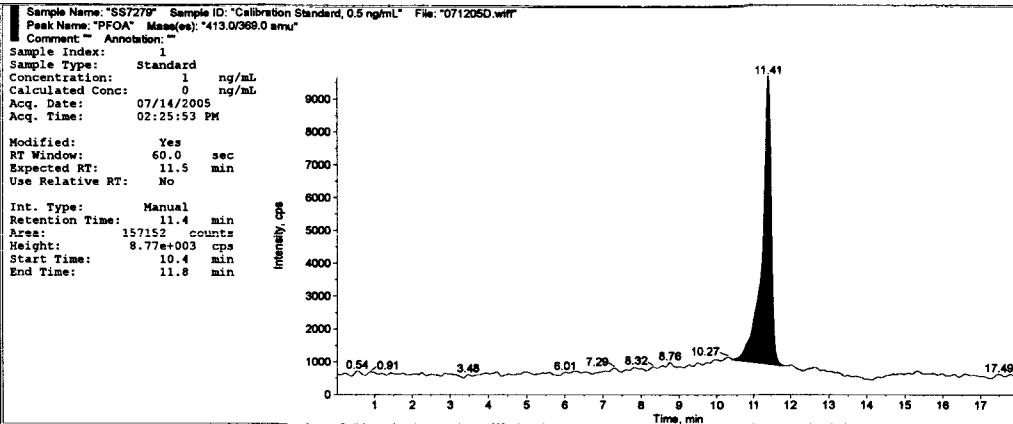
	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound:	PFOA	Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049876 Spk D	DF06-V01-AVP001-0-041007	=Raw Data!H143	=(C9*2)/5
10				
11	C0049879 Spk J	DF06-V02-AVP001-0-041007	=Raw Data!H158	=(C11*2)/5
12				

0247

BRK 09/29/05

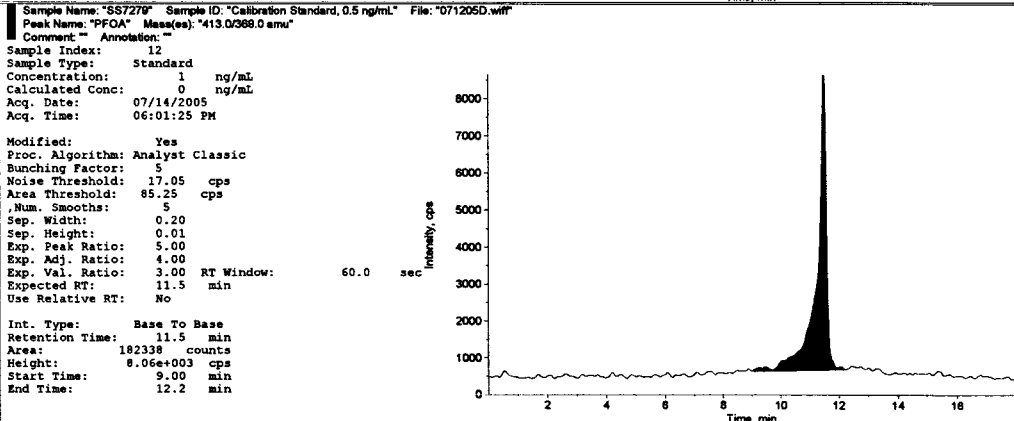
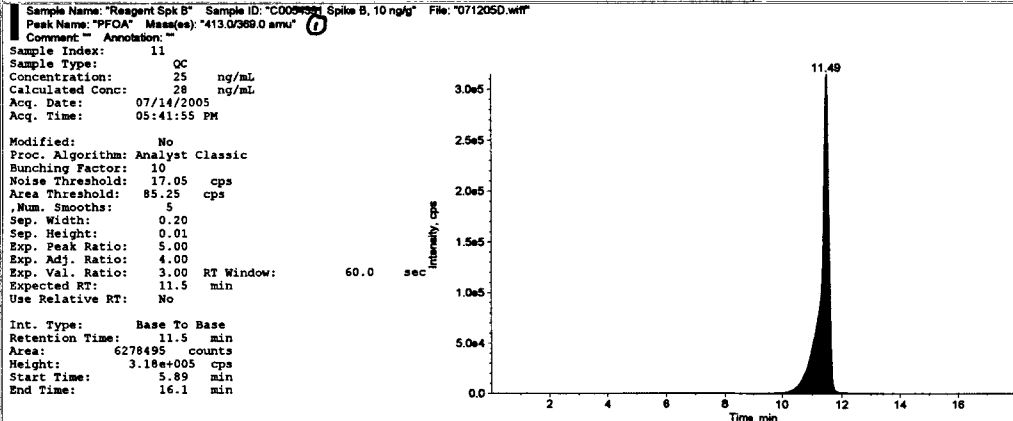
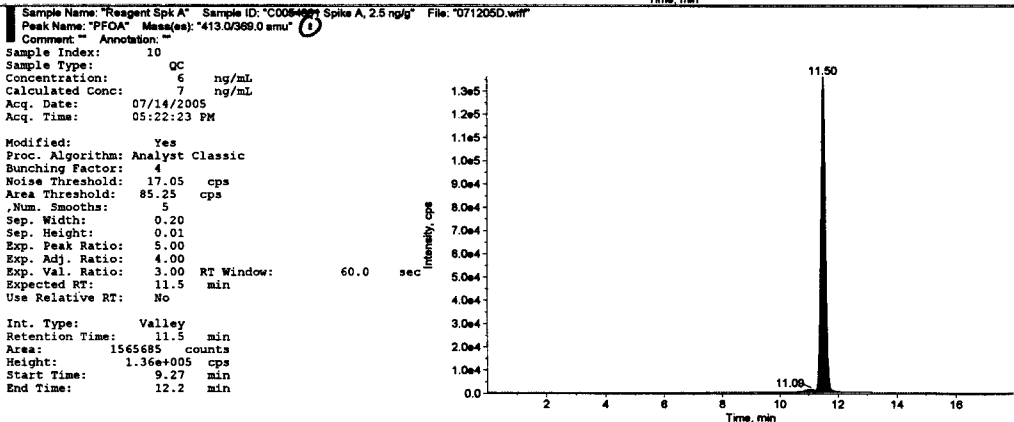
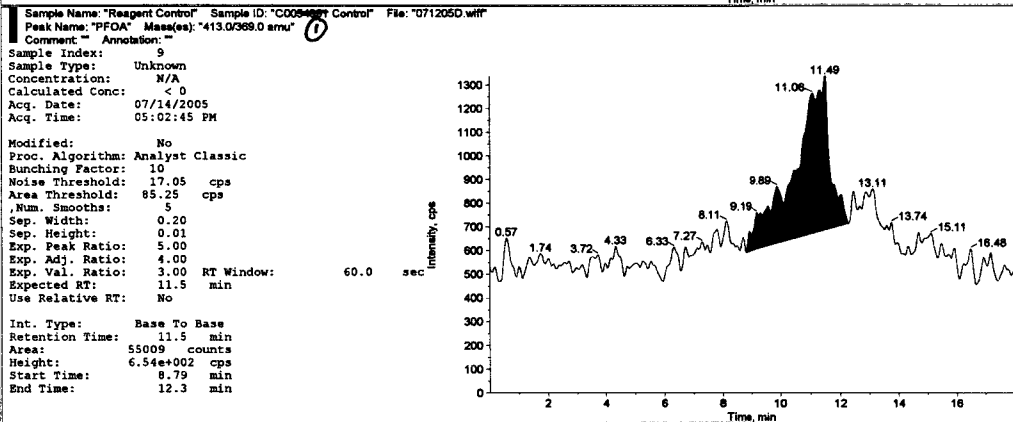
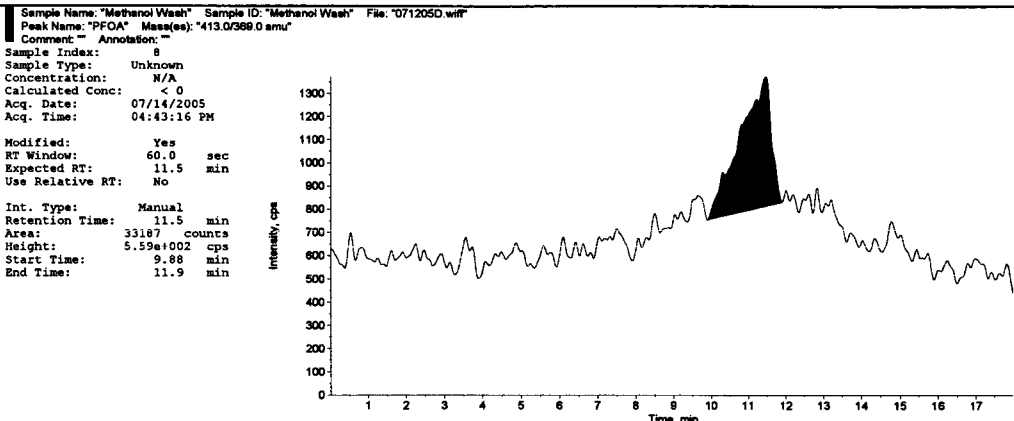
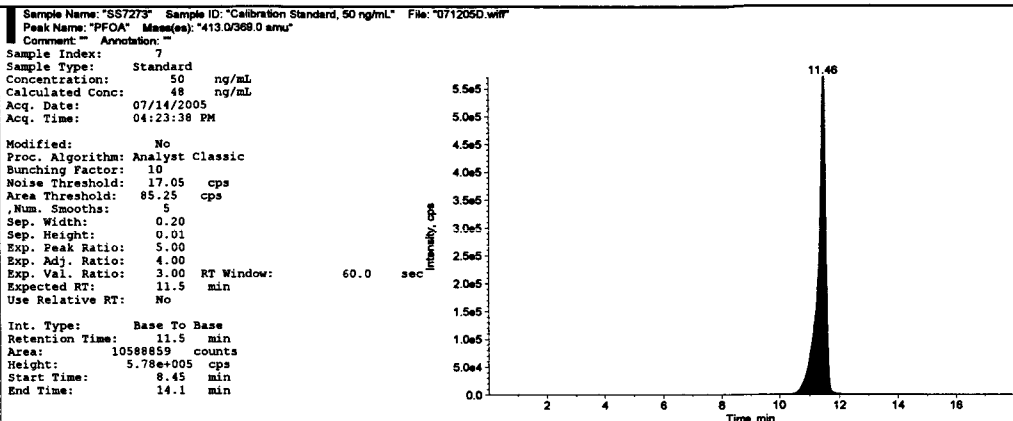
AMB 07/19/05





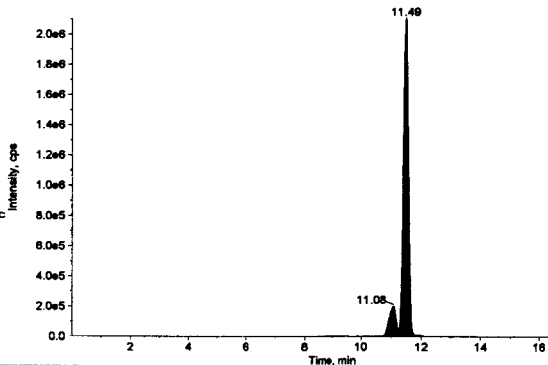
Initials AMB
 Date 07/19/05
 Bun# 1 To 38
 Sample Index (C) AMB 07/19/05

Verified by: [Signature]
 Initials: [Signature] Date 09/22/05
 Sample Index: 1 To: 38

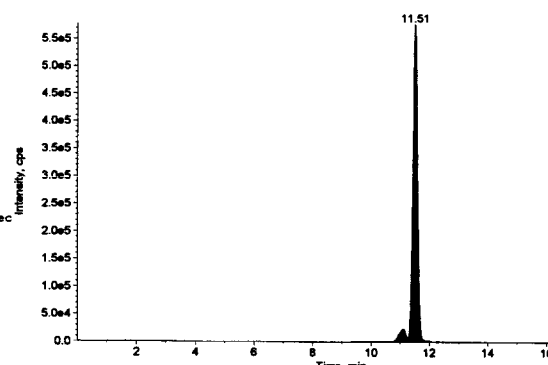


07 (RE) 73483 MLC 9/21/05

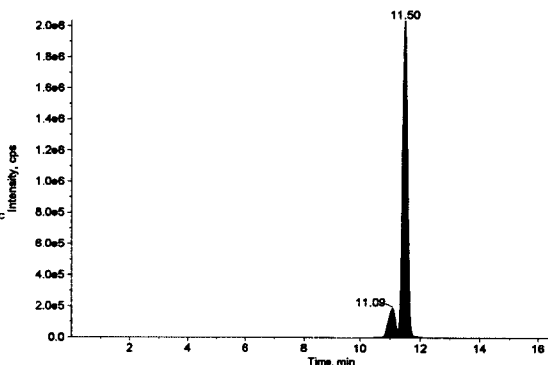
Sample Name: "C0049878 Spike C" Sample ID: "DF06V01AVP0010041007, 10 ng/g" File: "071205D.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: "Annotation: ""
Sample Index: 13
Sample Type: QC
Concentration: 25 ng/mL
Calculated Conc: 131 ng/mL
Acq. Date: 07/14/2005
Acq. Time: 06:21:03 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Valley
Retention Time: 11.5 min
Area: 28926901 counts
Height: 2.11e+006 cps
Start Time: 10.1 min
End Time: 18.0 min



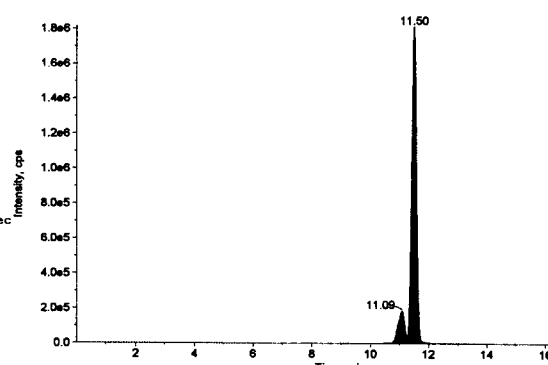
Sample Name: "C0049878 Spike D" Sample ID: "DF06V01AVP0010041007, 100 ng/g, DF=10" File: "071205D.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: "Annotation: ""
Sample Index: 14
Sample Type: QC
Concentration: 250 ng/mL
Calculated Conc: 322 ng/mL
Acq. Date: 07/14/2005
Acq. Time: 06:40:41 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 7195952 counts
Height: 5.79e+005 cps
Start Time: 7.14 min
End Time: 14.1 min



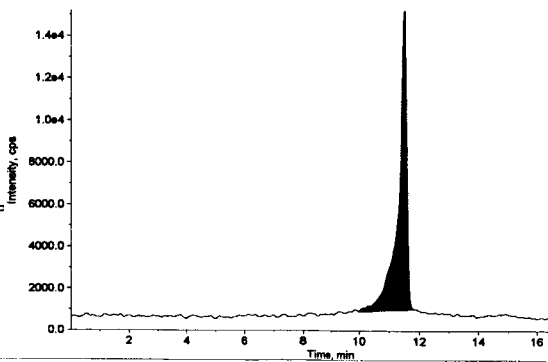
Sample Name: "C0049878" Sample ID: "DF06V01AVP0010041007" File: "071205D.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: "Annotation: ""
Sample Index: 15
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 124 ng/mL
Acq. Date: 07/14/2005
Acq. Time: 07:00:16 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 27411925 counts
Height: 2.04e+006 cps
Start Time: 10.2 min
End Time: 16.7 min



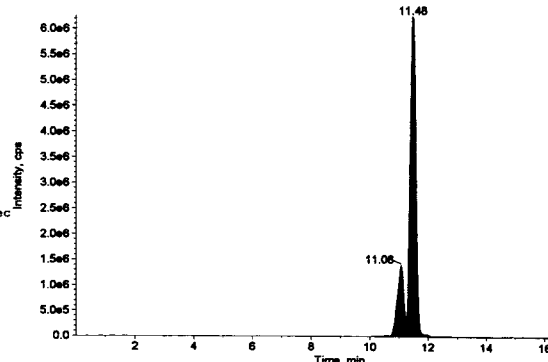
Sample Name: "C0049878 Rep" Sample ID: "DF06V01AVP0010041007" File: "071205D.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: "Annotation: ""
Sample Index: 16
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 113 ng/mL
Acq. Date: 07/14/2005
Acq. Time: 07:19:56 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 24894626 counts
Height: 1.82e+006 cps
Start Time: 10.0 min
End Time: 14.3 min



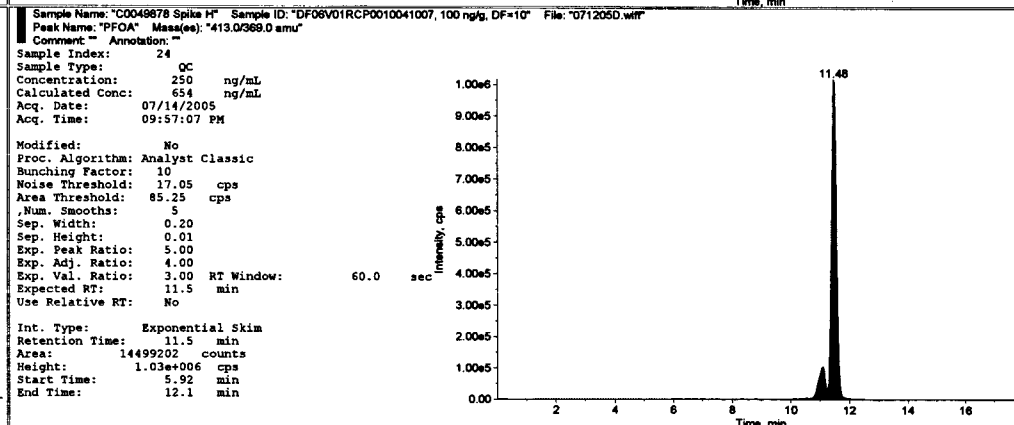
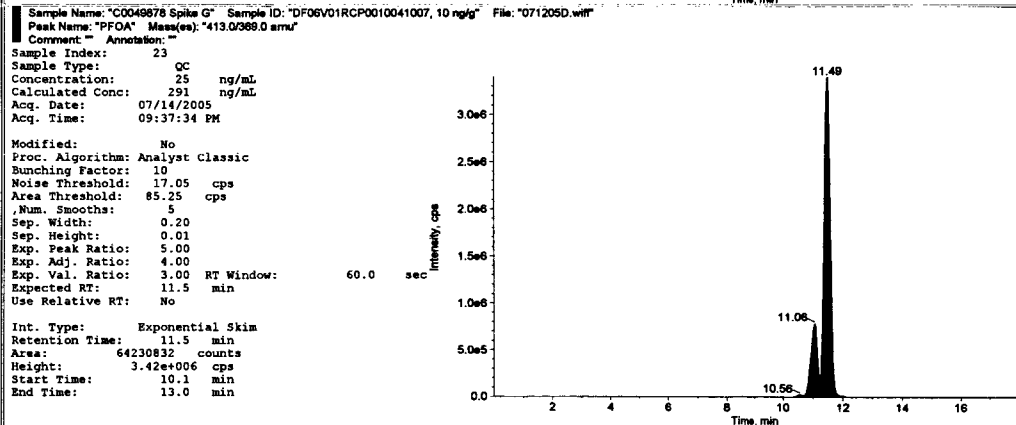
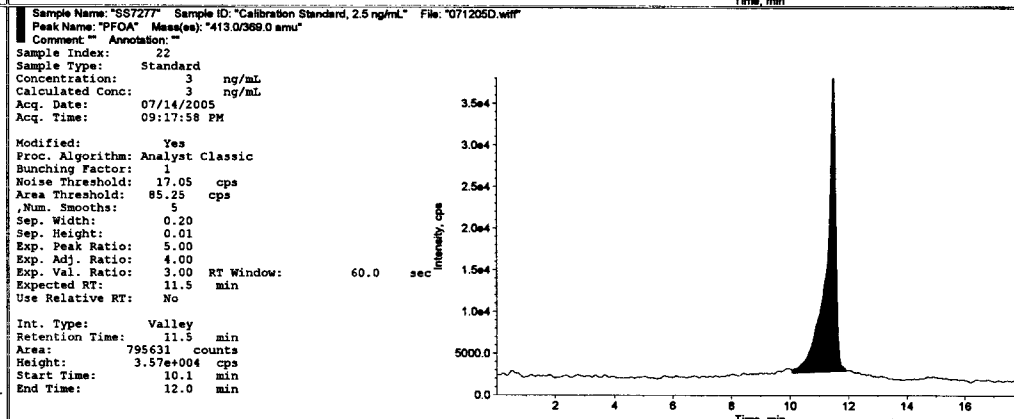
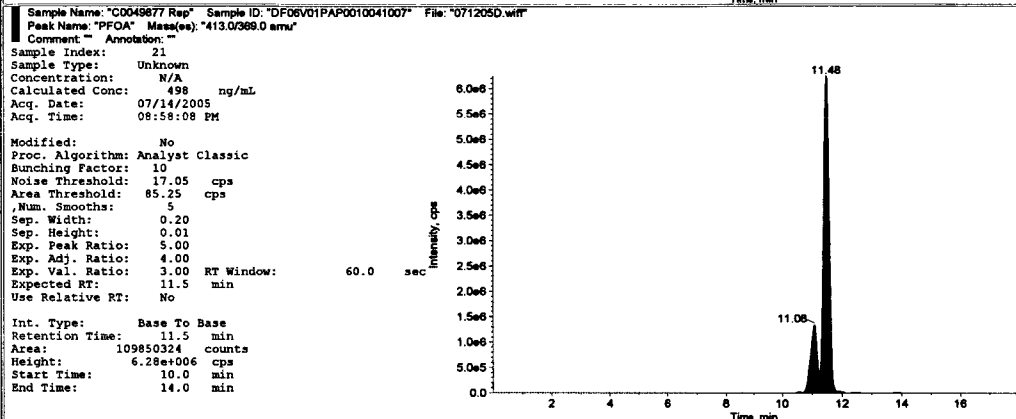
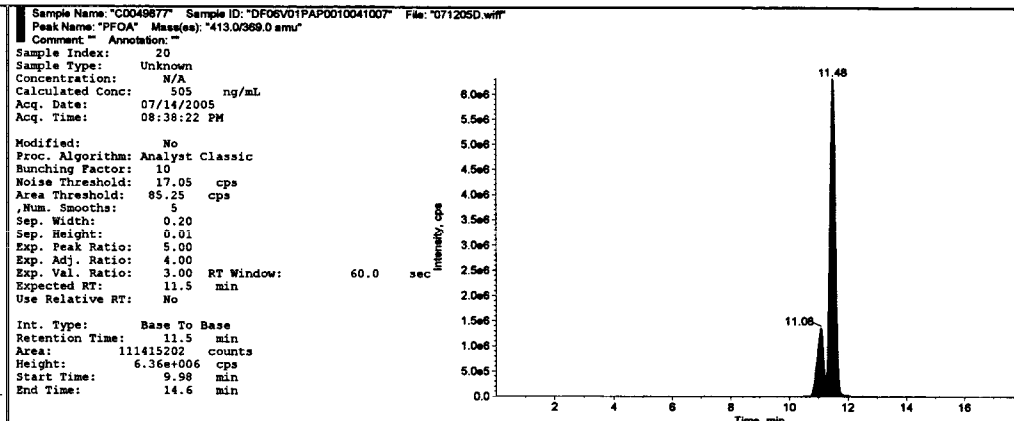
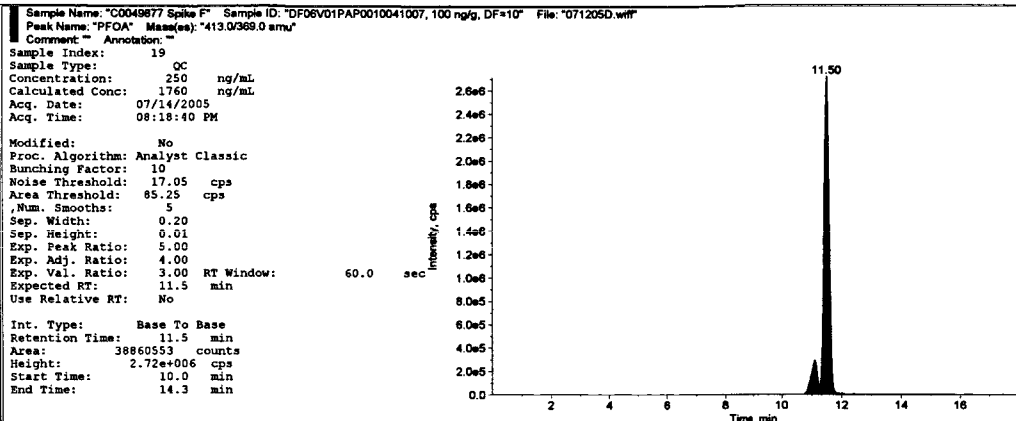
Sample Name: "SS7276" Sample ID: "Calibration Standard, 1.0 ng/mL" File: "071205D.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: "Annotation: ""
Sample Index: 17
Sample Type: Standard
Concentration: 1 ng/mL
Calculated Conc: 1 ng/mL
Acq. Date: 07/14/2005
Acq. Time: 07:39:32 PM
Modified: Yes
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Valley
Retention Time: 11.5 min
Area: 279854 counts
Height: 1.43e+004 cps
Start Time: 9.98 min
End Time: 11.9 min



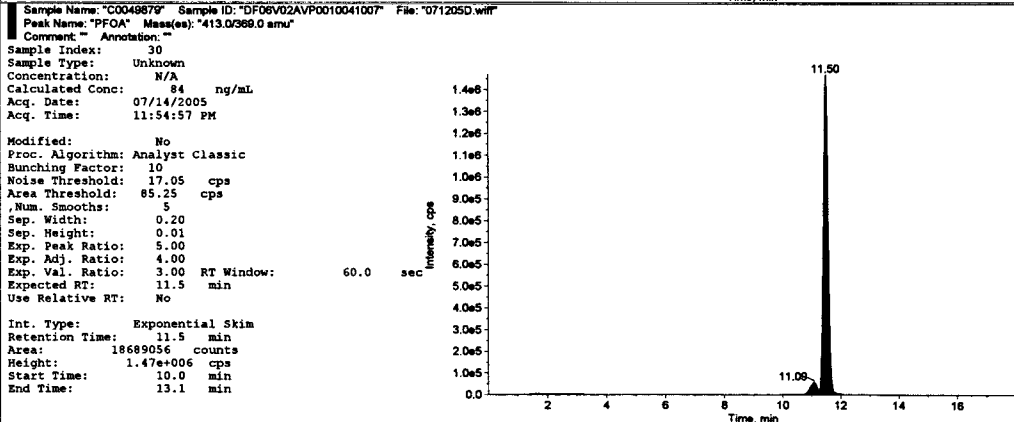
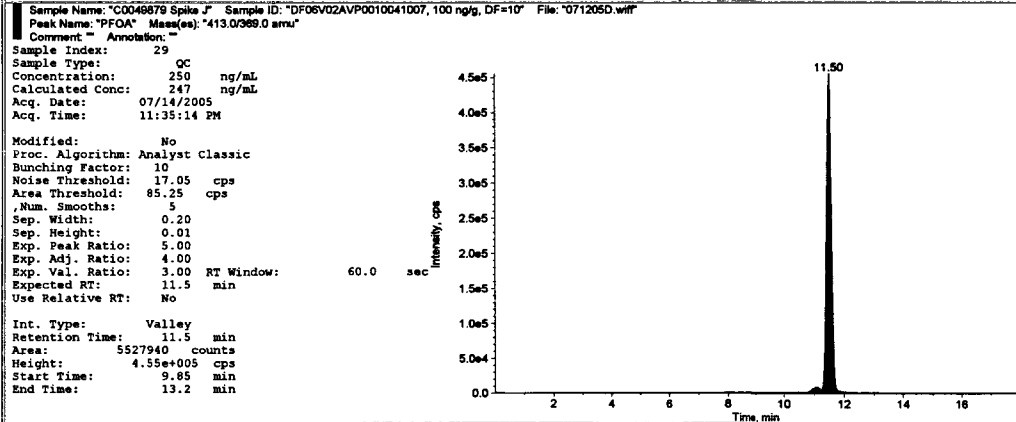
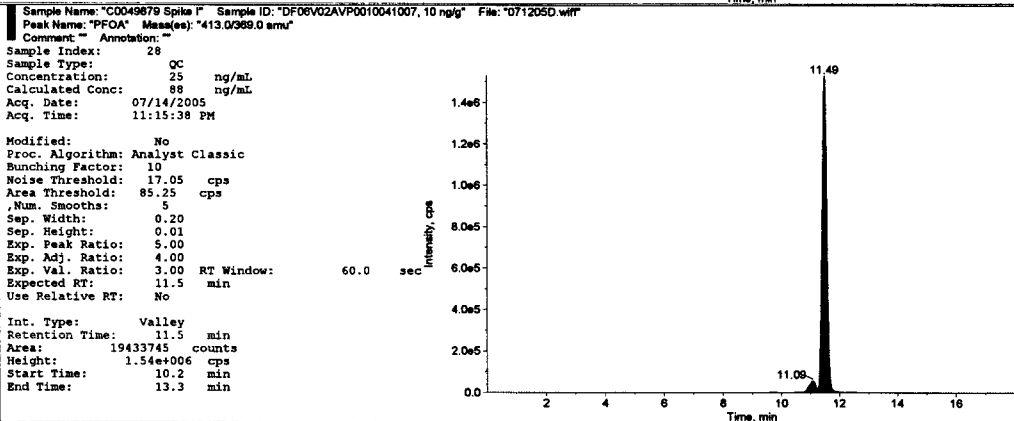
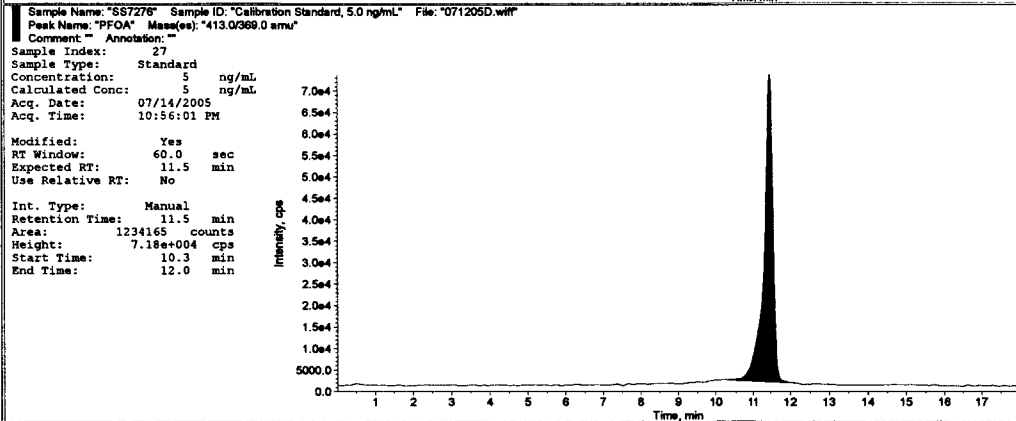
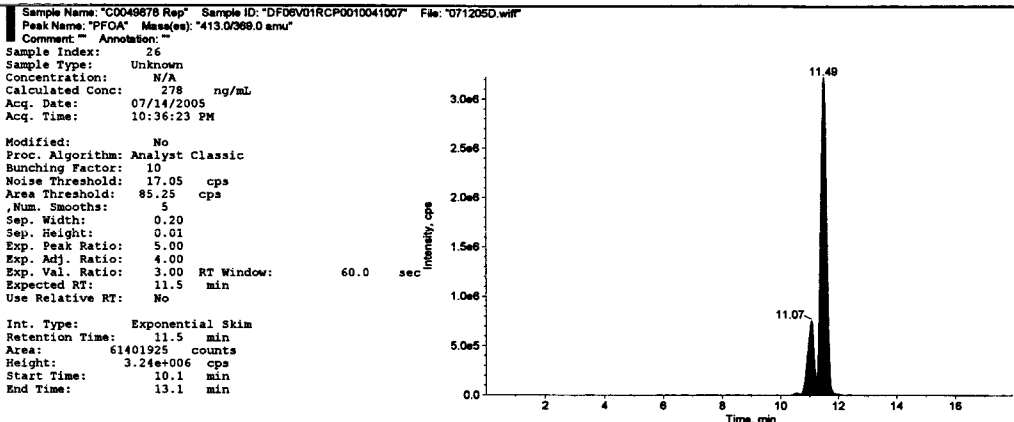
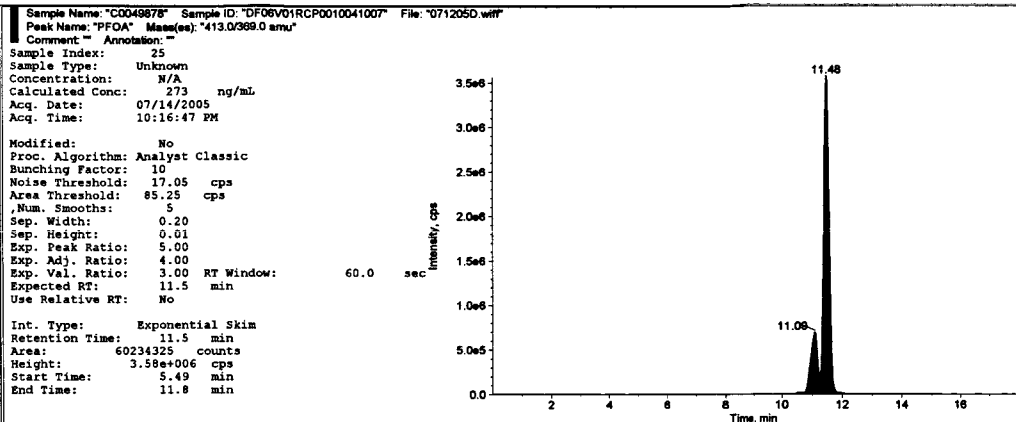
Sample Name: "C0049877 Spike E" Sample ID: "DF06V01PAP0010041007, 10 ng/g" File: "071205D.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: "Annotation: ""
Sample Index: 18
Sample Type: QC
Concentration: 25 ng/mL
Calculated Conc: 508 ng/mL
Acq. Date: 07/14/2005
Acq. Time: 07:59:06 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 111947693 counts
Height: 6.30e+006 cps
Start Time: 10.1 min
End Time: 14.1 min



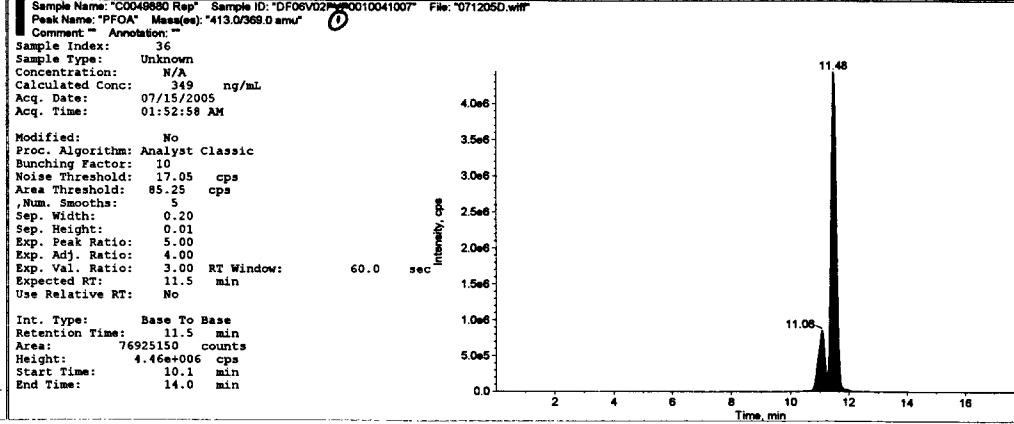
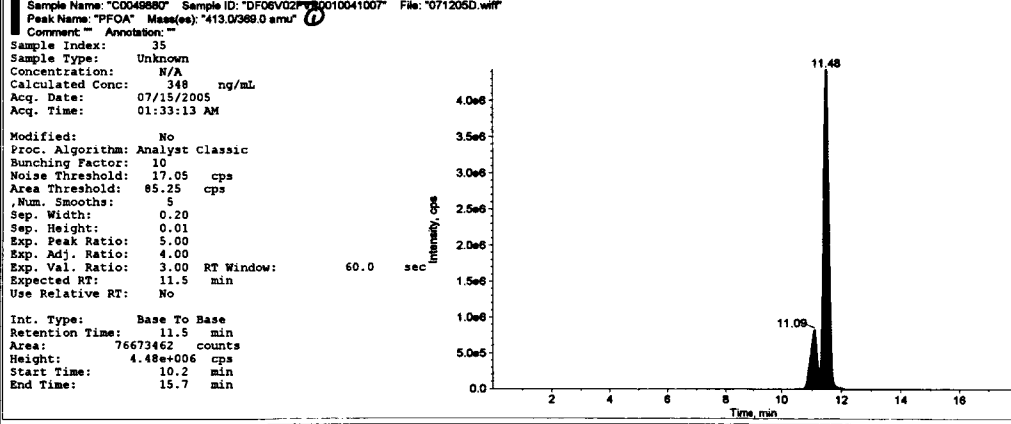
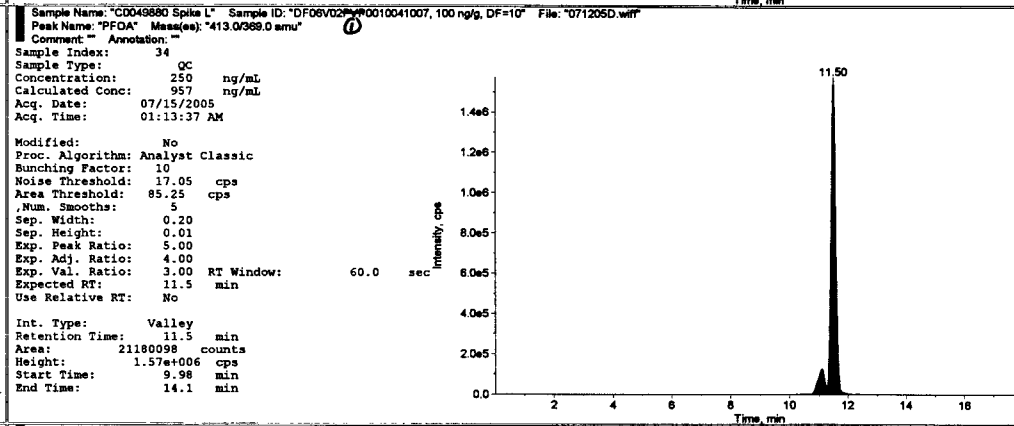
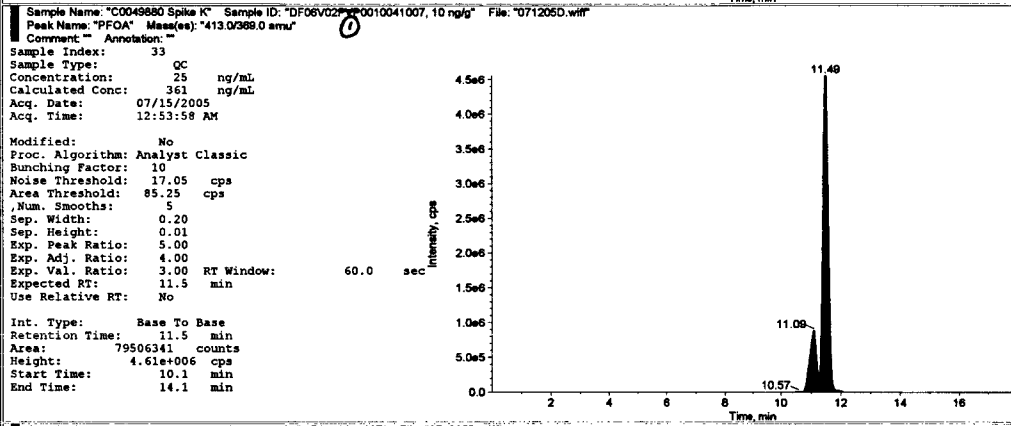
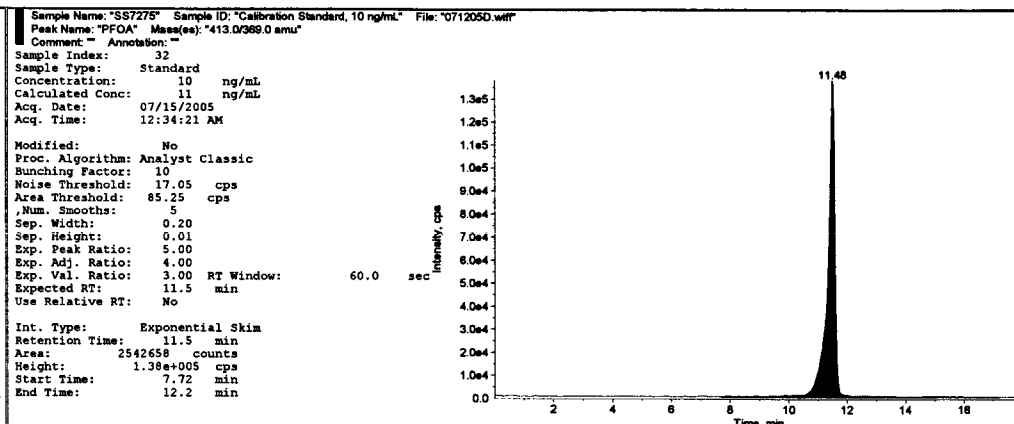
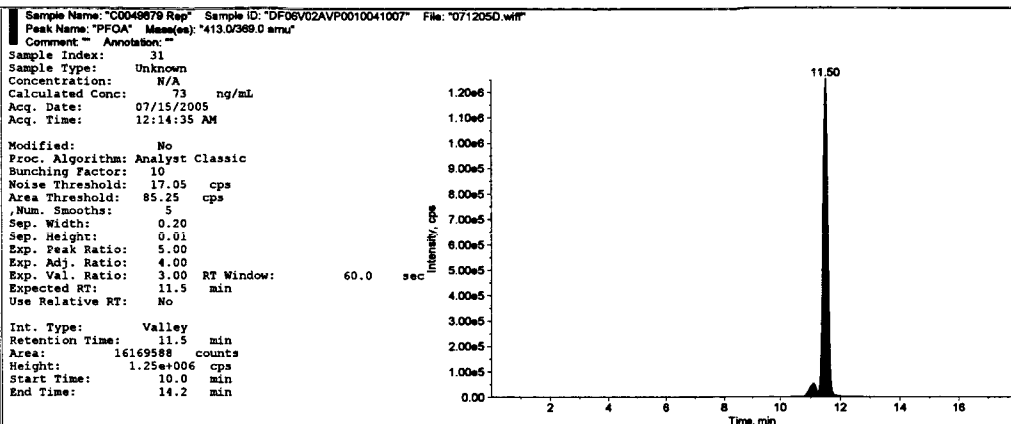
0251



0252



0253



0254

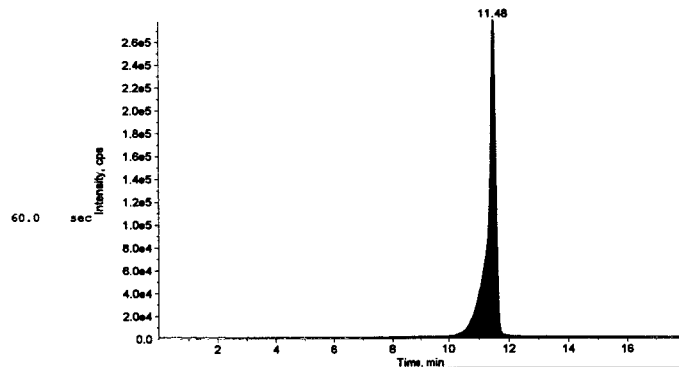
URE PAP m/c 9/21/05

Sample Name: "S57274" Sample ID: "Calibration Standard, 25 ng/mL" File: "071205D.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: "" Annotation: ""

Sample Index: 37
Sample Type: Standard
Concentration: 25 ng/mL
Calculated Conc: 25 ng/mL
Acq. Date: 07/15/2005
Acq. Time: 02:12:28 AM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
.Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No

Int. Type: Exponential Skim
Retention Time: 11.5 min
Area: 5535612 counts
Height: 2.80e+005 cps
Start Time: 7.14 min
End Time: 12.4 min

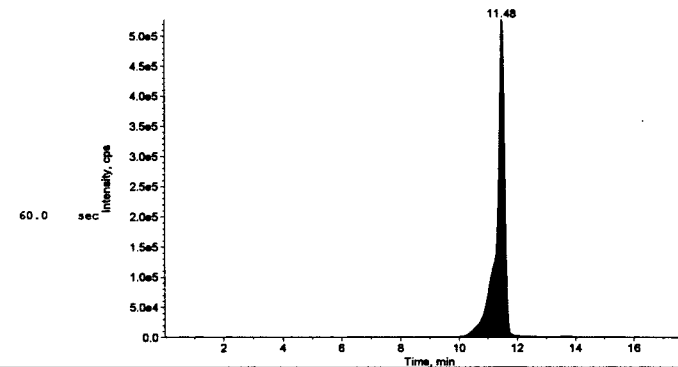


Sample Name: "S57273" Sample ID: "Calibration Standard, 50 ng/mL" File: "071205D.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: "" Annotation: ""

Sample Index: 38
Sample Type: Standard
Concentration: 50 ng/mL
Calculated Conc: 47 ng/mL
Acq. Date: 07/15/2005
Acq. Time: 02:32:20 AM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
.Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 11.5 min
Area: 10338038 counts
Height: 5.32e+005 cps
Start Time: 6.22 min
End Time: 13.9 min



CEE 07/28/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
1 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5124	0.500	0.375	071205D-101
2 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	7525	1.00	0.742	071205D-102
3 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	19472	2.50	2.57	071205D-103
4 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	39820	5.00	5.69	071205D-104
5 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	78150	10.0	11.6	071205D-105
6 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	173073	25.0	26.1	071205D-106
7 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	324747	50.0	49.3	071205D-107
8 Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	071205D-108
9 Reagent Control	C0073483 Control	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	071205D-109
10 Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFOA Confirm Ion	45692	6.25	6.59	071205D-110
11 Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFOA Confirm Ion	181044	25.0	27.3	071205D-111
12 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5864	0.500	0.488	071205D-112
13 C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	2186904	① 25.0	334.	071205D-113
14 C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	339187	① 25.0	515.	071205D-114
15 C0049876	DF06V01AVP0010041007	Unknown	1	PFOA Confirm Ion	2114215	① N/A	323.	071205D-115
16 C0049876 Rep	DF06V01AVP0010041007	Unknown	1	PFOA Confirm Ion	1973341	① N/A	302.	071205D-116
17 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	7862	1.00	0.794	071205D-117
18 C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	11085187	① 25.0	1700.	071205D-118
19 C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	2315485	① 25.0	3540.	071205D-119
20 C0049877	DF06V01PAP0010041007	Unknown	1	PFOA Confirm Ion	11180365	① N/A	1710.	071205D-120
21 C0049877 Rep	DF06V01PAP0010041007	Unknown	1	PFOA Confirm Ion	10797565	① N/A	1650.	071205D-121
22 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	25797	2.50	3.54	071205D-122
23 C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	6257622	① 25.0	958.	071205D-123
24 C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	914744	① 25.0	1400.	071205D-124
25 C0049878	DF06V01RCP0010041007	Unknown	1	PFOA Confirm Ion	5962671	① N/A	912.	071205D-125
26 C0049878 Rep	DF06V01RCP0010041007	Unknown	1	PFOA Confirm Ion	6229410	① N/A	953.	071205D-126
27 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	34350	5.00	4.85	071205D-127
28 C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	833604	① 25.0	127.	071205D-128
29 C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	188799	① 25.0	285.	071205D-129
30 C0049879	DF06V02AVP0010041007	Unknown	1	PFOA Confirm Ion	823847	① N/A	126.	071205D-130
31 C0049879 Rep	DF06V02AVP0010041007	Unknown	1	PFOA Confirm Ion	734342	① N/A	112.	071205D-131
32 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	72660	10.0	10.7	071205D-132
33 C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	7523563	① 25.0	1150.	071205D-133
34 C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	1193542	① 25.0	1820.	071205D-134
35 C0049880	DF06V02PVP0010041007	Unknown	1	PFOA Confirm Ion	7086802	① N/A	1080.	071205D-135
36 C0049880 Rep	DF06V02PVP0010041007	Unknown	1	PFOA Confirm Ion	7020974	① N/A	1070.	071205D-136
37 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	167552	25.0	25.2	071205D-137
38 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	303410	50.0	46.0	071205D-138

① Dilution required, see data set 071205DR - BAK 07/28/05

② ② PAP MLC 9/21/05

Vegetation Conversion

Compound: PFOA Confirm. Ion

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049876 Spk D	DF06-V01-AVP001-0-041007	515	206
C0049879 Spk J	DF06-V02-AVP001-0-041007	285	114

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = ~~Peak Area 0 AT OR ABOVE 0.2 ng/g~~ - ①

NQ = Not quantifiable = Measured concentration ⁿ below Limit of Quantitation (LOQ) which is 0.5 ng/g.

BETWEEN 0.2 ng/g AND ①

4 ①

① RE mlc 9/20/05

0257

BAK 07/29/05

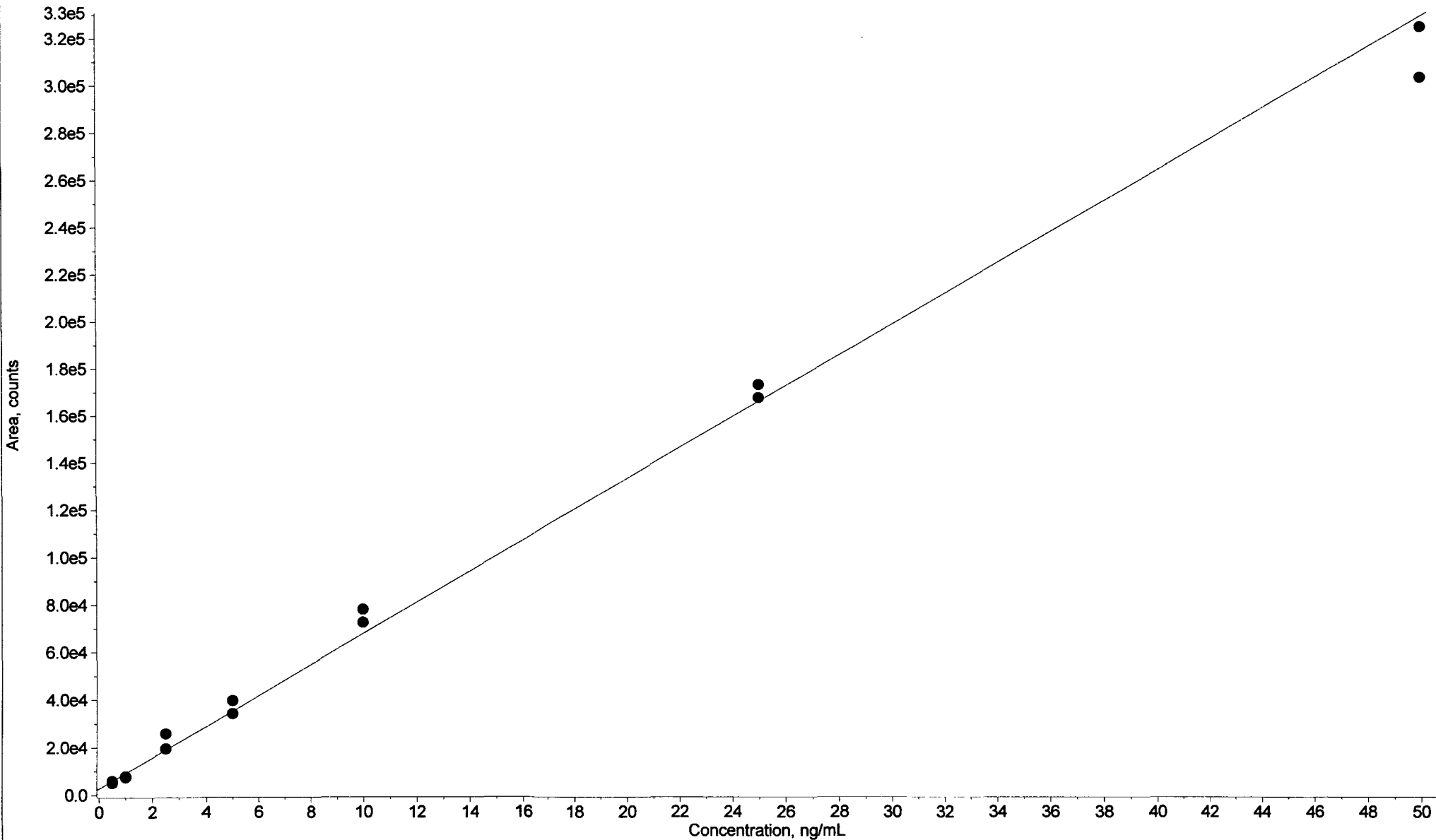
	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound:	PFOA Confirm. Ion	Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049876 Spk D	DF06-V01-AVP001-0-041007	=Raw Data!H185	=(C9*2)/5
10				
11	C0049879 Spk J	DF06-V02-AVP001-0-041007	=Raw Data!H200	=(C11*2)/5
12				

0258

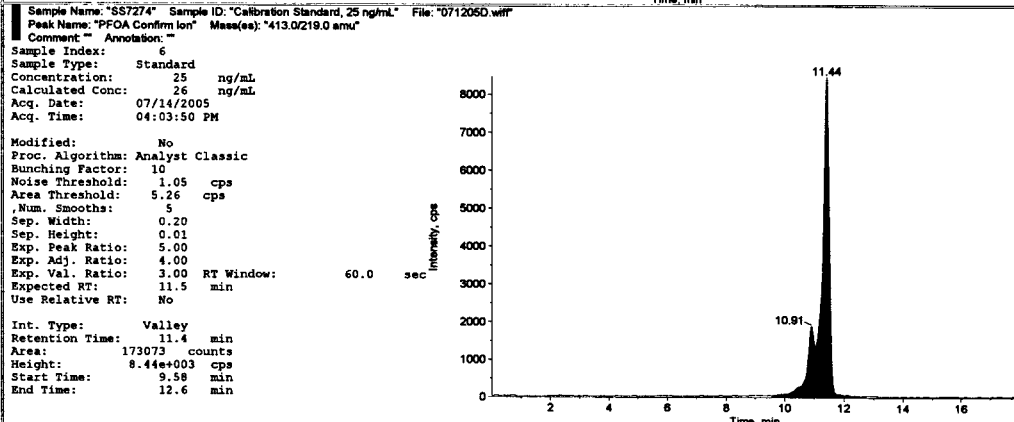
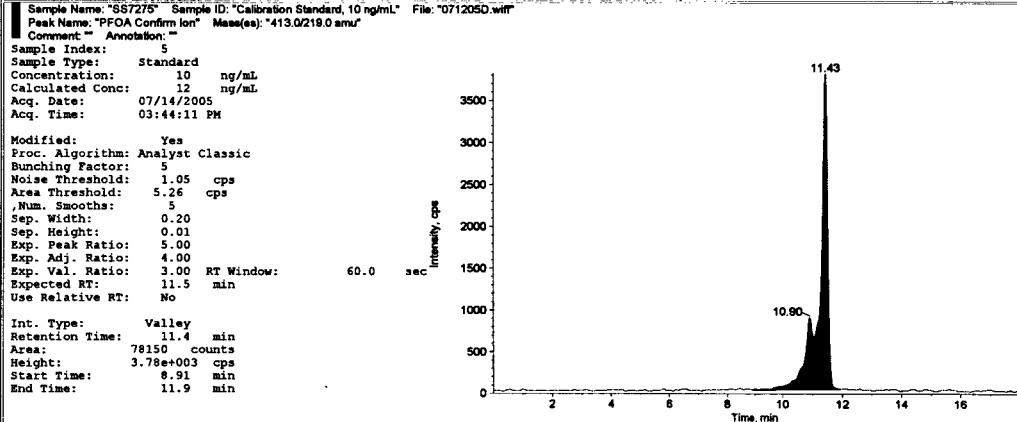
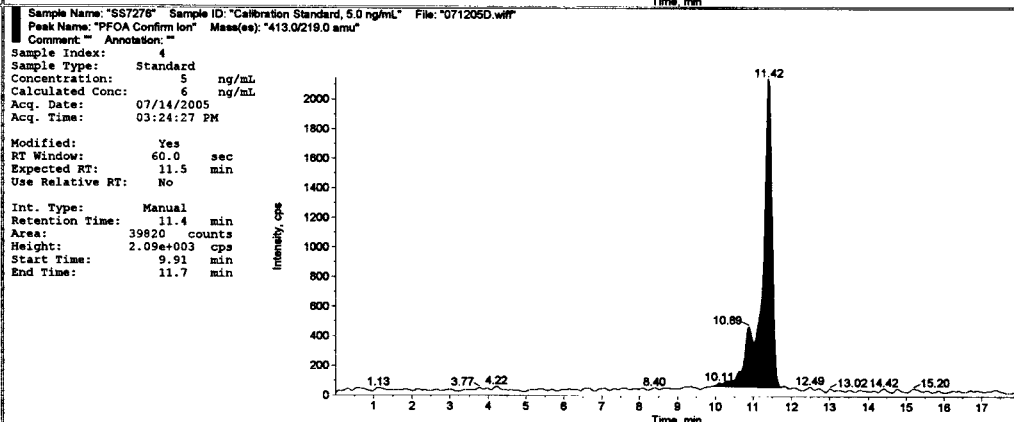
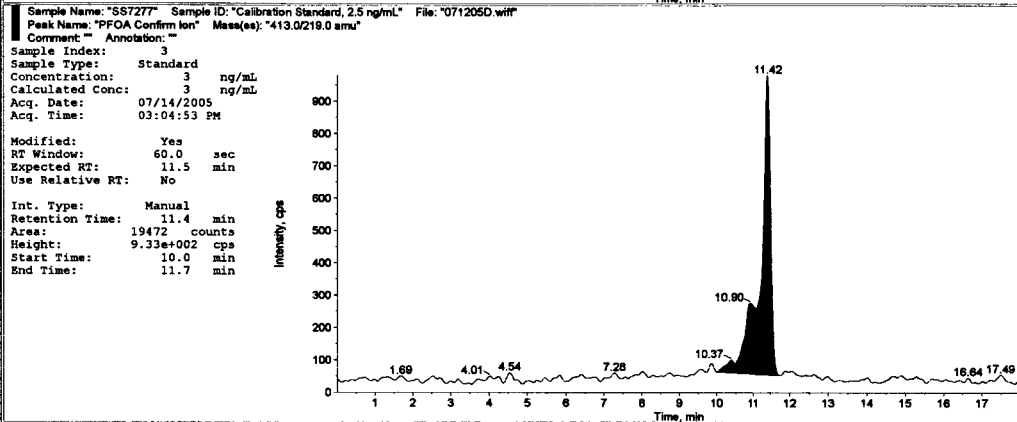
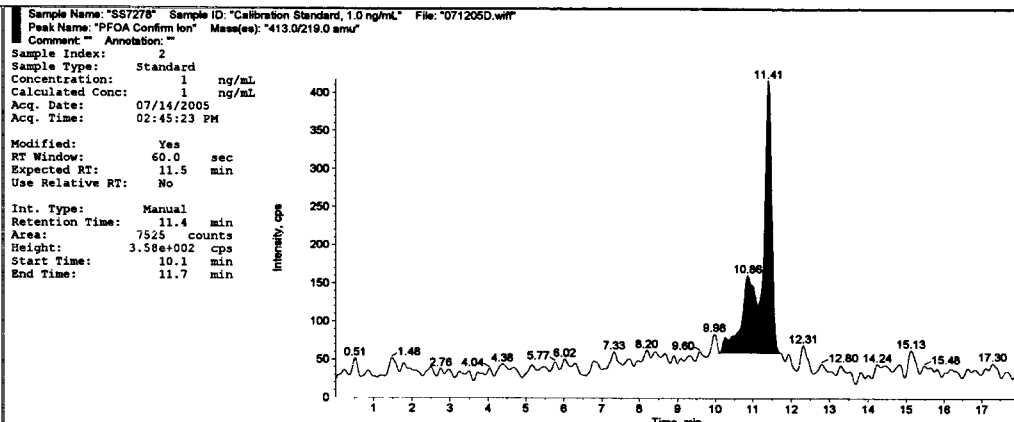
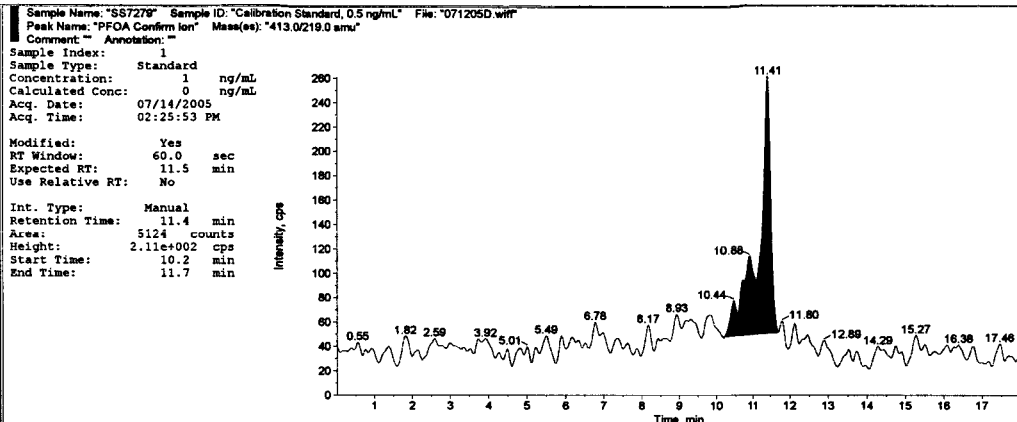
BAK 07/29/05

AMB 07/19/05

■ Untitled 2 (PFOA Confirm Ion): "Linear" Regression ("1 / x" weighting): $y = 6.53e+003 x + 2.68e+003$ ($r = 0.9959$)



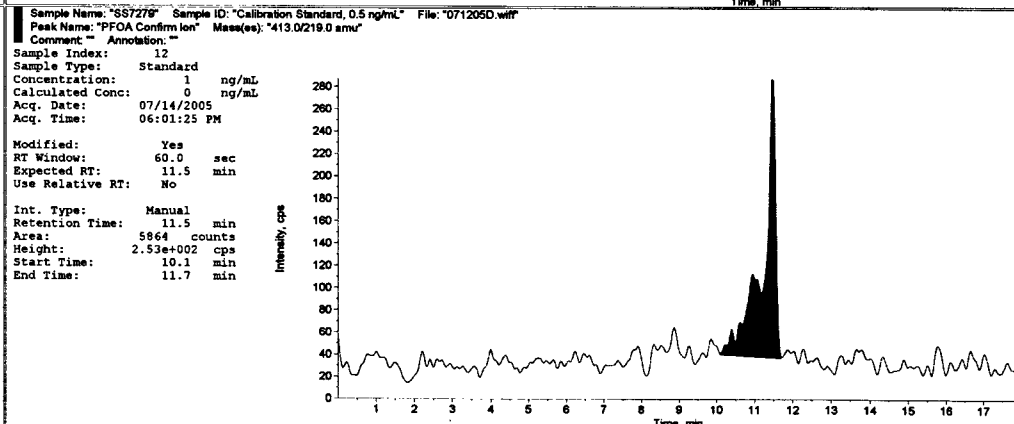
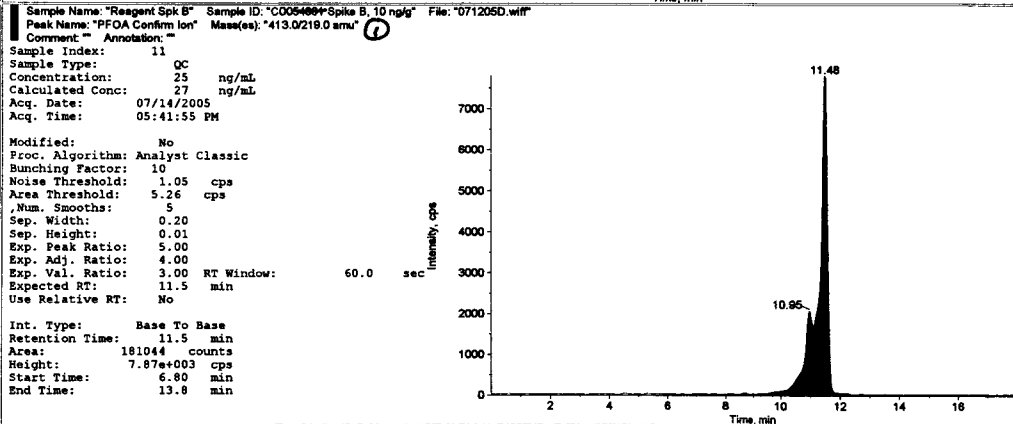
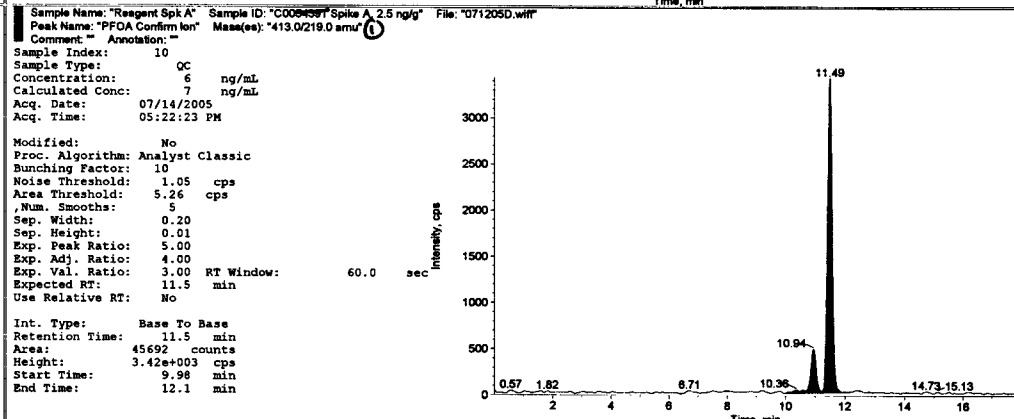
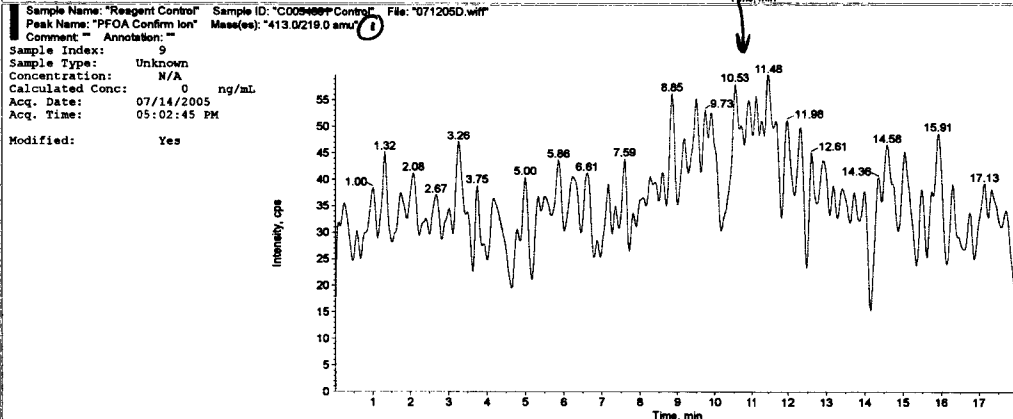
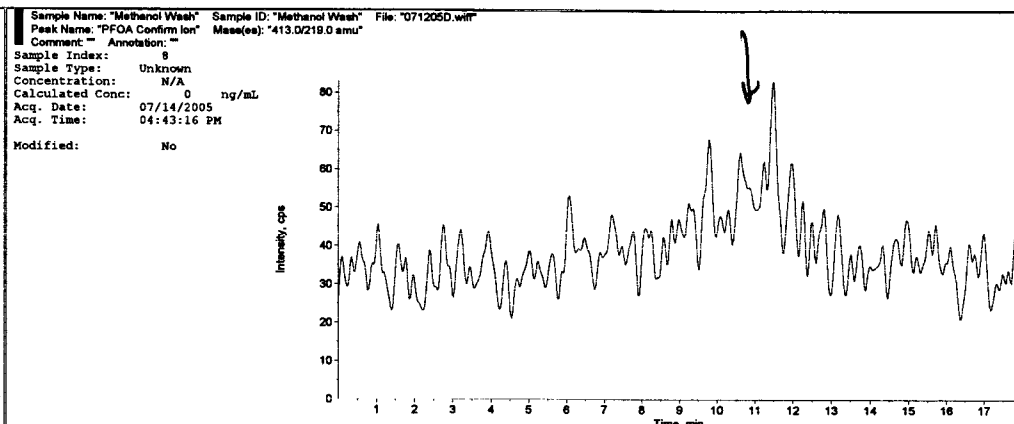
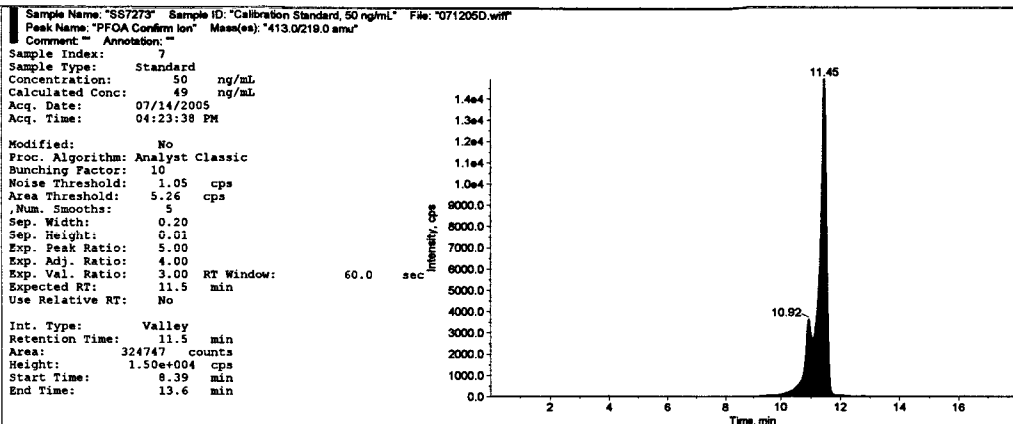
0259



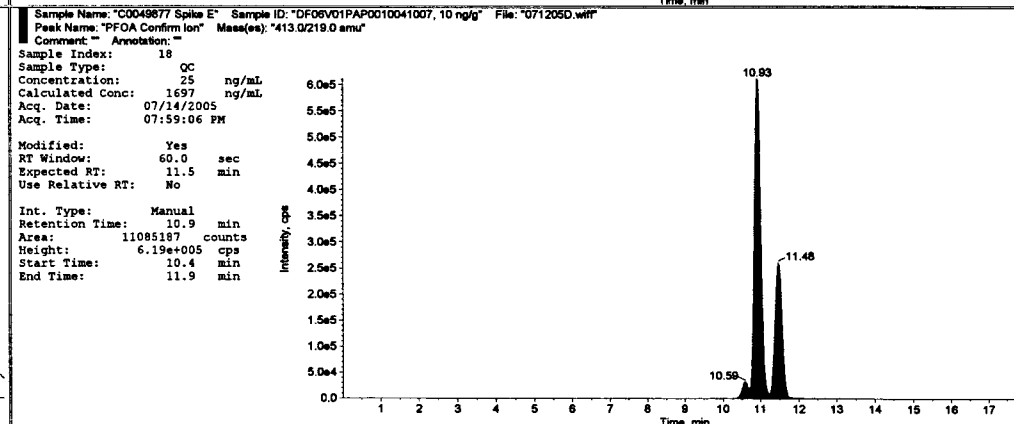
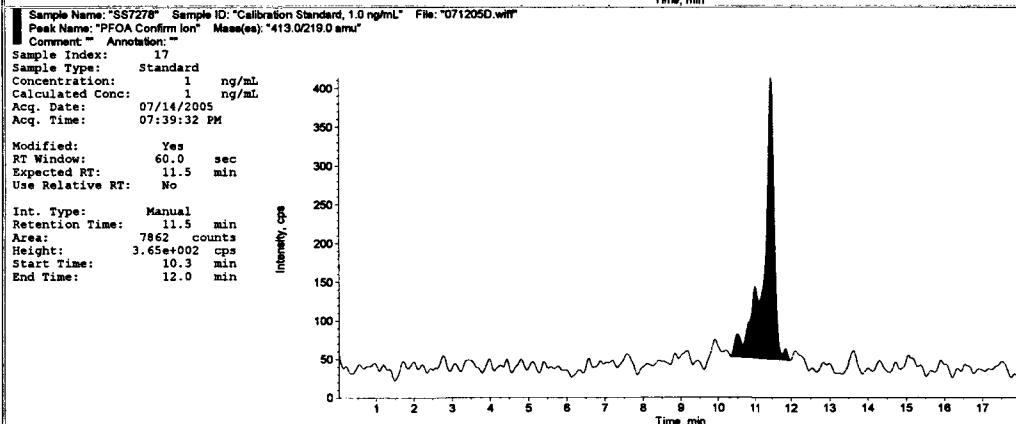
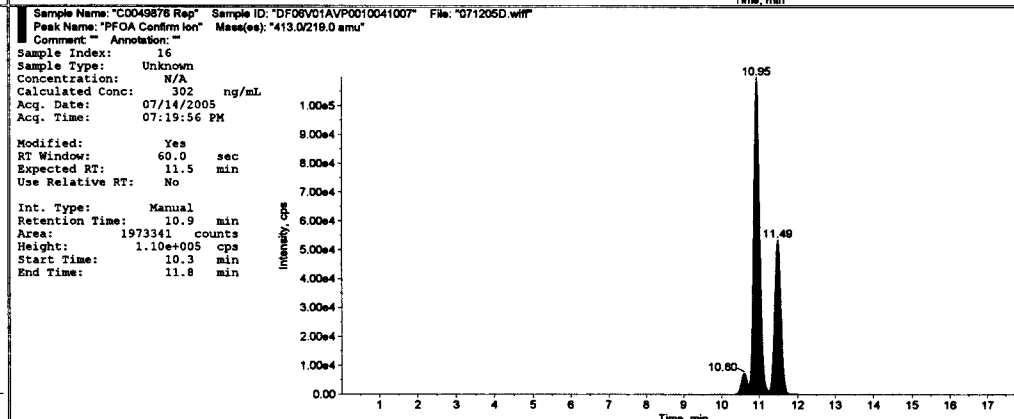
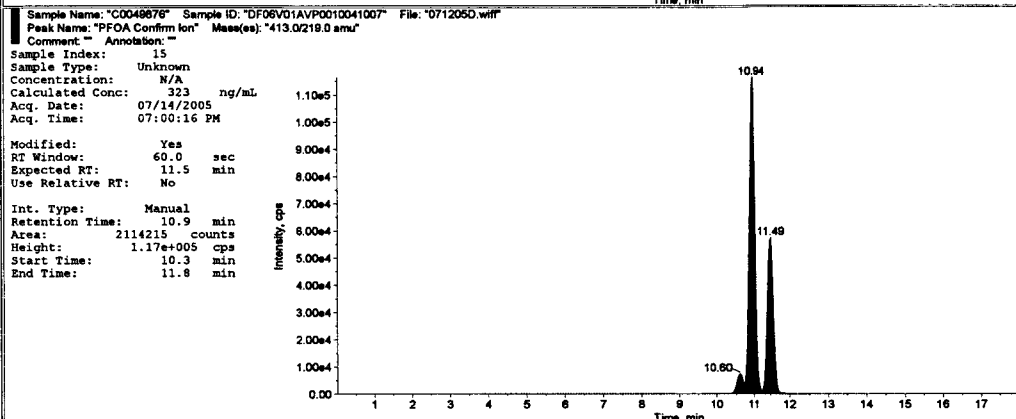
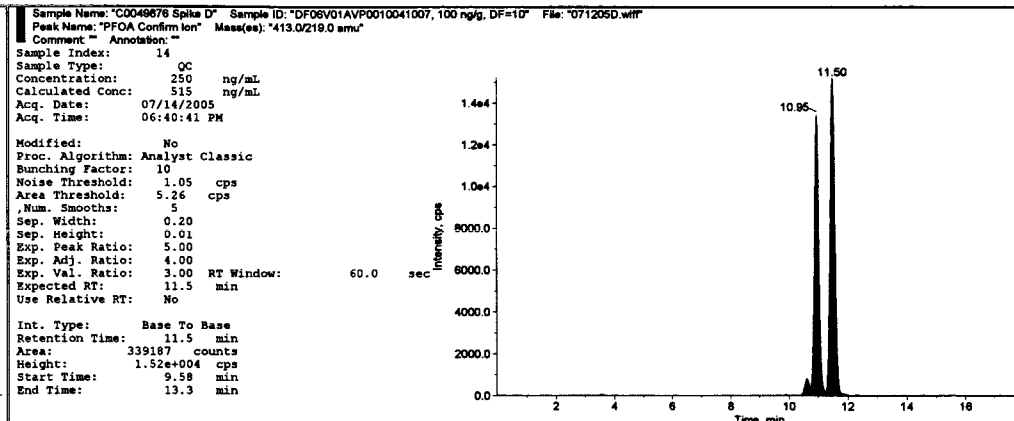
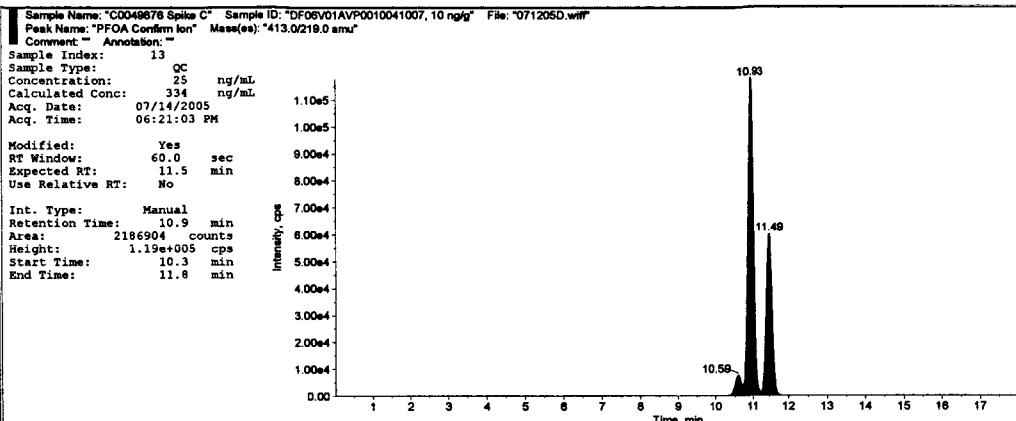
0260

Initials AMB
 Date 07/19/05
 Run# To 38
 Sample Index @AMB 07/19/05

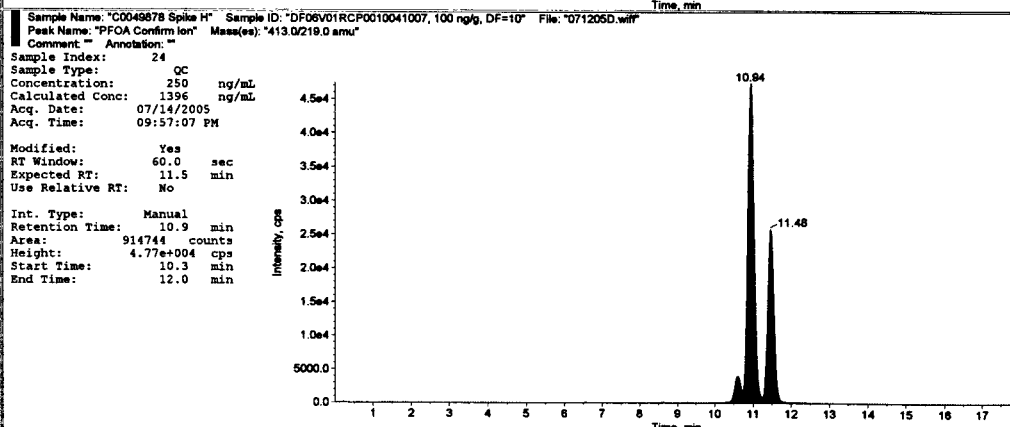
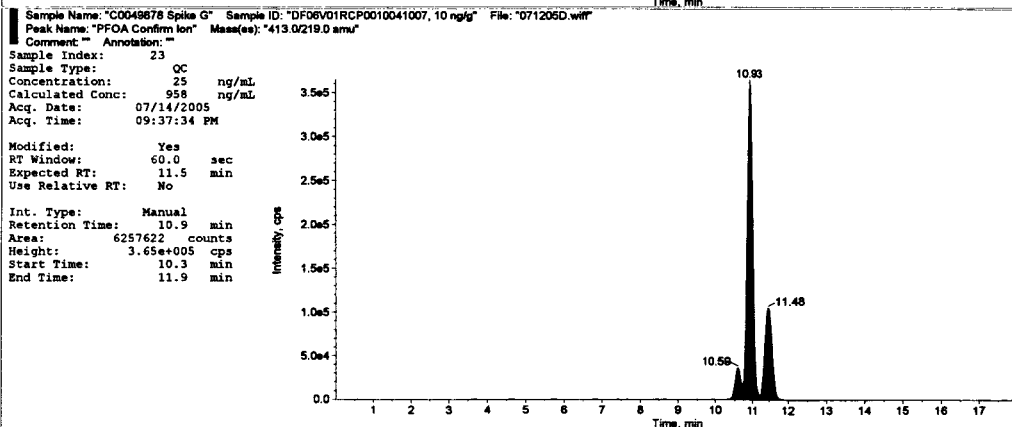
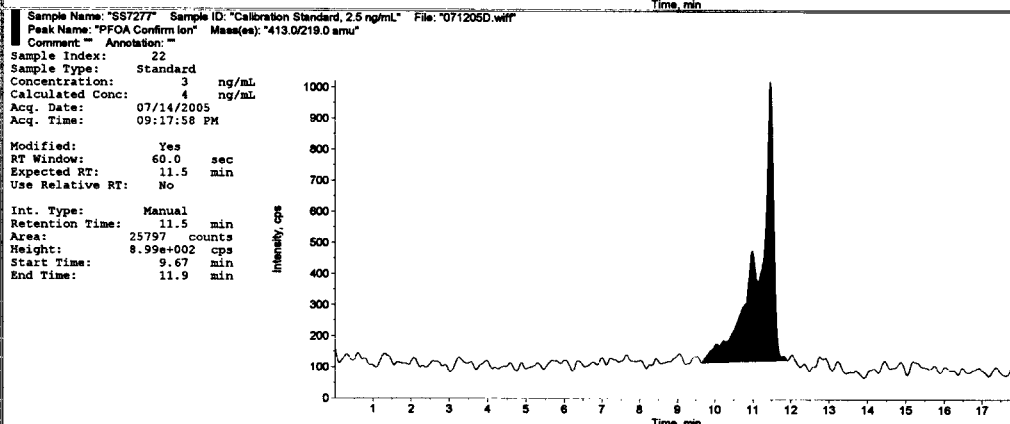
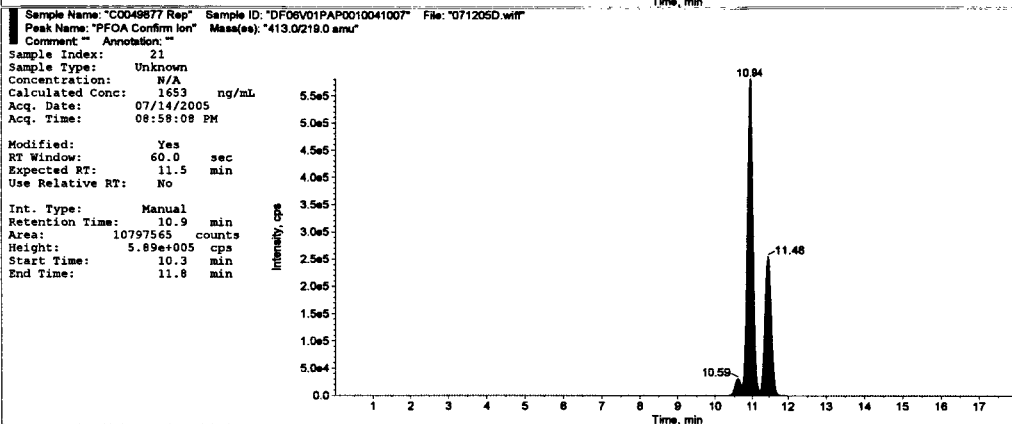
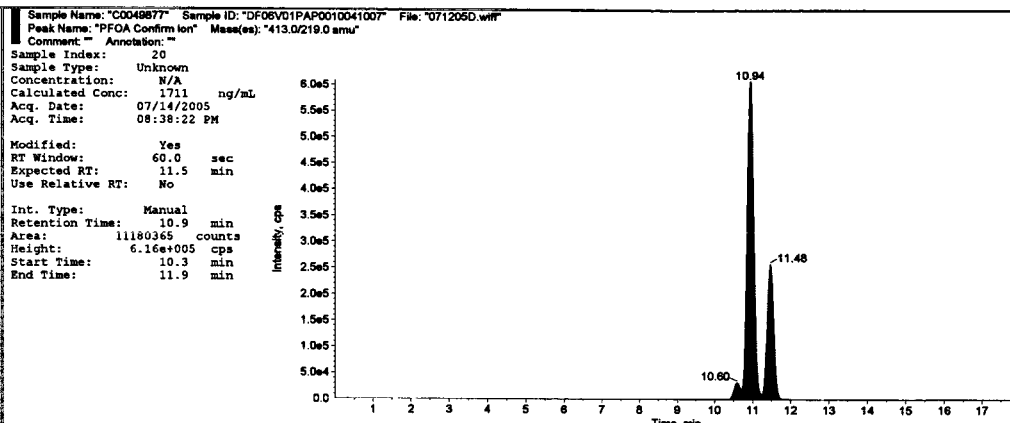
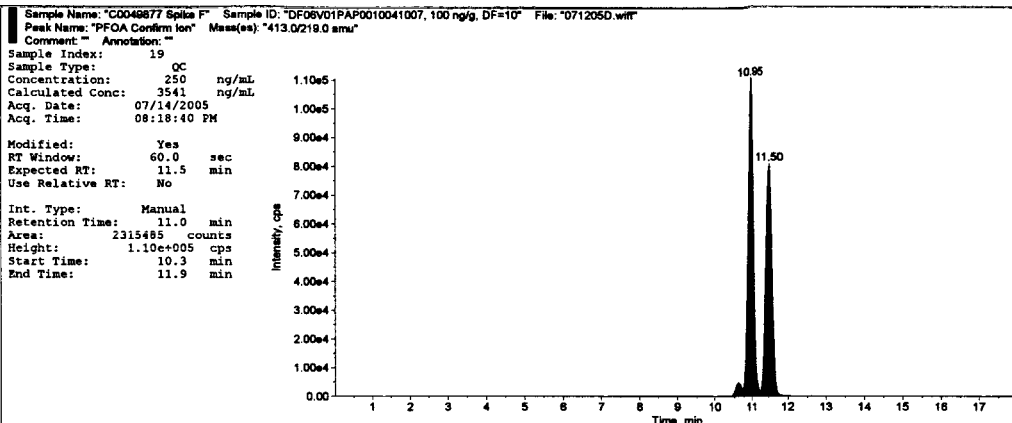
Verified by AS
 Initials: AS Date 09/22/05
 Sample Index: 1 To: 38

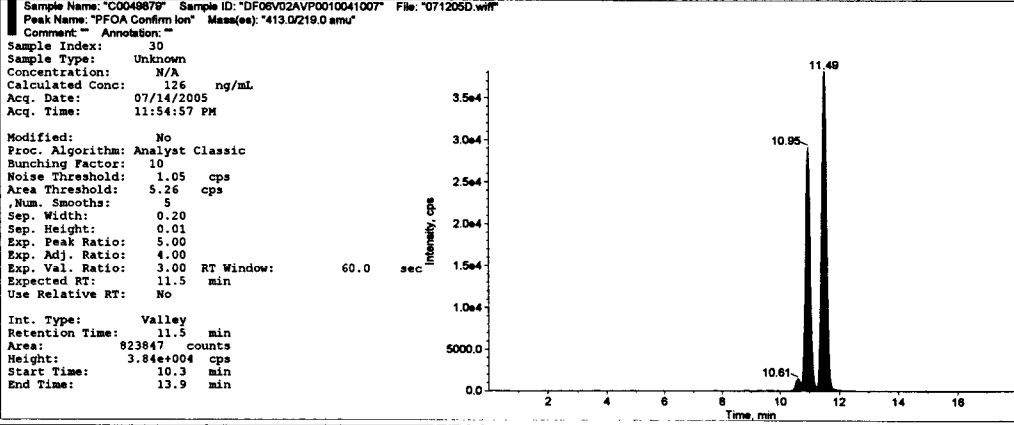
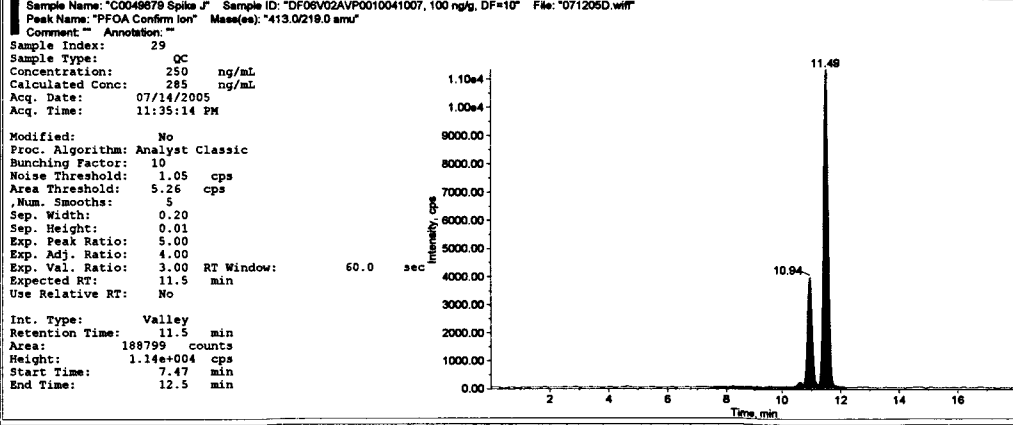
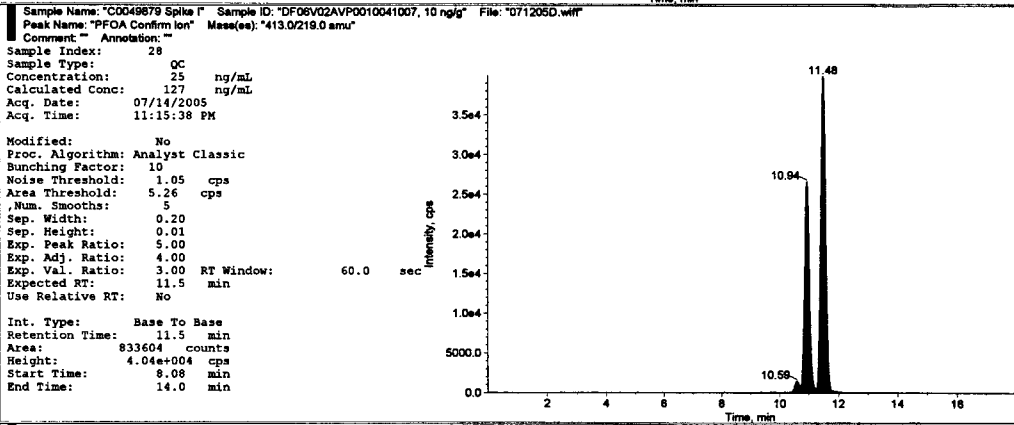
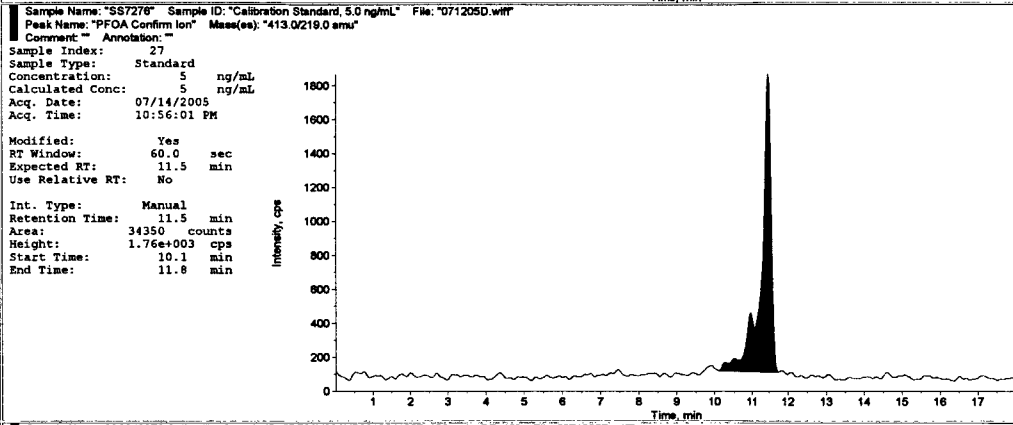
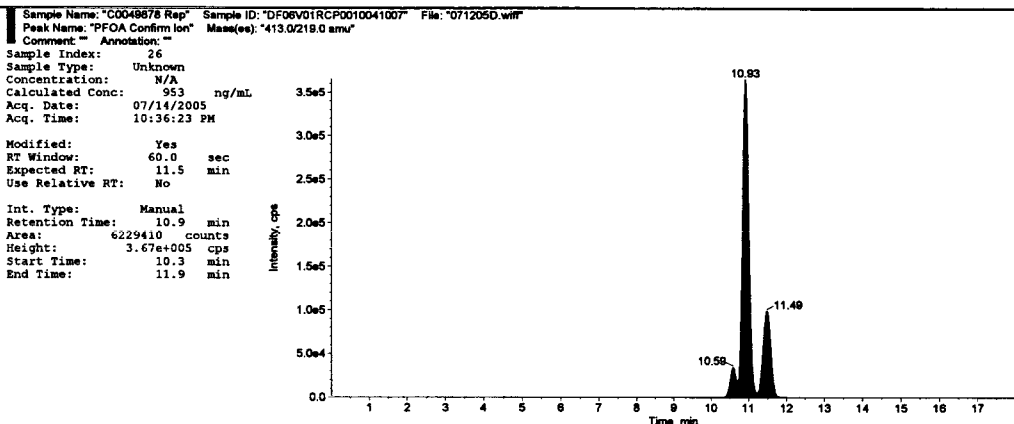
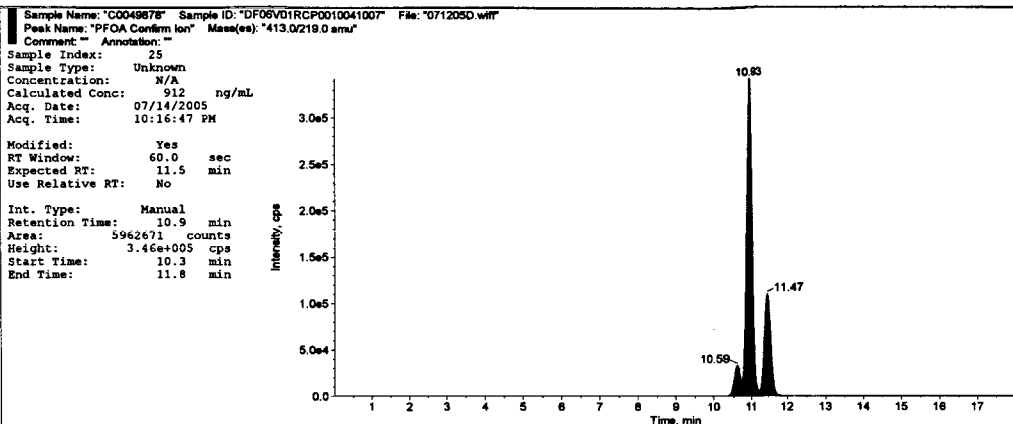


0201RE 73483 MLC 9/2/05
 1961

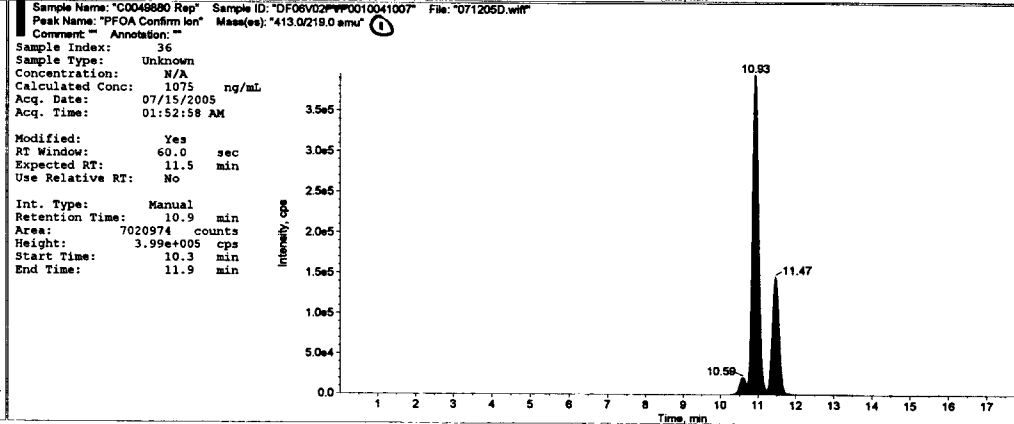
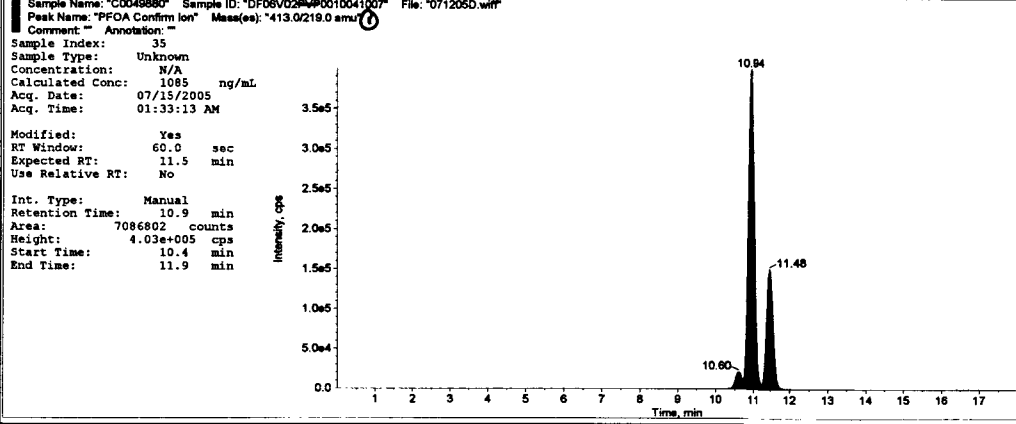
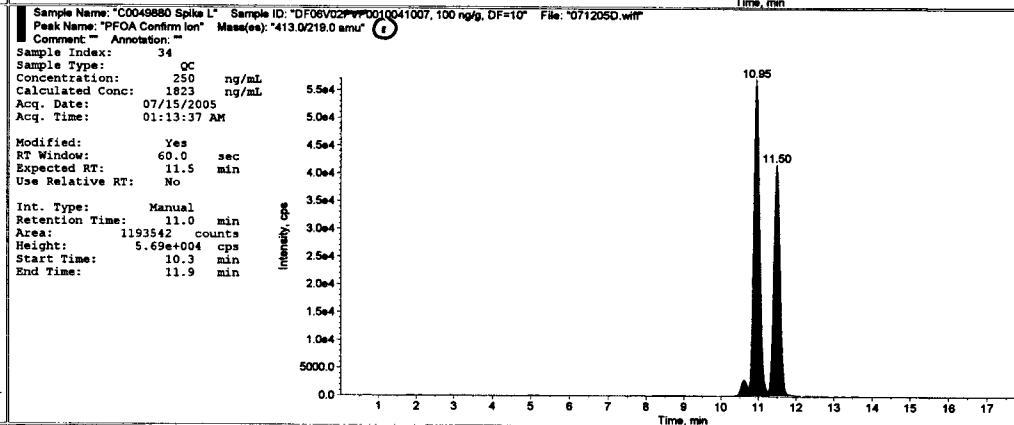
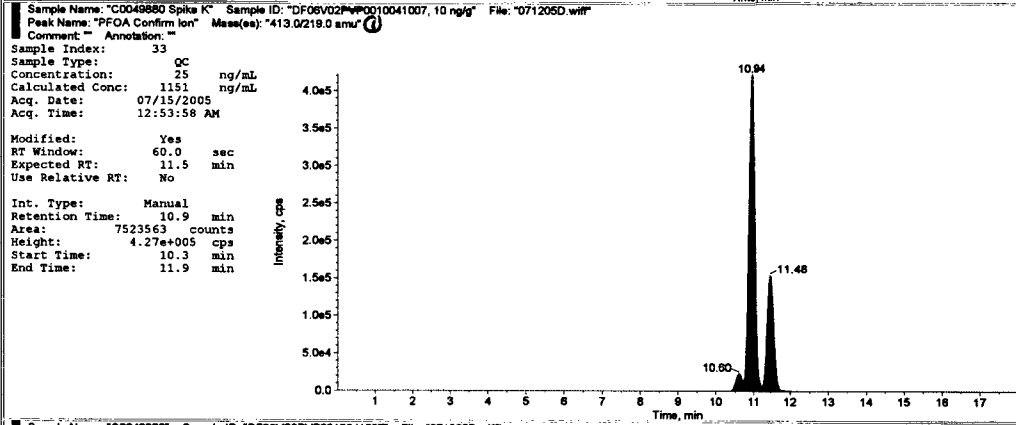
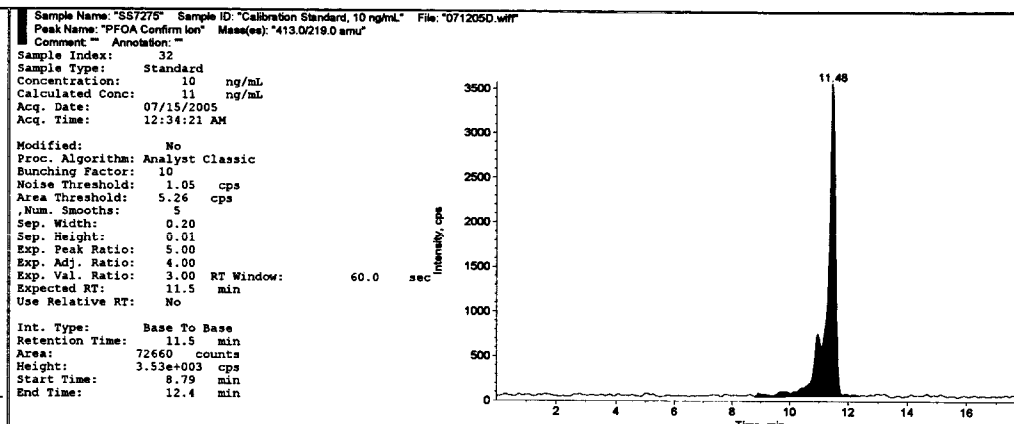
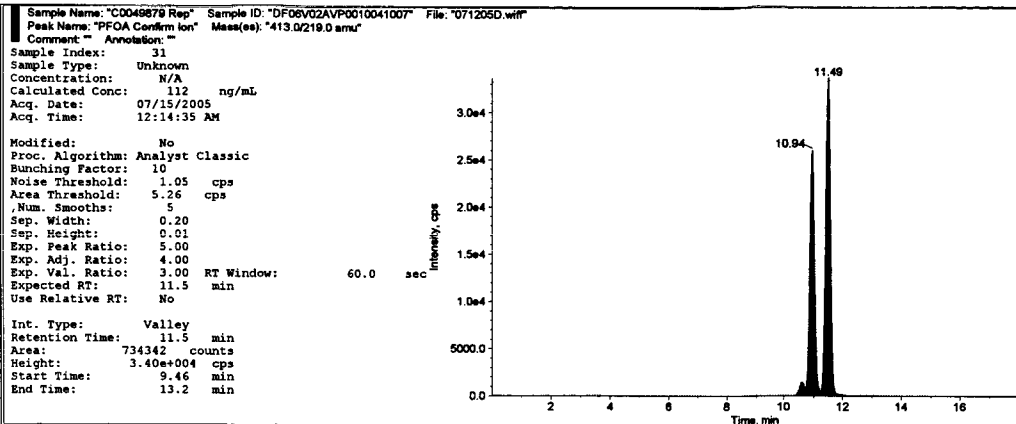


0262

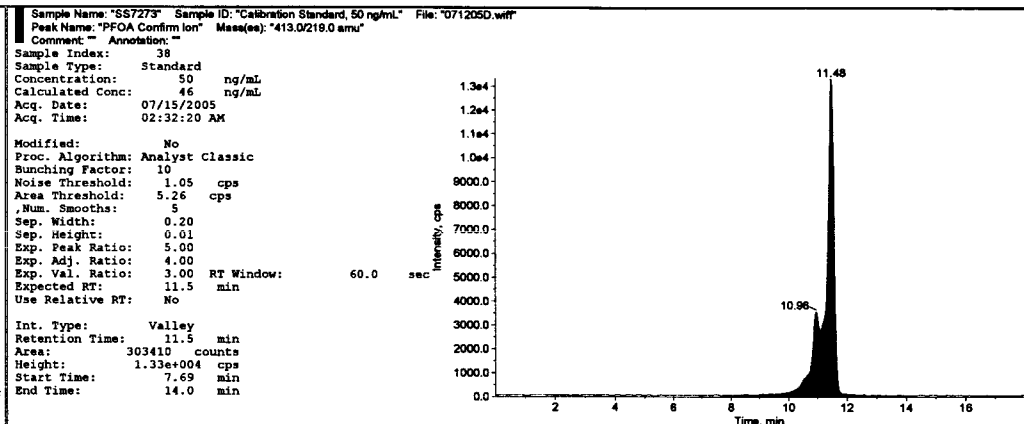
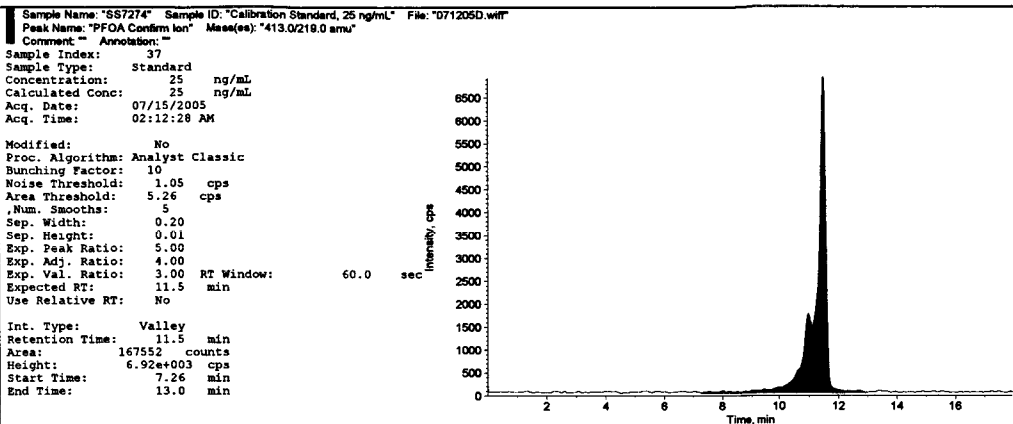




0264



0230 (P) PAP MLC 9/21/05
 965



CEE 07/28/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	191184	0.500	0.396	071205D-101
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	322297	1.00	1.11	071205D-102
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	661753	2.50	2.95	071205D-103
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1253715	5.00	6.17	071205D-104
SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2363097	10.0	12.2	071205D-105
SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	5058273	25.0	28.9	071205D-106
SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9068072	50.0	48.6	071205D-107
Methanol Wash	Methanol Wash	Unknown	1	13C PFOA	87164	N/A	< 0	071205D-108
Reagent Control	C0073483 Control	Unknown	1	13C PFOA	82546	N/A	< 0	071205D-109
Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	13C PFOA	1417383	6.25	7.06	071205D-110
Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	13C PFOA	5247686	25.0	27.9	071205D-111
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	210338	0.500	0.500	071205D-112
C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2290668	25.0	11.8	071205D-113
C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3073680	250.	161.	071205D-114
C0049876	DF06V01AVP0010041007	Unknown	1	13C PFOA	2022570	N/A	10.4	071205D-115
C0049876 Rep	DF06V01AVP0010041007	Unknown	1	13C PFOA	2027839	N/A	10.4	071205D-116
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	241040	1.00	0.667	071205D-117
C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	928767	25.0	4.41	071205D-118
C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2151699	250.	111.	071205D-119
C0049877	DF06V01PAP0010041007	Unknown	1	13C PFOA	780894	N/A	3.60	071205D-120
C0049877 Rep	DF06V01PAP0010041007	Unknown	1	13C PFOA	793091	N/A	3.67	071205D-121
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	495812	2.50	2.05	071205D-122
C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2146505	25.0	11.0	071205D-123
C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3355179	250.	176.	071205D-124
C0049878	DF06V01RCP0010041007	Unknown	1	13C PFOA	1985702	N/A	10.1	071205D-125
C0049878 Rep	DF06V01RCP0010041007	Unknown	1	13C PFOA	1876787	N/A	9.56	071205D-126
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1021436	5.00	4.91	071205D-127
C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2511028	25.0	13.0	071205D-128
C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2802805	250.	146.	071205D-129
C0049879	DF06V02AVP0010041007	Unknown	1	13C PFOA	2162038	N/A	11.1	071205D-130
C0049879 Rep	DF06V02AVP0010041007	Unknown	1	13C PFOA	2208352	N/A	11.4	071205D-131
SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2034291	10.0	10.4	071205D-132
C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1680155	25.0	8.49	071205D-133
C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3125994	250.	163.	071205D-134
C0049880	DF06V02PVP0010041007	Unknown	1	13C PFOA	1358262	N/A	6.74	071205D-135
C0049880 Rep	DF06V02PVP0010041007	Unknown	1	13C PFOA	1398654	N/A	6.96	071205D-136
SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	4561876	25.0	24.2	071205D-137
SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	8761878	50.0	47.0	071205D-138

①
②
③

① (RE) PAP MLC 9/21/05

Vegetation Conversion

Compound: 13C PFOA

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049876	DF06-V01-AVP001-0-041007	10.4	4.16
C0049876 Rep	DF06-V01-AVP001-0-041007	10.4	4.16
C0049876 Spk C	DF06-V01-AVP001-0-041007	11.8	4.72
C0049876 Spk D	DF06-V01-AVP001-0-041007	161	64.4
C0049877	DF06-V01-PAP001-0-041007	3.60	1.44
C0049877 Rep	DF06-V01-PAP001-0-041007	3.67	1.47
C0049877 Spk E	DF06-V01-PAP001-0-041007	4.41	1.76
C0049877 Spk F	DF06-V01-PAP001-0-041007	111	44.4
C0049878	DF06-V01-RCP001-0-041007	10.1	4.04
C0049878 Rep	DF06-V01-RCP001-0-041007	9.56	3.82
C0049878 Spk G	DF06-V01-RCP001-0-041007	11.0	4.40
C0049878 Spk H	DF06-V01-RCP001-0-041007	176	70.4
C0049879	DF06-V02-AVP001-0-041007	11.1	4.44
C0049879 Rep	DF06-V02-AVP001-0-041007	11.4	4.56
C0049879 Spk I	DF06-V02-AVP001-0-041007	13.0	5.20
C0049879 Spk J	DF06-V02-AVP001-0-041007	146	58.4
C0049880	DF06-V02-PVP001-0-041007	6.74	2.70
C0049880 Rep	DF06-V02-PVP001-0-041007	6.96	2.78
C0049880 Spk K	DF06-V02-PVP001-0-041007	8.49	3.40
C0049880 Spk L	DF06-V02-PVP001-0-041007	163	65.2

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = Peak Area 0 AT OR ABOVE 0.2 ng/g ①

NQ = Not quantifiable = Measured concentration below Limit of Quantitation (LOQ) which is 0.6 ng/g.

^
BETWEEN 0.2 ng/g AND ①

4①

① RE MLC 9/20/05

② RP PAP 9/21/05 MLC

0268
BMC 02/29/05

	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound:	13C PFOA	Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049876	DF06-V01-AVP001-0-041007	=Raw Data!H102	=(C9*2)/5
10	C0049876 Rep	DF06-V01-AVP001-0-041007	=Raw Data!H103	=(C10*2)/5
11	C0049876 Spk C	DF06-V01-AVP001-0-041007	=Raw Data!H100	=(C11*2)/5
12	C0049876 Spk D	DF06-V01-AVP001-0-041007	=Raw Data!H101	=(C12*2)/5
13				
14	C0049877	DF06-V01-PAP001-0-041007	=Raw Data!H107	=(C14*2)/5
15	C0049877 Rep	DF06-V01-PAP001-0-041007	=Raw Data!H108	=(C15*2)/5
16	C0049877 Spk E	DF06-V01-PAP001-0-041007	=Raw Data!H105	=(C16*2)/5
17	C0049877 Spk F	DF06-V01-PAP001-0-041007	=Raw Data!H106	=(C17*2)/5
18				
19	C0049878	DF06-V01-RCP001-0-041007	=Raw Data!H112	=(C19*2)/5
20	C0049878 Rep	DF06-V01-RCP001-0-041007	=Raw Data!H113	=(C20*2)/5
21	C0049878 Spk G	DF06-V01-RCP001-0-041007	=Raw Data!H110	=(C21*2)/5
22	C0049878 Spk H	DF06-V01-RCP001-0-041007	=Raw Data!H111	=(C22*2)/5
23				
24	C0049879	DF06-V02-AVP001-0-041007	=Raw Data!H117	=(C24*2)/5
25	C0049879 Rep	DF06-V02-AVP001-0-041007	=Raw Data!H118	=(C25*2)/5
26	C0049879 Spk I	DF06-V02-AVP001-0-041007	=Raw Data!H115	=(C26*2)/5
27	C0049879 Spk J	DF06-V02-AVP001-0-041007	=Raw Data!H116	=(C27*2)/5
28				
29	C0049880	DF06-V02- PVP P001-0-041007	=Raw Data!H122	=(C29*2)/5
30	C0049880 Rep	DF06-V02- PVP P001-0-041007	=Raw Data!H123	=(C30*2)/5
31	C0049880 Spk K	DF06-V02- PVP P001-0-041007	=Raw Data!H120	=(C31*2)/5
32	C0049880 Spk L	DF06-V02- PVP P001-0-041007	=Raw Data!H121	=(C32*2)/5
33				

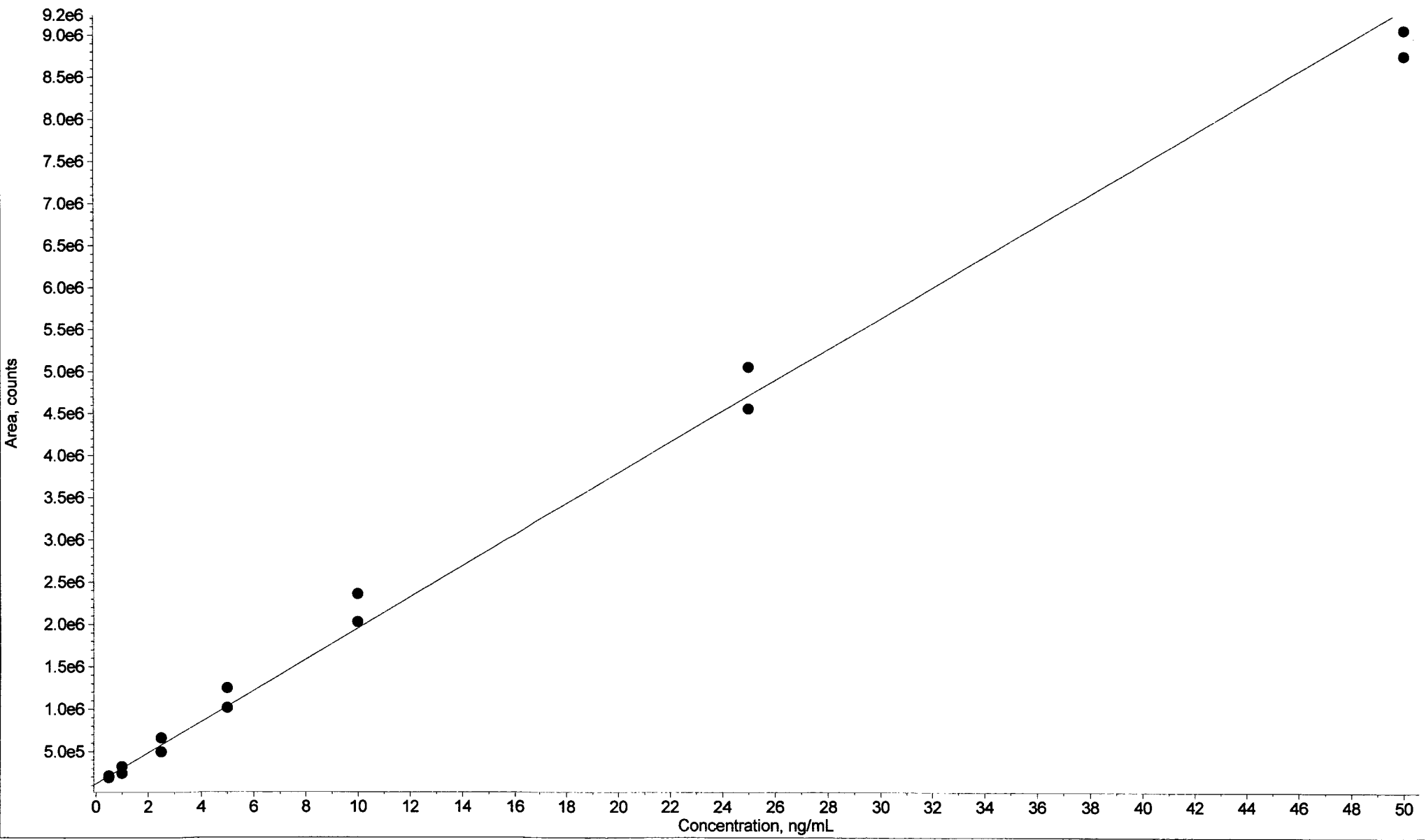
① (RE) PAP MLC 9/21/05

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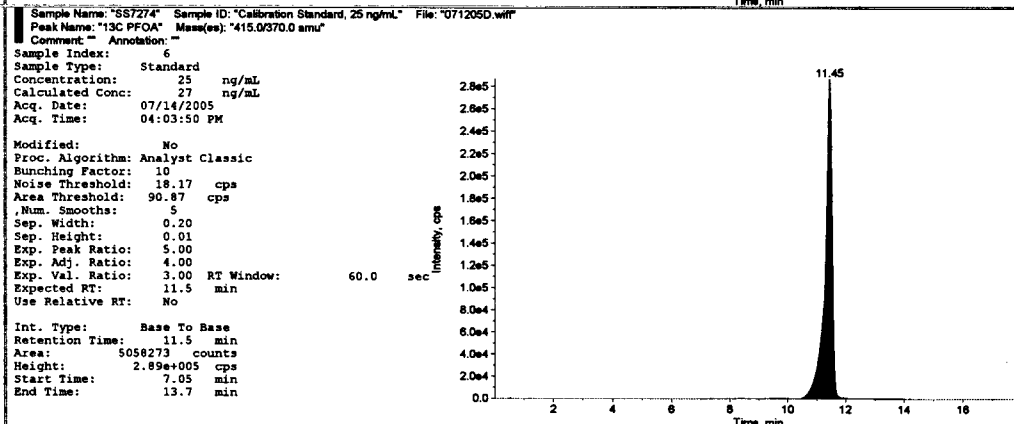
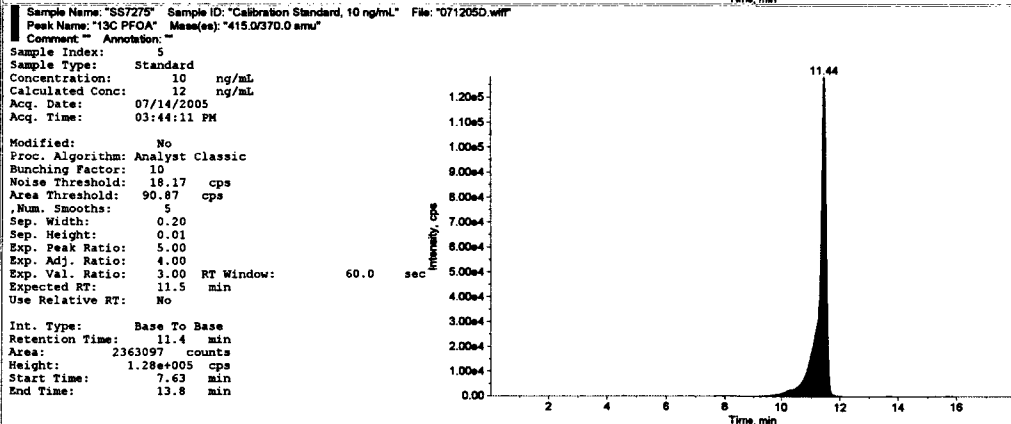
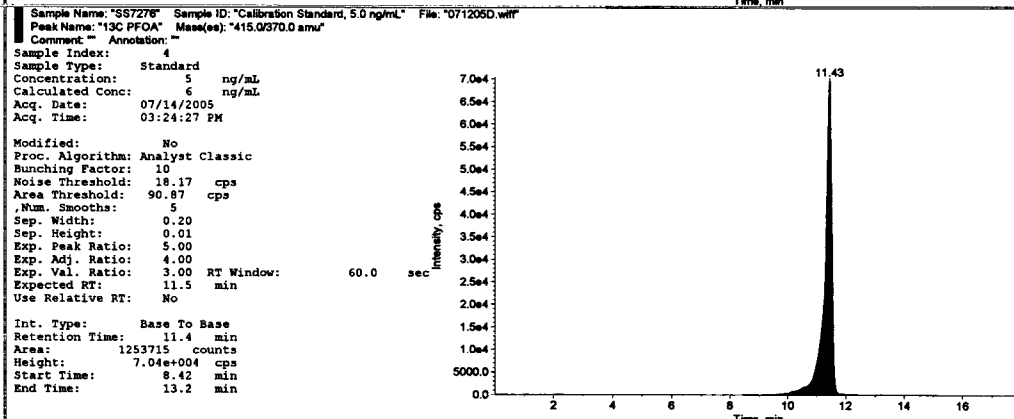
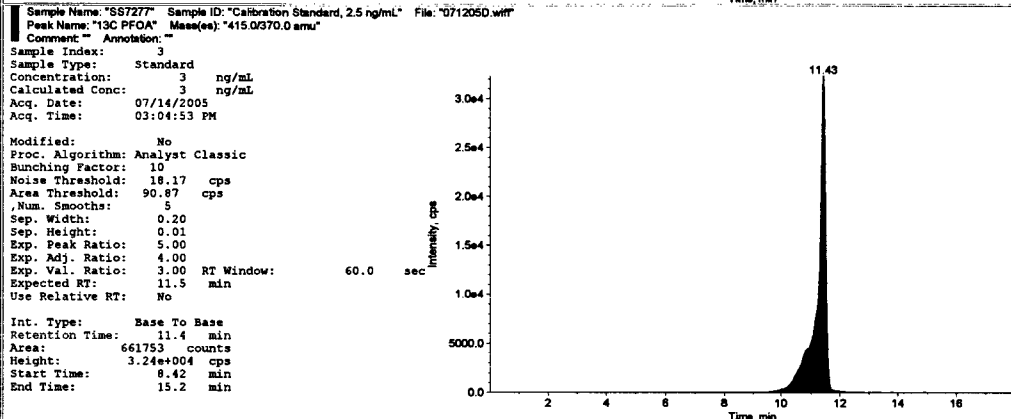
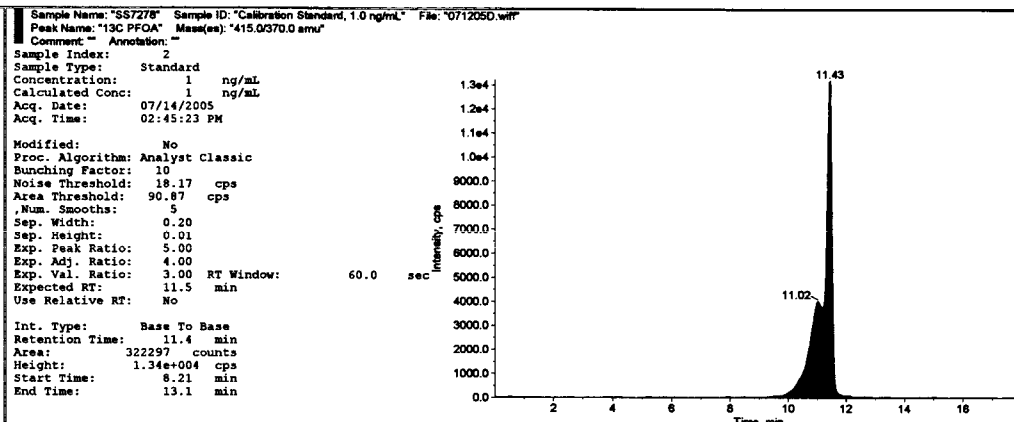
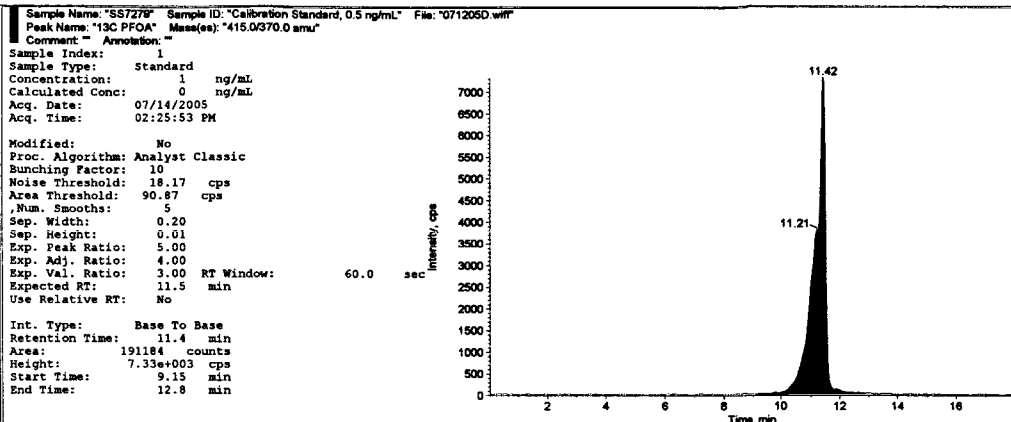
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AMB 07/19/05

■ Untitled 2 (13C PFOA): "Linear" Regression ("1/x" weighting): $y = 1.84e+005 x + 1.18e+005$ ($r = 0.9955$)

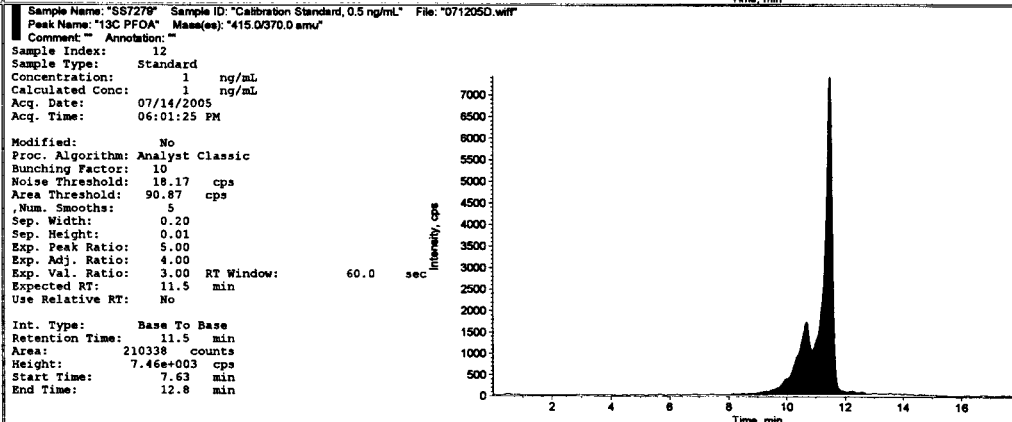
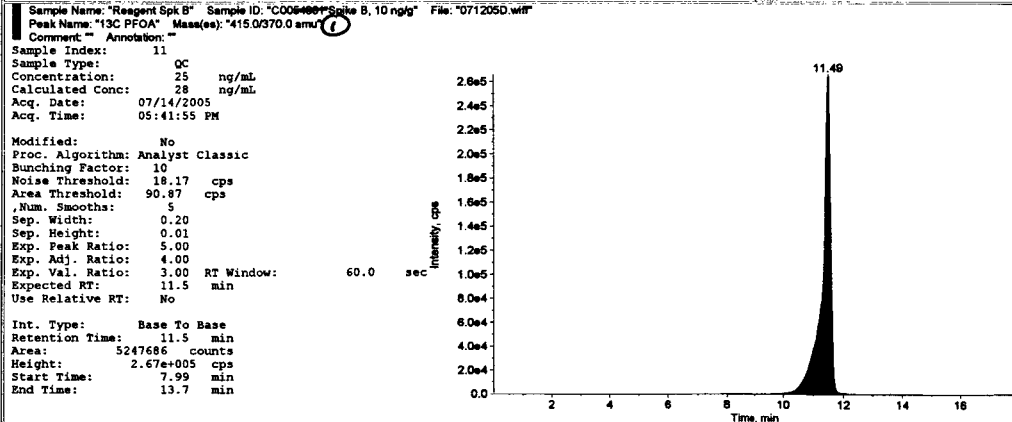
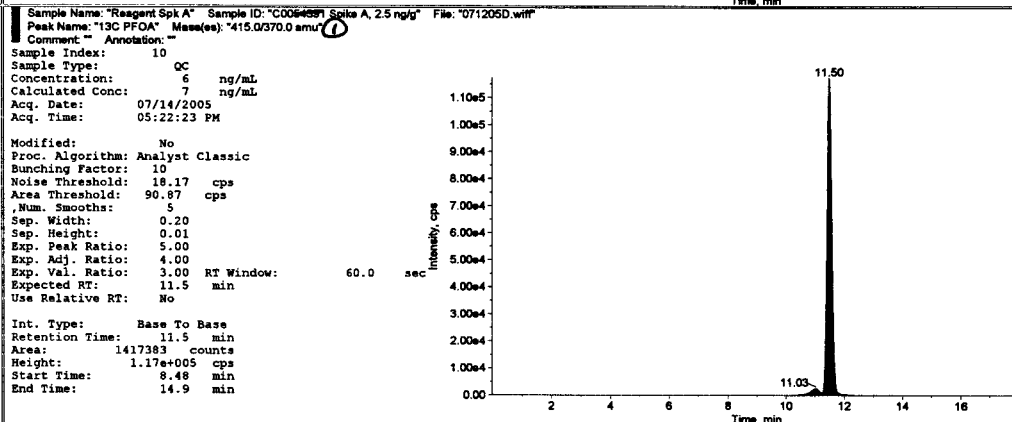
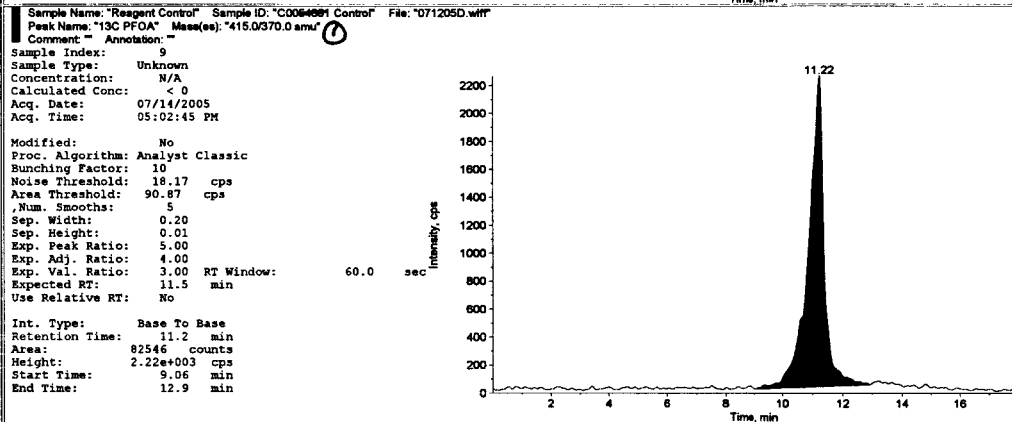
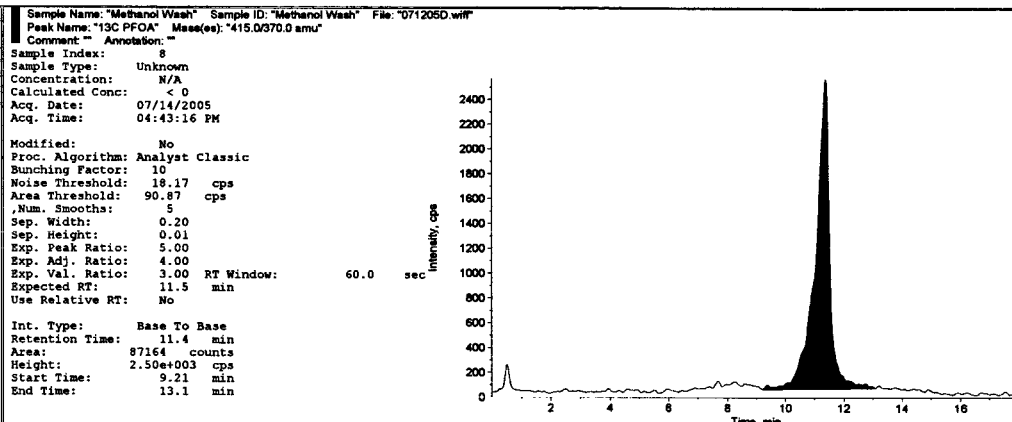
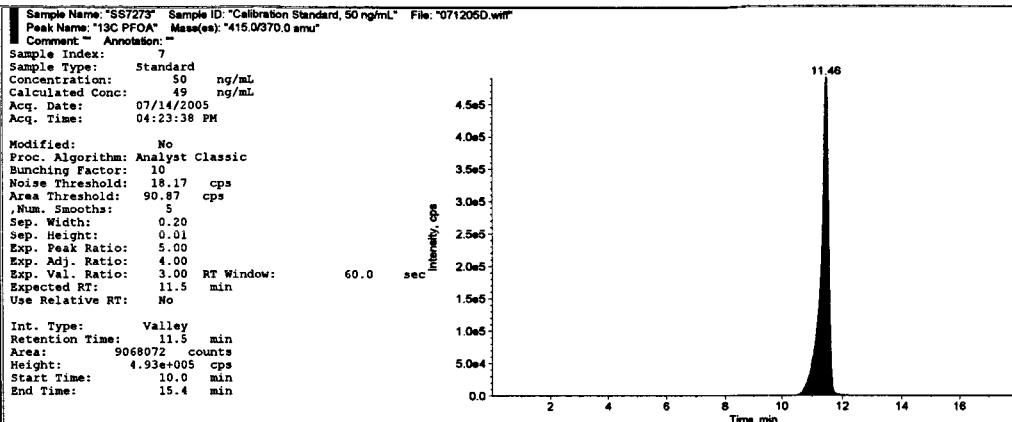


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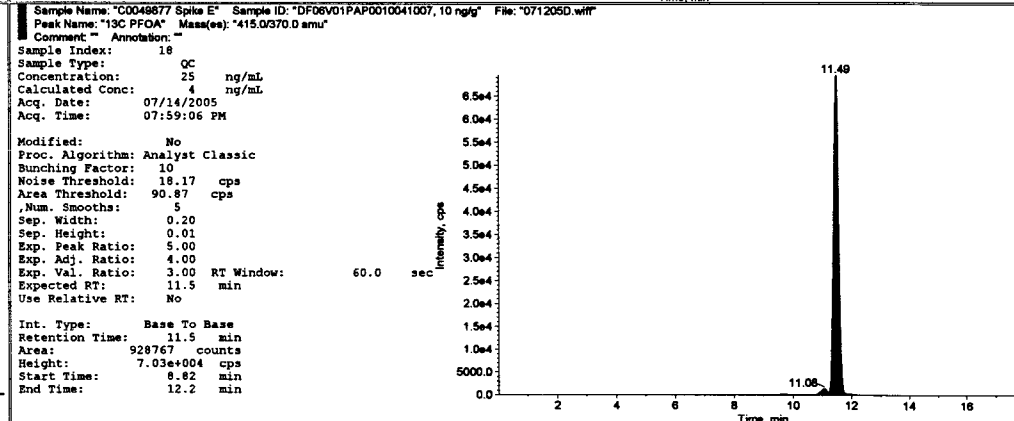
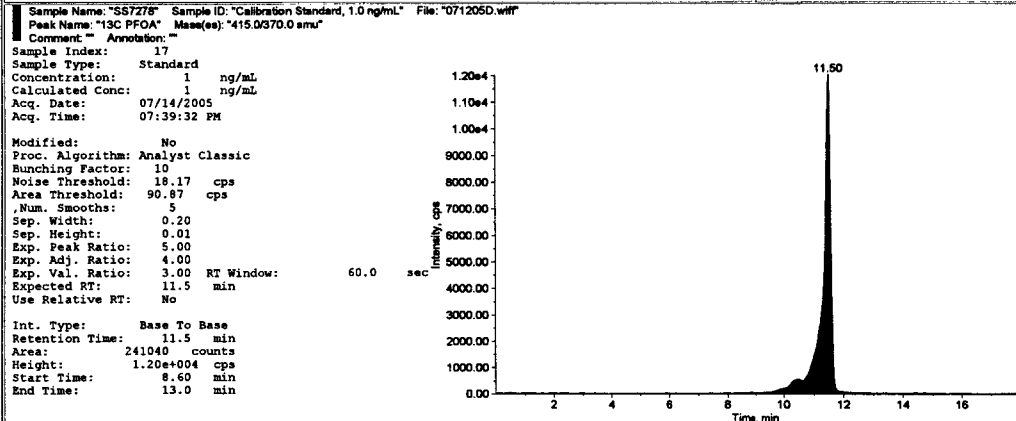
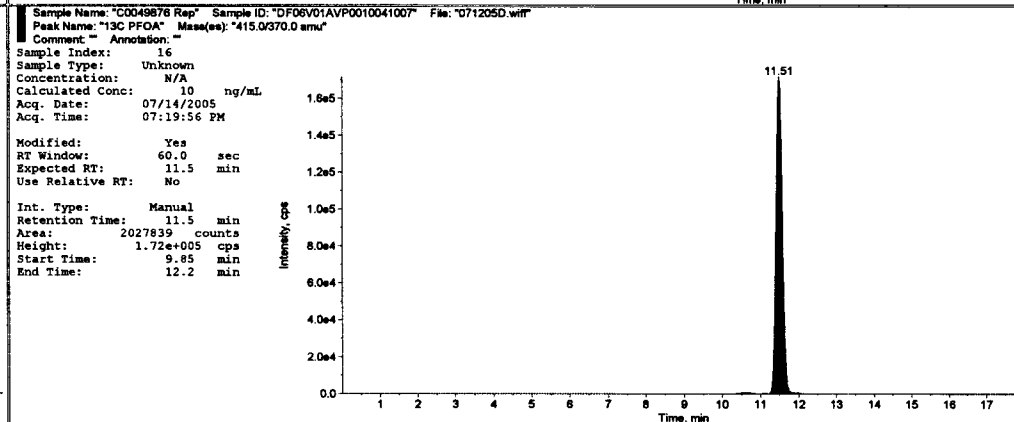
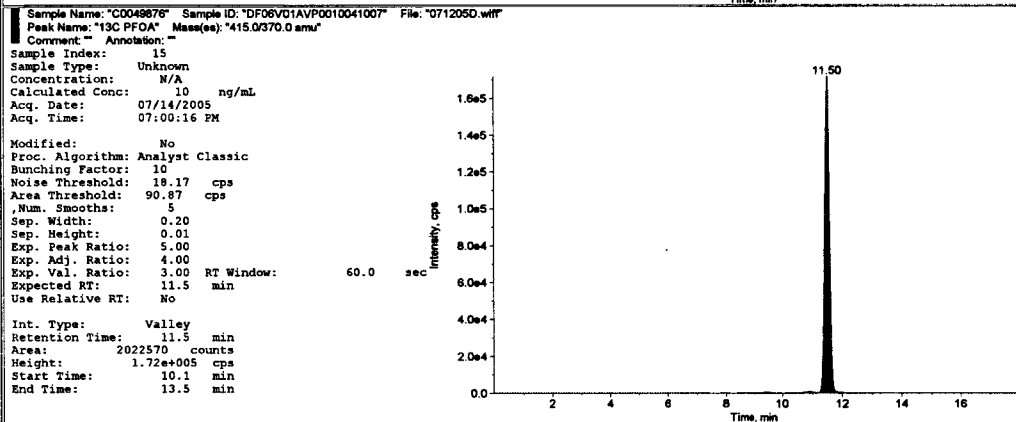
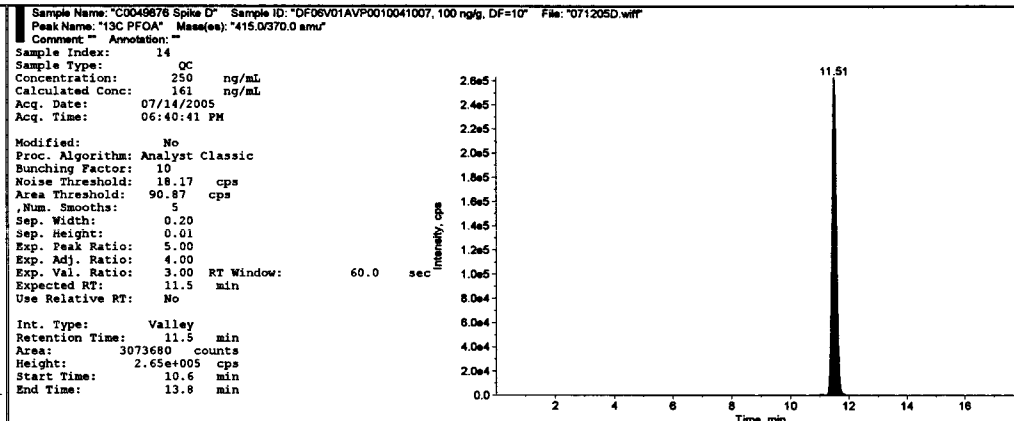
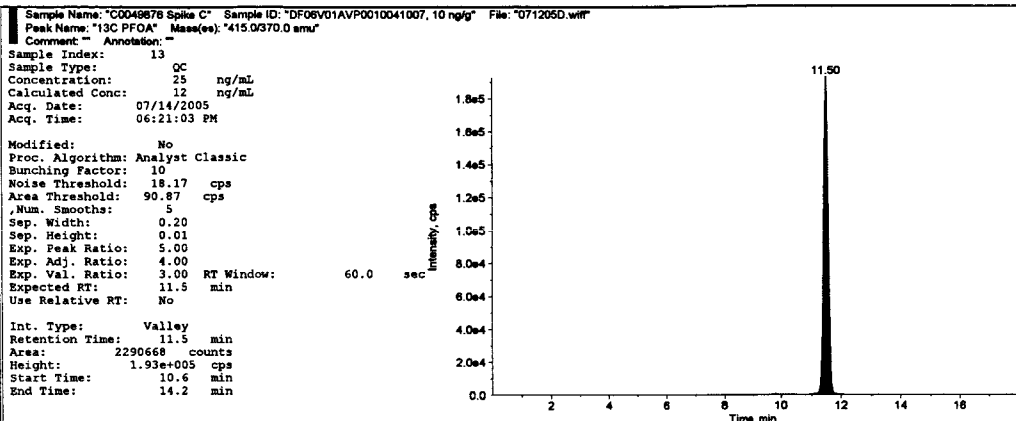


0271 Initials AMB
 Date 07/19/05
 Run# 1 To 38
 Sample Index @AMB 07/19/05

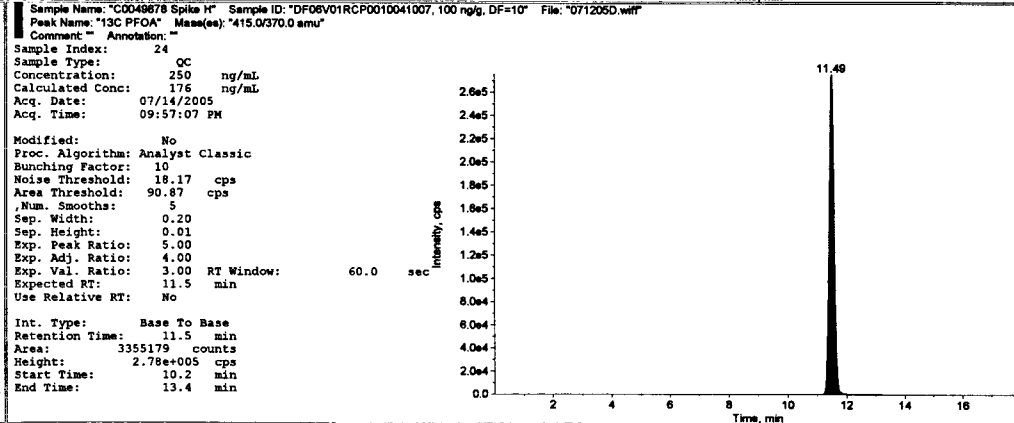
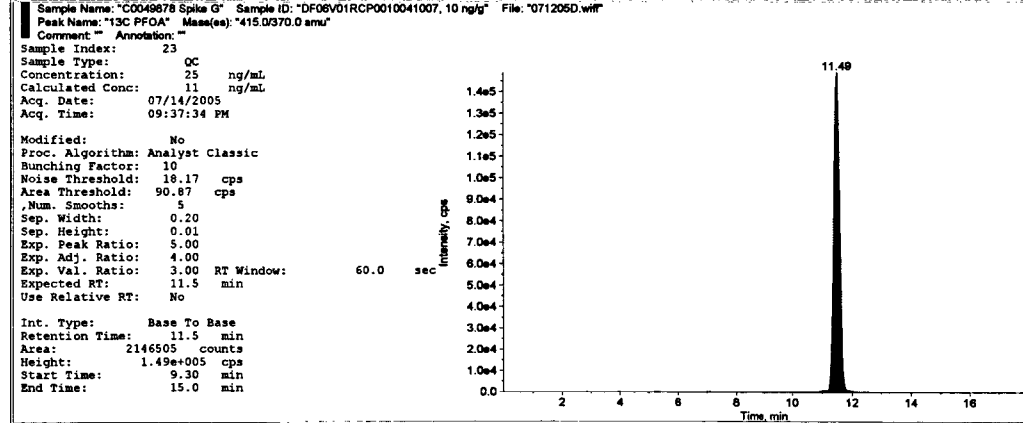
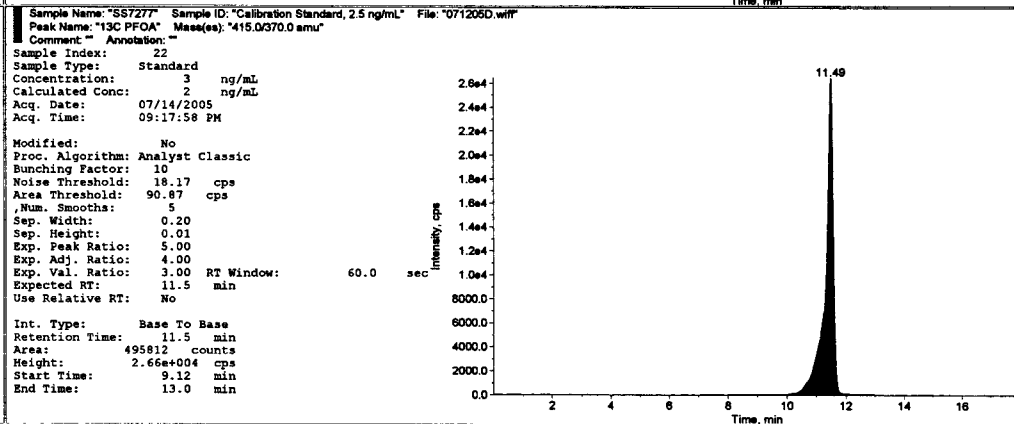
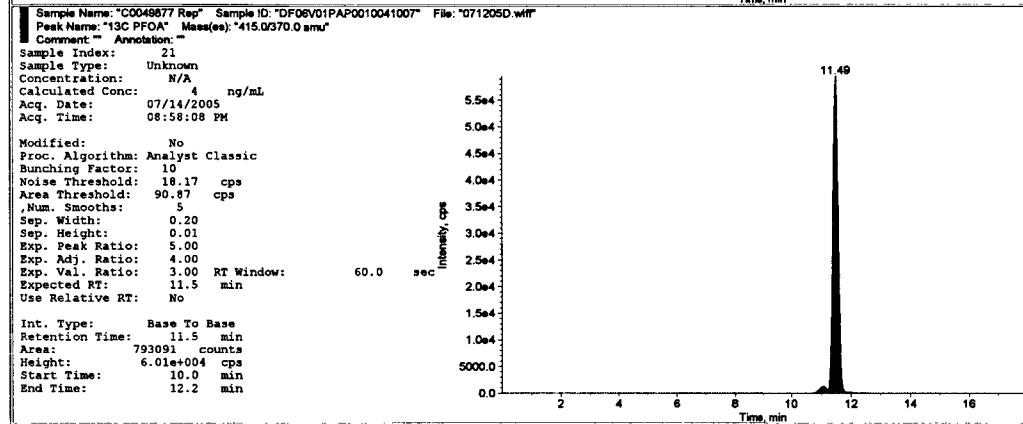
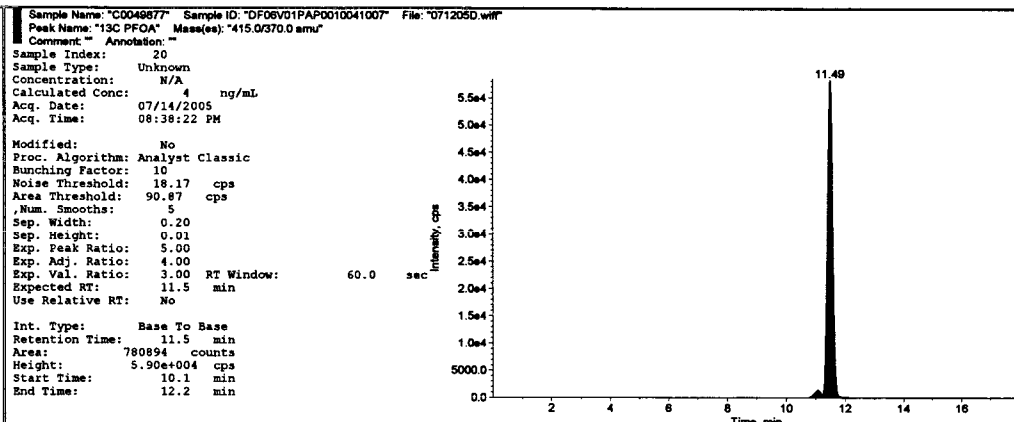
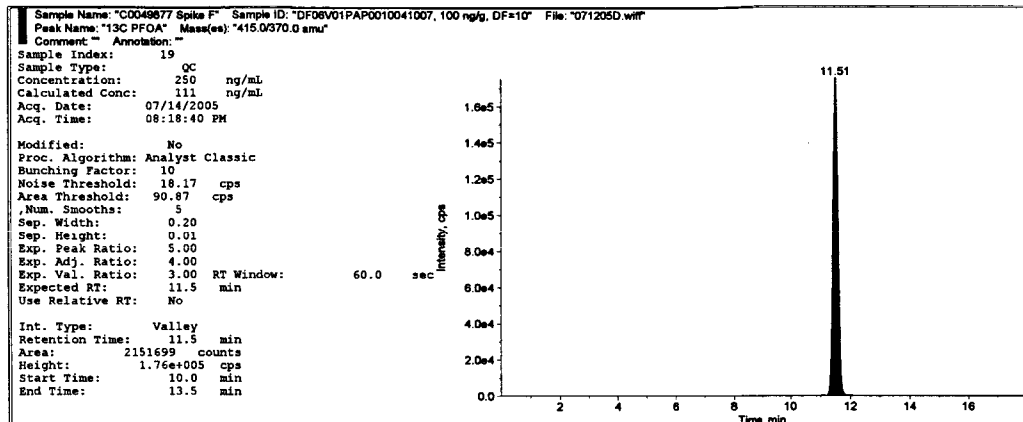
Verified by:
 Initials: AK Date 09/22/05
 Sample Index: 1 To: 38



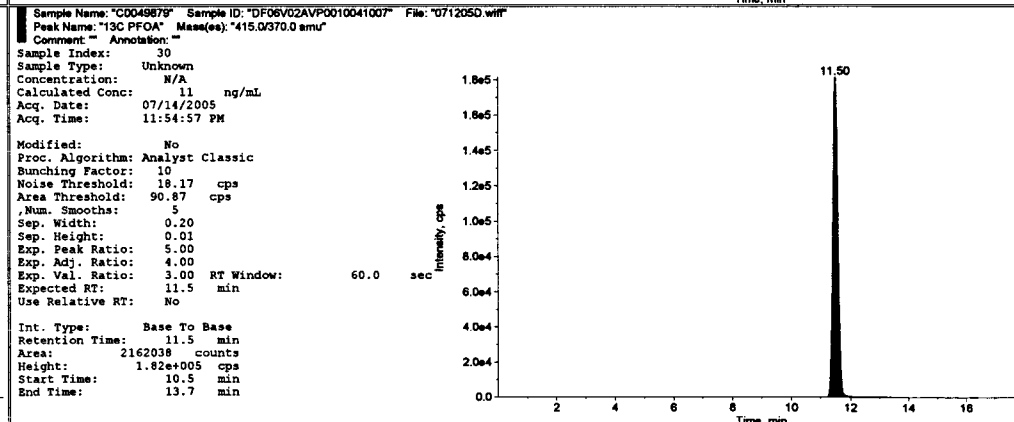
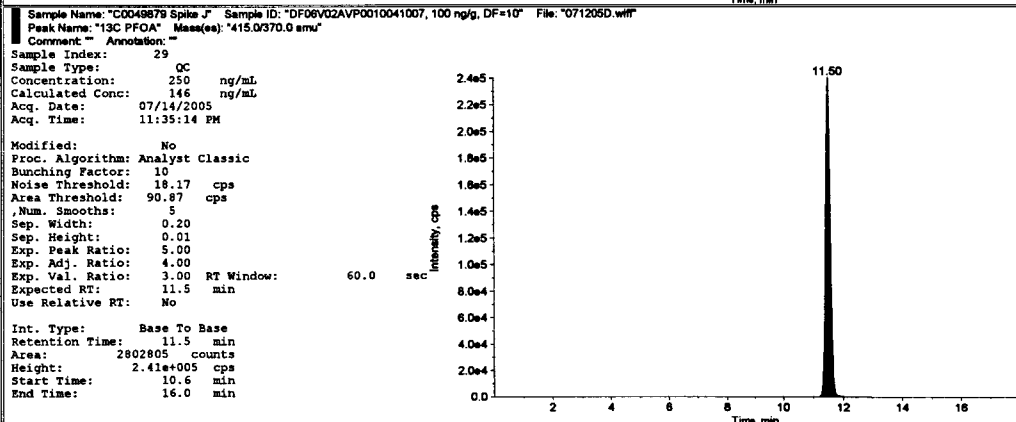
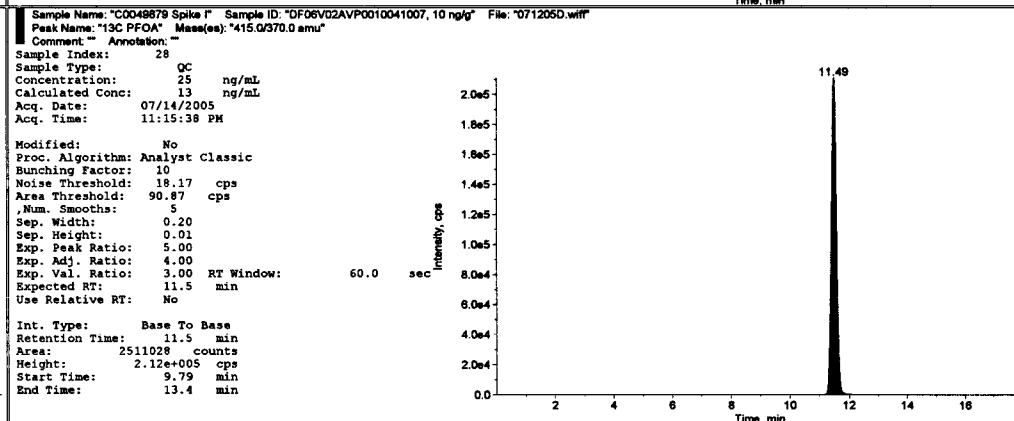
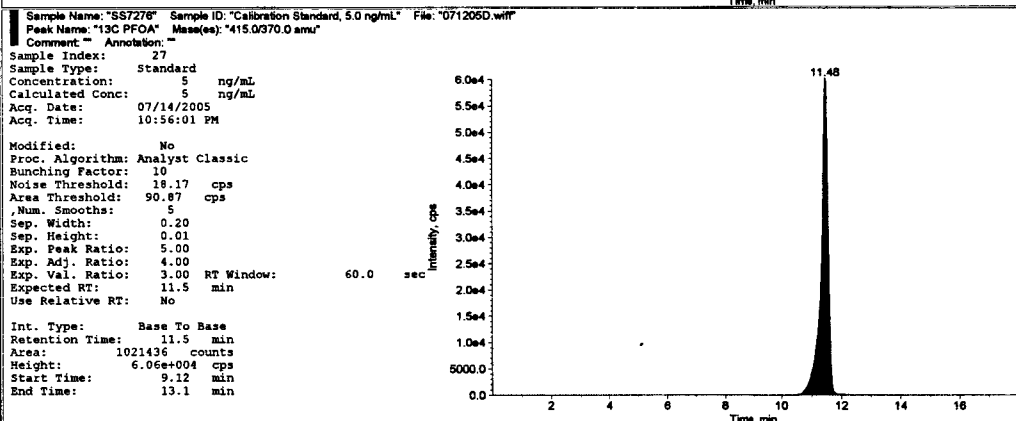
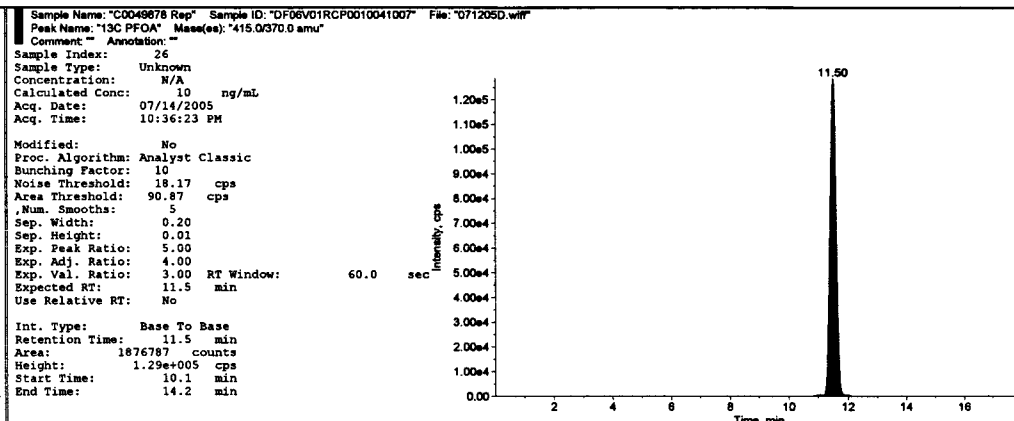
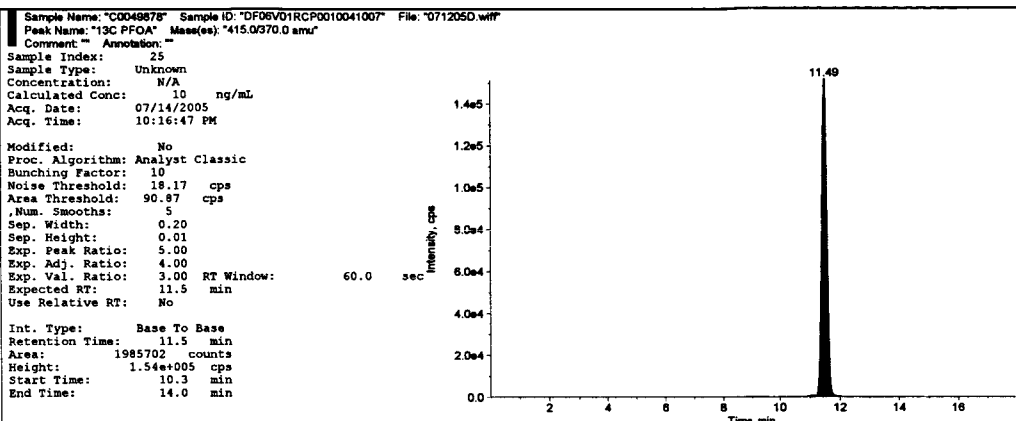
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2720 (R) 73483 MLC 8/21/05



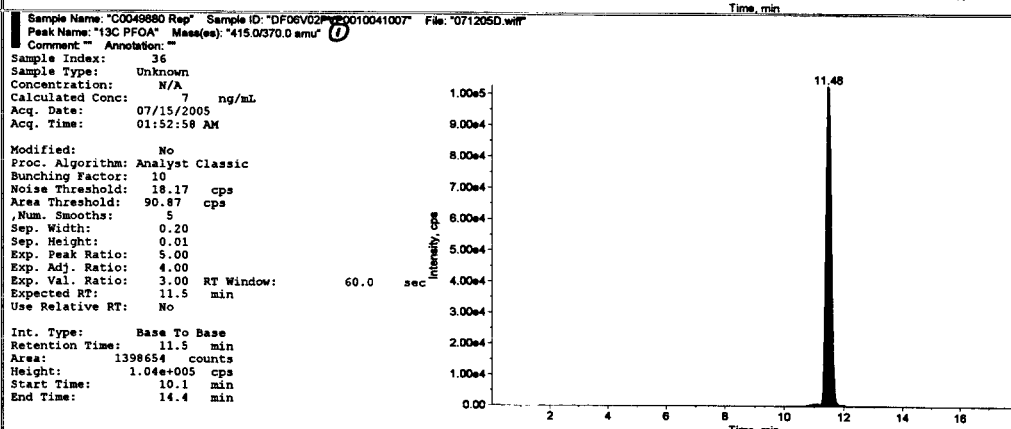
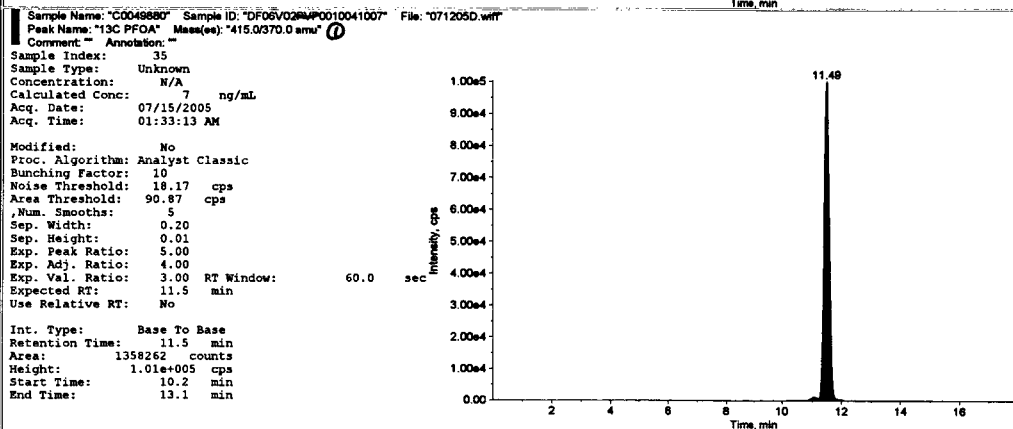
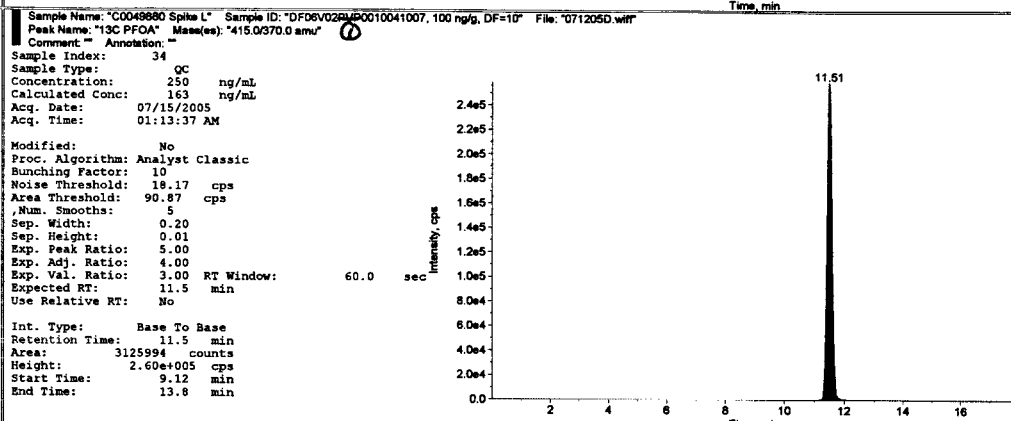
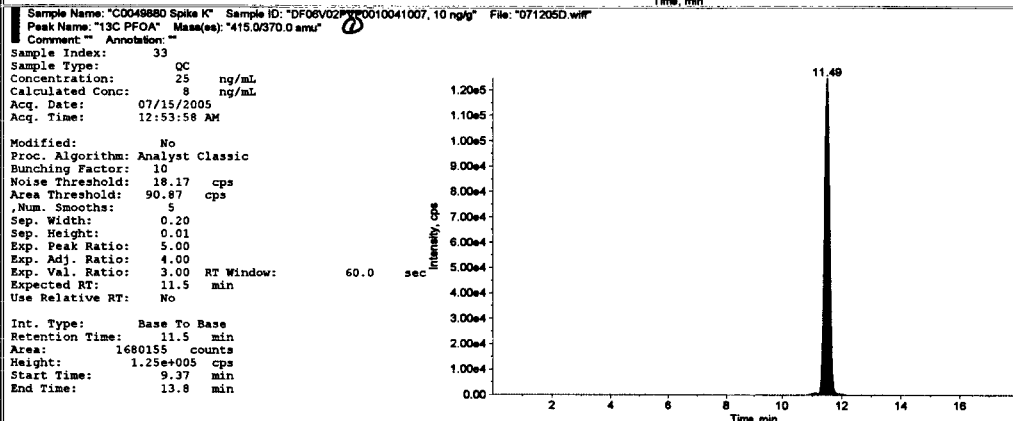
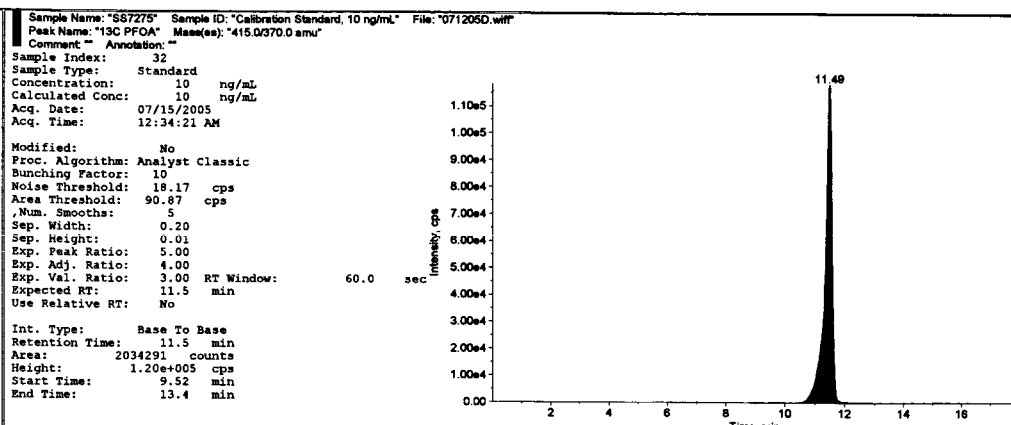
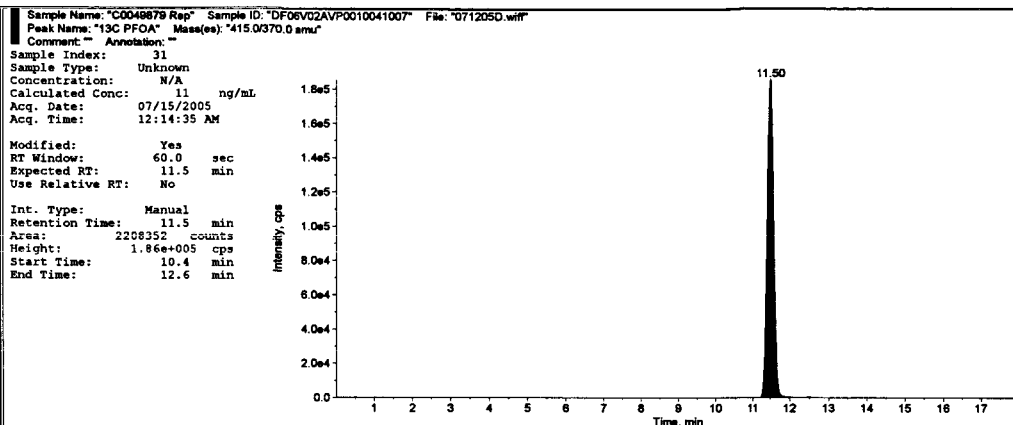
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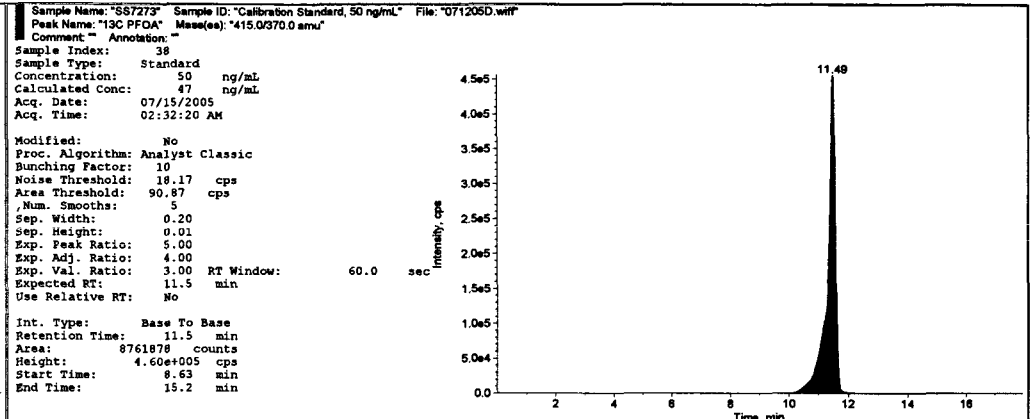
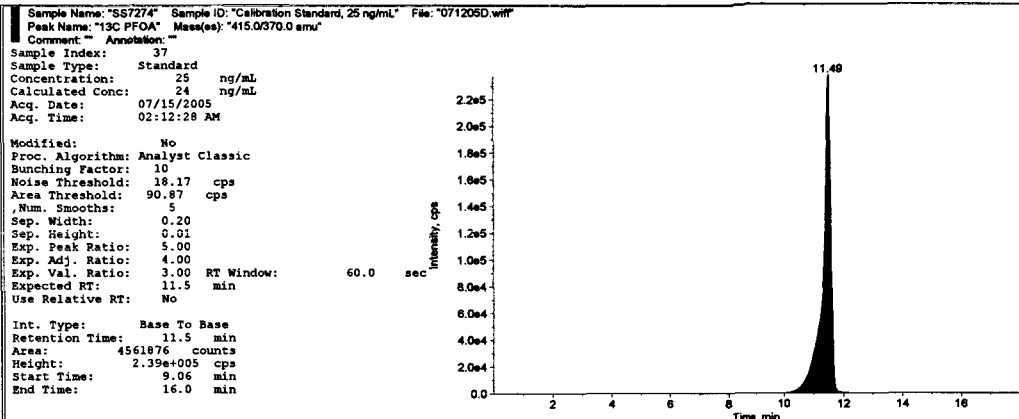


0275



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Raw Data

A	B	C	D	E	F	G	H	I	J
PFBS									
1									
2	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng/mL)	Inculated Concentration (ng/mL)	Run #
3	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFBS	17346	0.5	0.469	71205D-101
4	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFBS	31955	1	0.992	71205D-102
5	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFBS	79734	2.5	2.7	71205D-103
6	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFBS	159101	5	5.54	71205D-104
7	SS7275	Calibration Standard, 10 ng/mL	Standard	1.0	PFBS	303974	10	10.7	71205D-105
8	SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PFBS	722877	25	25.7	71205D-106
9	SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PFBS	1469875	50	52.4	71205D-107
10	Methanol Wash	Methanol Wash	Unknown	1.0	PFBS	0	N/A	No Peak	71205D-108
11	Reagent Control	C0073483 Control	Unknown	1.0	PFBS	0	N/A	No Peak	71205D-109
12	Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1.0	PFBS	179426	6.25	6.27	71205D-110
13	Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1.0	PFBS	750126	25	26.7	71205D-111
14	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFBS	16857	0.5	0.451	71205D-112
15	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	Quality Control	1.0	PFBS	1365532	25	48.7	71205D-113
16	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	574176	250	204	71205D-114
17	C0049876	DF06V01AVP0010041007	Unknown	1.0	PFBS	823471	N/A	29.3	71205D-115
18	C0049876 Rep	DF06V01AVP0010041007	Unknown	1.0	PFBS	779738	N/A	27.7	71205D-116
19	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFBS	32143	1	0.998	71205D-117
20	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	Quality Control	1.0	PFBS	2243214	25	80.1	71205D-118
21	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	763858	250	272	71205D-119
22	C0049877	DF06V01PAP0010041007	Unknown	1.0	PFBS	1815365	N/A	64.8	71205D-120
23	C0049877 Rep	DF06V01PAP0010041007	Unknown	1.0	PFBS	1716375	N/A	61.2	71205D-121
24	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFBS	69970	2.5	2.35	71205D-122
25	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	Quality Control	1.0	PFBS	873154	25	31.1	71205D-123
26	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	610017	250	217	71205D-124
27	C0049878	DF06V01RCP0010041007	Unknown	1.0	PFBS	317339	N/A	11.2	71205D-125
28	C0049878 Rep	DF06V01RCP0010041007	Unknown	1.0	PFBS	309212	N/A	10.9	71205D-126
29	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFBS	145486	5	5.05	71205D-127
30	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	Quality Control	1.0	PFBS	1212030	25	43.2	71205D-128
31	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	537737	250	191	71205D-129
32	C0049879	DF06V02AVP0010041007	Unknown	1.0	PFBS	821426	N/A	29.2	71205D-130
33	C0049879 Rep	DF06V02AVP0010041007	Unknown	1	PFBS	7.39E+05	N/A	26.3	71205D-131
34	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFBS	2.86E+05	10.0	10.1	71205D-132
35	C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g	Quality Control	1	PFBS	1.20E+06	25.0	42.8	71205D-133
36	C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFBS	6.32E+05	250.	224	71205D-134
37	C0049880	DF06V02PVP0010041007	Unknown	1	PFBS	6.12E+05	N/A	21.7	71205D-135
38	C0049880 Rep	DF06V02PVP0010041007	Unknown	1	PFBS	6.13E+05	N/A	21.8	71205D-136
39	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFBS	6.53E+05	25.0	23.2	71205D-137
40	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFBS	1.33E+06	50.0	47.3	71205D-138
41									
42									
PFHS									
43									
44	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng/mL)	Inculated Concentration (ng/mL)	Run #
45	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFHS	28955	0.500	0.484	71205D-101
46	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFHS	53414	1.00	0.98	71205D-102
47	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFHS	131161	2.50	2.56	71205D-103
48	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFHS	271488	5.00	5.4	71205D-104
49	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFHS	540373	10.0	10.9	71205D-105
50	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFHS	1290715	25.0	26.1	71205D-106
51	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFHS	2469958	50.0	50	71205D-107
52	Methanol Wash	Methanol Wash	Unknown	1	PFHS	0	N/A	No Peak	71205D-108
53	Reagent Control	C0073483 Control	Unknown	1	PFHS	0	N/A	No Peak	71205D-109
54	Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFHS	375254	6.25	7.51	71205D-110
55	Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFHS	1207643	25.0	24.4	71205D-111
56	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFHS	27562	0.500	0.456	71205D-112
57	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	Quality Control	1	PFHS	5734245	25.0	116	71205D-113
58	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1464497	250.	296	71205D-114
59	C0049876	DF06V01AVP0010041007	Unknown	1	PFHS	4482184	N/A	90.8	71205D-115
60	C0049876 Rep	DF06V01AVP0010041007	Unknown	1	PFHS	4231503	N/A	85.7	71205D-116
61	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFHS	53779	1.00	0.988	71205D-117
62	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	Quality Control	1	PFHS	22185873	25.0	450	71205D-118
63	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	5615335	250.	1140	71205D-119
64	C0049877	DF06V01PAP0010041007	Unknown	1	PFHS	24481935	N/A	497	71205D-120
65	C0049877 Rep	DF06V01PAP0010041007	Unknown	1	PFHS	24512789	N/A	497	71205D-121
66	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFHS	128384	2.50	2.5	71205D-122
67	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	Quality Control	1	PFHS	3716249	25.0	75.3	71205D-123
68	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1490780	250.	301	71205D-124
69	C0049878	DF06V01RCP0010041007	Unknown	1	PFHS	2583140	N/A	52.3	71205D-125
70	C0049878 Rep	DF06V01RCP0010041007	Unknown	1	PFHS	2692401	N/A	54.5	71205D-126
71	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFHS	250209	5.00	4.97	71205D-127
72	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	Quality Control	1	PFHS	3090660	25.0	62.6	71205D-128
73	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1096745	250.	221	71205D-129
74	C0049879	DF06V02AVP0010041007	Unknown	1	PFHS	2288294	N/A	46.3	71205D-130
75	C0049879 Rep	DF06V02AVP0010041007	Unknown	1	PFHS	2017138	N/A	40.8	71205D-131
76	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFHS	508313	10.0	10.2	71205D-132
77	C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g	Quality Control	1	PFHS	6664619	25.0	135	71205D-133
78	C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1794140	250.	363	71205D-134
79	C0049880	DF06V02PVP0010041007	Unknown	1	PFHS	5309787	N/A	108	71205D-135
80	C0049880 Rep	DF06V02PVP0010041007	Unknown	1	PFHS	5441732	N/A	110	71205D-136
81	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFHS	1117696	25.0	22.6	71205D-137
82	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFHS	2465467	50.0	49.9	71205D-138

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Raw Data

	A	B	C	D	E	F	G	H	I	J
84										
85										
86			13C PFOA							
87	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng/L)	Inculated Concentration (ng/L)	Run #	
88	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	191184	0.5	0.396	71205D-101	
89	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	322297	1	1.11	71205D-102	
90	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	661753	2.5	2.95	71205D-103	
91	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1253715	5	6.17	71205D-104	
92	SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2363097	10	12.2	71205D-105	
93	SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	5058273	25	26.9	71205D-106	
94	SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9068072	50	48.6	71205D-107	
95	Methanol Wash	Methanol Wash	Unknown	1	13C PFOA	87164	N/A	< 0	71205D-108	
96	Reagent Control	C0073483 Control	Unknown	1	13C PFOA	82546	N/A	< 0	71205D-109	
97	Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	13C PFOA	1417383	6.25	7.06	71205D-110	
98	Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	13C PFOA	5247686	25	27.9	71205D-111	
99	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	210338	0.5	0.5	71205D-112	
100	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2290668	25	11.8	71205D-113	
101	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3073680	250	161	71205D-114	
102	C0049876	DF06V01AVP0010041007	Unknown	1	13C PFOA	2022570	N/A	10.4	71205D-115	
103	C0049876 Rep	DF06V01AVP0010041007	Unknown	1	13C PFOA	2027839	N/A	10.4	71205D-116	
104	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	241040	1	0.667	71205D-117	
105	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	928767	25	4.41	71205D-118	
106	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2151699	250	111	71205D-119	
107	C0049877	DF06V01PAP0010041007	Unknown	1	13C PFOA	780894	N/A	3.6	71205D-120	
108	C0049877 Rep	DF06V01PAP0010041007	Unknown	1	13C PFOA	793091	N/A	3.67	71205D-121	
109	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	495812	2.5	2.05	71205D-122	
110	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2146505	25	11	71205D-123	
111	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3355179	250	176	71205D-124	
112	C0049878	DF06V01RCP0010041007	Unknown	1	13C PFOA	1985702	N/A	10.1	71205D-125	
113	C0049878 Rep	DF06V01RCP0010041007	Unknown	1	13C PFOA	1876787	N/A	9.56	71205D-126	
114	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1021436	5	4.91	71205D-127	
115	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2511028	25	13	71205D-128	
116	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2802805	250	146	71205D-129	
117	C0049879	DF06V02AVP0010041007	Unknown	1	13C PFOA	2162038	N/A	11.1	71205D-130	
118	C0049879 Rep	DF06V02AVP0010041007	Unknown	1	13C PFOA	2208352	N/A	11.4	71205D-131	
119	SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2034291	10	10.4	71205D-132	
120	C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1680155	25	8.49	71205D-133	
121	C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3125994	250	163	71205D-134	
122	C0049880	DF06V02PVP0010041007	Unknown	1	13C PFOA	1358262	N/A	6.74	71205D-135	
123	C0049880 Rep	DF06V02PVP0010041007	Unknown	1	13C PFOA	1398654	N/A	6.96	71205D-136	
124	SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	4561876	25	24.2	71205D-137	
125	SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	8761878	50	47	71205D-138	
126										
127										
128			PFOA							
129	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng/L)	Inculated Concentration (ng/L)	Run #	
130	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	157152	0.5	0.288	71205D-101	
131	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	293199	1	0.906	71205D-102	
132	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	697320	2.5	2.74	71205D-103	
133	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1444221	5	6.13	71205D-104	
134	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2725320	10	11.9	71205D-105	
135	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5908891	25	26.4	71205D-106	
136	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10588859	50	47.6	71205D-107	
137	Methanol Wash	Methanol Wash	Unknown	1	PFOA	33187	N/A	< 0	71205D-108	
138	Reagent Control	C0073483 Control	Unknown	1	PFOA	55009	N/A	< 0	71205D-109	
139	Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFOA	1565685	6.25	6.68	71205D-110	
140	Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFOA	6278495	25	28.1	71205D-111	
141	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	182338	0.5	0.403	71205D-112	
142	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	Quality Control	1	PFOA	28926901	25	131	71205D-113	
143	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	7195952	250	322	71205D-114	
144	C0049876	DF06V01AVP0010041007	Unknown	1	PFOA	27411925	N/A	124	71205D-115	
145	C0049876 Rep	DF06V01AVP0010041007	Unknown	1	PFOA	24894626	N/A	113	71205D-116	
146	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	279854	1	0.845	71205D-117	
147	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	Quality Control	1	PFOA	111947693	25	508	71205D-118	
148	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	38860553	250	1760	71205D-119	
149	C0049877	DF06V01PAP0010041007	Unknown	1	PFOA	111415202	N/A	505	71205D-120	
150	C0049877 Rep	DF06V01PAP0010041007	Unknown	1	PFOA	109850324	N/A	498	71205D-121	
151	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	795631	2.5	3.19	71205D-122	
152	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	Quality Control	1	PFOA	64230832	25	291	71205D-123	
153	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	14499202	250	654	71205D-124	
154	C0049878	DF06V01RCP0010041007	Unknown	1	PFOA	60234325	N/A	273	71205D-125	
155	C0049878 Rep	DF06V01RCP0010041007	Unknown	1	PFOA	61401925	N/A	278	71205D-126	
156	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1234165	5	5.18	71205D-127	
157	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	Quality Control	1	PFOA	19433745	25	87.8	71205D-128	
158	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	5527940	250	247	71205D-129	
159	C0049879	DF06V02AVP0010041007	Unknown	1	PFOA	18689056	N/A	84.4	71205D-130	
160	C0049879 Rep	DF06V02AVP0010041007	Unknown	1	PFOA	16169588	N/A	73	71205D-131	
161	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2542658	10	11.1	71205D-132	
162	C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g	Quality Control	1	PFOA	79506341	25	361	71205D-133	
163	C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	21180098	250	957	71205D-134	
164	C0049880	DF06V02PVP0010041007	Unknown	1	PFOA	76673462	N/A	348	71205D-135	
165	C0049880 Rep	DF06V02PVP0010041007	Unknown	1	PFOA	76925150	N/A	349	71205D-136	
166	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5535612	25	24.7	71205D-137	

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BAL 07/29/05

Raw Data

A	B	C	D	E	F	G	H	I	J
167	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10338038	50	46.5	71205D-138
168									
169									
170	PFOA Confirm Ion								
171	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Peak Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
172	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5124	0.5	0.375	71205D-101
173	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	7525	1	0.742	71205D-102
174	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	19472	2.5	2.57	71205D-103
175	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	39820	5	5.69	71205D-104
176	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	78150	10	11.6	71205D-105
177	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	173073	25	26.1	71205D-106
178	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	324747	50	49.3	71205D-107
179	Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	71205D-108
180	Reagent Control	C0073483 Control	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	71205D-109
181	Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFOA Confirm Ion	45692	6.25	6.59	71205D-110
182	Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFOA Confirm Ion	181044	25	27.3	71205D-111
183	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5864	0.5	0.488	71205D-112
184	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	2186904	25	334	71205D-113
185	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	339187	250	515	71205D-114
186	C0049876	DF06V01AVP0010041007	Unknown	1	PFOA Confirm Ion	2114215	N/A	323	71205D-115
187	C0049876 Rep	DF06V01AVP0010041007	Unknown	1	PFOA Confirm Ion	1973341	N/A	302	71205D-116
188	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	7862	1	0.794	71205D-117
189	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	11085187	25	1700	71205D-118
190	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	2315485	250	3540	71205D-119
191	C0049877	DF06V01PAP0010041007	Unknown	1	PFOA Confirm Ion	11180365	N/A	1710	71205D-120
192	C0049877 Rep	DF06V01PAP0010041007	Unknown	1	PFOA Confirm Ion	10797565	N/A	1650	71205D-121
193	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	25797	2.5	3.54	71205D-122
194	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	6257622	25	958	71205D-123
195	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	914744	250	1400	71205D-124
196	C0049878	DF06V01RCP0010041007	Unknown	1	PFOA Confirm Ion	5962671	N/A	912	71205D-125
197	C0049878 Rep	DF06V01RCP0010041007	Unknown	1	PFOA Confirm Ion	6229410	N/A	953	71205D-126
198	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	34350	5	4.85	71205D-127
199	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	833604	25	127	71205D-128
200	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	188799	250	285	71205D-129
201	C0049879	DF06V02AVP0010041007	Unknown	1	PFOA Confirm Ion	823847	N/A	126	71205D-130
202	C0049879 Rep	DF06V02AVP0010041007	Unknown	1	PFOA Confirm Ion	734342	N/A	112	71205D-131
203	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	72660	10	10.7	71205D-132
204	C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	7523563	25	1150	71205D-133
205	C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	1193542	250	1820	71205D-134
206	C0049880	DF06V02PVP0010041007	Unknown	1	PFOA Confirm Ion	7086802	N/A	1080	71205D-135
207	C0049880 Rep	DF06V02PVP0010041007	Unknown	1	PFOA Confirm Ion	7020974	N/A	1070	71205D-136
208	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	167552	25	25.2	71205D-137
209	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	303410	50	46	71205D-138

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BVK 07/29/08

Vegetation Set #1

Data Set 071205DR

Extracted on 07/12/05

(Analyzed on 07/24/05)

AMB 07/22/05

t:\Abbeyroad\PE Sciox Data\Projects\P1131 Decatur Monitoring Study Batch:071205DR VEG P760 Tab:Sample Set:SET1 AcqMethod:P760-1131 Isomer Separatio

Sample	Sample Name	Sample ID	Vial Position	Data File	Run #
1	SS7279	Calibration Standard, 0.5 ng/mL	1	08_071205DR071205DR	071205DR-101
2	SS7278	Calibration Standard, 1.0 ng/mL	2	08_071205DR071205DR	071205DR-102
3	SS7277	Calibration Standard, 2.5 ng/mL	3	08_071205DR071205DR	071205DR-103
4	SS7276	Calibration Standard, 5.0 ng/mL	4	08_071205DR071205DR	071205DR-104
5	SS7275	Calibration Standard, 10 ng/mL	5	08_071205DR071205DR	071205DR-105
6	SS7274	Calibration Standard, 25 ng/mL	6	08_071205DR071205DR	071205DR-106
7	SS7273	Calibration Standard, 50 ng/mL	7	08_071205DR071205DR	071205DR-107
8	Methanol Wash	Methanol Wash	92	08_071205DR071205DR	071205DR-108
9	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=100	11	08_071205DR071205DR	071205DR-109
10	C0049876 Spike C	DF06V01PAP0010041007, 10 ng/g, DF=10	12	08_071205DR071205DR	071205DR-110
11	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=100	13	08_071205DR071205DR	071205DR-111
12	C0049876	DF06V01AVP0010041007, DF=100	14	08_071205DR071205DR	071205DR-112
13	C0049876 Rep	DF06V01AVP0010041007, DF=100	15	08_071205DR071205DR	071205DR-113
14	C0049876	DF06V01AVP0010041007, DF=10	16	08_071205DR071205DR	071205DR-114
15	C0049876 Rep	DF06V01AVP0010041007, DF=10	17	08_071205DR071205DR	071205DR-115
16	SS7279	Calibration Standard, 0.5 ng/mL	1	08_071205DR071205DR	071205DR-116
17	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=1000	18	08_071205DR071205DR	071205DR-117
18	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=100	19	08_071205DR071205DR	071205DR-118
19	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=10	20	08_071205DR071205DR	071205DR-119
20	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=1000	21	08_071205DR071205DR	071205DR-120
21	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=100	22	08_071205DR071205DR	071205DR-121
22	SS7278	Calibration Standard, 1.0 ng/mL	2	08_071205DR071205DR	071205DR-122
23	C0049877	DF06V01PAP0010041007, DF=1000	23	08_071205DR071205DR	071205DR-123
24	C0049877 Rep	DF06V01PAP0010041007, DF=1000	24	08_071205DR071205DR	071205DR-124
25	C0049877	DF06V01PAP0010041007, DF=100	25	08_071205DR071205DR	071205DR-125
26	C0049877 Rep	DF06V01PAP0010041007, DF=100	26	08_071205DR071205DR	071205DR-126
27	C0049877	DF06V01PAP0010041007, DF=10	27	08_071205DR071205DR	071205DR-127
28	C0049877 Rep	DF06V01PAP0010041007, DF=10	28	08_071205DR071205DR	071205DR-128
29	SS7277	Calibration Standard, 2.5 ng/mL	3	08_071205DR071205DR	071205DR-129
30	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=100	29	08_071205DR071205DR	071205DR-130
31	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=10	30	08_071205DR071205DR	071205DR-131
32	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=100	31	08_071205DR071205DR	071205DR-132
33	C0049878	DF06V01RCP0010041007, DF=100	32	08_071205DR071205DR	071205DR-133
34	C0049878 Rep	DF06V01RCP0010041007, DF=100	33	08_071205DR071205DR	071205DR-134
35	C0049878	DF06V01RCP0010041007, DF=10	34	08_071205DR071205DR	071205DR-135
36	C0049878 Rep	DF06V01RCP0010041007, DF=10	35	08_071205DR071205DR	071205DR-136
37	SS7276	Calibration Standard, 5.0 ng/mL	4	08_071205DR071205DR	071205DR-137
38	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=100	36	08_071205DR071205DR	071205DR-138
39	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=10	37	08_071205DR071205DR	071205DR-139
40	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=100	38	08_071205DR071205DR	071205DR-140
41	C0049879	DF06V02AVP0010041007, DF=100	39	08_071205DR071205DR	071205DR-141
42	C0049879 Rep	DF06V02AVP0010041007, DF=100	40	08_071205DR071205DR	071205DR-142
43	C0049879	DF06V02AVP0010041007, DF=10	41	08_071205DR071205DR	071205DR-143
44	C0049879 Rep	DF06V02AVP0010041007, DF=10	42	08_071205DR071205DR	071205DR-144
45	SS7275	Calibration Standard, 10 ng/mL	5	08_071205DR071205DR	071205DR-145
46	C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g, DF=100	43	08_071205DR071205DR	071205DR-146
47	C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g, DF=10	44	08_071205DR071205DR	071205DR-147
48	C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=100	45	08_071205DR071205DR	071205DR-148
49	C0049880	DF06V02PVP0010041007, DF=100	46	08_071205DR071205DR	071205DR-149
50	C0049880 Rep	DF06V02PVP0010041007, DF=100	47	08_071205DR071205DR	071205DR-150
51	C0049880	DF06V02PVP0010041007, DF=10	48	08_071205DR071205DR	071205DR-151
52	C0049880 Rep	DF06V02PVP0010041007, DF=10	49	08_071205DR071205DR	071205DR-152
53	SS7274	Calibration Standard, 25 ng/mL	6	08_071205DR071205DR	071205DR-153

① ② PAP MLC 9/24/05

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AMB 07/22/05

Path: \\Abbeyroad\PE Sciex Data\Projects\P1131 Decatur Monitoring Study Batch:071205DR VEG P760 Tab:Sample Set:SET1 AcqMethod:P760-1131 Isomer Separatic

	Sample Name	Sample ID	Vial Position	Data File	Run #
54	SS7273	Calibration Standard, 50 ng/mL	7	08_071205DR\071205DR	071205DR-154

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Study No: NA

Exygen Study No: P760/P1131

Instrument: API 4000 LC/MS/MS System, (LC/MS/MS #8)
Turbo Ion Spray Liquid Introduction Interface

Computer: DELL OptiPlex GX400

Software: Windows NT, Analyst 1.4

HPLC Equipment: 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 (Exygen ID MA0018925)

Column Temperature: 35°C

Mobile Phase (A) : 2 mM ammonium acetate in water (SL0014984)

Mobile Phase (B) : Methanol (RE0020196)

<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

Injected Volume: 15 µL

Ions monitored:

<u>Analyte</u>	<u>Mode</u>	<u>Transition Monitored</u>
PFBS	negative	299 → 99
PFHS	negative	399 → 80
PFOS	negative	499 → 80
PFOA	negative	413 → 369
PFOA Confirm Ion	negative	413 → 219
¹³ C PFOA	negative	415 → 370

Analyst: Amy Sheehan *AMS 07/22/05*
Exygen Research
3058 Research Drive, State College, PA 16801
Phone : (814) 272-1039 FAX : (814) 231-1580

NOTE: ALL HANDWRITTEN PEAK IDENTIFICATIONS BY CEE / 07/26/05

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CEE 08/02/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
1 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	174611	0.500	0.360	071205DR-101
2 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	324088	1.00	0.977	071205DR-102
3 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	791206	2.50	2.90	071205DR-103
4 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1609734	5.00	6.28	071205DR-104
5 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2891639	10.0	11.6	071205DR-105
6 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	6627517	25.0	27.0	071205DR-106
7 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	12158513	50.0	49.8	071205DR-107
8 Methanol Wash	Methanol Wash	Unknown	1	PFOA	16847	N/A	< 0	071205DR-108
9 C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	720266	* 25.0	261	071205DR-109
10 C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	6079998	* 25.0	247	071205DR-110
11 C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	1060489	* 250	402	071205DR-111
12 C0049876	DF06V01AVP0010041007, DF=100	Unknown	100	PFOA	647854	* N/A	231	071205DR-112
13 C0049876 Rep	DF06V01AVP0010041007, DF=100	Unknown	100	PFOA	619660	* N/A	220	071205DR-113
14 C0049876	DF06V01AVP0010041007, DF=10	Unknown	10	PFOA	5547750	* N/A	225	071205DR-114
15 C0049876 Rep	DF06V01AVP0010041007, DF=10	Unknown	10	PFOA	4838272	* N/A	196	071205DR-115
16 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	166503	0.500	0.326	071205DR-116
17 C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=1000	Quality Control	1000	PFOA	1202424	* 25.0	4600	071205DR-117
18 C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	8606920	* 25.0	3520	071205DR-118
19 C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	42307709	* 25.0	1740	071205DR-119
20 C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=1000	Quality Control	1000	PFOA	1140004	* 250	4340	071205DR-120
21 C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	9045369	* 250	3700	071205DR-121
22 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	307011	1.00	0.806	071205DR-122
23 C0049877	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOA	1324428	* N/A	5100	071205DR-123
24 C0049877 Rep	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOA	1145416	* N/A	4370	071205DR-124
25 C0049877	DF06V01PAP0010041007, DF=100	Unknown	100	PFOA	9251337	* N/A	3780	071205DR-125
26 C0049877 Rep	DF06V01PAP0010041007, DF=100	Unknown	100	PFOA	8849443	* N/A	3620	071205DR-126
27 C0049877	DF06V01PAP0010041007, DF=10	Unknown	10	PFOA	44632879	* N/A	1840	071205DR-127
28 C0049877 Rep	DF06V01PAP0010041007, DF=10	Unknown	10	PFOA	42664598	* N/A	1760	071205DR-128
29 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	720897	2.50	2.61	071205DR-129
30 C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	1695021	* 25.0	663	071205DR-130
31 C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	13411624	* 25.0	550	071205DR-131
32 C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	2009465	* 250	793	071205DR-132
33 C0049878	DF06V01RCP0010041007, DF=100	Unknown	100	PFOA	1526180	* N/A	594	071205DR-133
34 C0049878 Rep	DF06V01RCP0010041007, DF=100	Unknown	100	PFOA	1630127	* N/A	637	071205DR-134
35 C0049878	DF06V01RCP0010041007, DF=10	Unknown	10	PFOA	12283753	* N/A	504	071205DR-135
36 C0049878 Rep	DF06V01RCP0010041007, DF=10	Unknown	10	PFOA	12748494	* N/A	522	071205DR-136
37 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1477271	5.00	5.74	071205DR-137
38 C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	356131	* 25.0	111	071205DR-138
39 C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	3076112	* 25.0	123	071205DR-139
40 C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	711312	* 250	257	071205DR-140
41 C0049879	DF06V02AVP0010041007, DF=100	Unknown	100	PFOA	329013	* N/A	99.7	071205DR-141
42 C0049879 Rep	DF06V02AVP0010041007, DF=100	Unknown	100	PFOA	268561	* N/A	74.8	071205DR-142
43 C0049879	DF06V02AVP0010041007, DF=10	Unknown	10	PFOA	2912346	* N/A	117	071205DR-143
44 C0049879 Rep	DF06V02AVP0010041007, DF=10	Unknown	10	PFOA	2373516	* N/A	94.3	071205DR-144
45 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2605000	10.0	10.4	071205DR-145
46 C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	3091503	* 25.0	1240	071205DR-146
47 C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	22248410	* 25.0	914	071205DR-147
48 C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	3353984	* 250	1360	071205DR-148
49 C0049880	DF06V02PVP0010041007, DF=100	Unknown	100	PFOA	2753273	* N/A	1100	071205DR-149
50 C0049880 Rep	DF06V02PVP0010041007, DF=100	Unknown	100	PFOA	2935446	* N/A	1180	071205DR-150
51 C0049880	DF06V02PVP0010041007, DF=10	Unknown	10	PFOA	20641529	* N/A	848	071205DR-151
52 C0049880 Rep	DF06V02PVP0010041007, DF=10	Unknown	10	PFOA	20694395	* N/A	850	071205DR-152
53 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5894625	25.0	24.0	071205DR-153
54 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	11033219	50.0	45.2	071205DR-154

* Analysis not required - BAK 08/02/05

① (R) PAP m/c 9/2/05

Vegetation Conversion

Compound: PFOA

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049876	DF06-V01-AVP001-0-041007	225	90.0
C0049876 Rep	DF06-V01-AVP001-0-041007	196	78.4
C0049876 Spk C	DF06-V01-AVP001-0-041007	247	98.8
C0049877	DF06-V01-PAP001-0-041007	3780	1512
C0049877 Rep	DF06-V01-PAP001-0-041007	3620	1448
C0049877 Spk E	DF06-V01-PAP001-0-041007	3520	1408
C0049877 Spk F	DF06-V01-PAP001-0-041007	3700	1480
C0049878	DF06-V01-RCP001-0-041007	594	238
C0049878 Rep	DF06-V01-RCP001-0-041007	637	255
C0049878 Spk G	DF06-V01-RCP001-0-041007	663	265
C0049878 Spk H	DF06-V01-RCP001-0-041007	793	317
C0049879	DF06-V02-AVP001-0-041007	117	46.8
C0049879 Rep	DF06-V02-AVP001-0-041007	94.3	37.7
C0049879 Spk I	DF06-V02-AVP001-0-041007	123	49.2
C0049880	DF06-V02-PVP001-0-041007 ^①	1100	440
C0049880 Rep	DF06-V02-PVP001-0-041007 ^②	1180	472
C0049880 Spk K	DF06-V02-PVP001-0-041007 ^③	1240	496
C0049880 Spk L	DF06-V02-PVP001-0-041007 ^④	1350	540

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = Peak Area 0 At or Above 0.2 ng/g^①

NQ = Not quantifiable = Measured concentration below Limit of Quantitation (LOQ) which is 0.7 ng/g.

↑
Between 0.2 ng/g And ①

① (RE) MLC 9/20/05

② (RE) PAP MLC 9/21/05

0286
BML 09/05/05

	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound: PFOA		Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049876	DF06-V01-AVP001-0-041007	=Raw Data!H193	=(C9*2)/5
10	C0049876 Rep	DF06-V01-AVP001-0-041007	=Raw Data!H194	=(C10*2)/5
11	C0049876 Spk C	DF06-V01-AVP001-0-041007	=Raw Data!H189	=(C11*2)/5
12				
13	C0049877	DF06-V01-PAP001-0-041007	=Raw Data!H204	=(C13*2)/5
14	C0049877 Rep	DF06-V01-PAP001-0-041007	=Raw Data!H205	=(C14*2)/5
15	C0049877 Spk E	DF06-V01-PAP001-0-041007	=Raw Data!H197	=(C15*2)/5
16	C0049877 Spk F	DF06-V01-PAP001-0-041007	=Raw Data!H200	=(C16*2)/5
17				
18	C0049878	DF06-V01-RCP001-0-041007	=Raw Data!H212	=(C18*2)/5
19	C0049878 Rep	DF06-V01-RCP001-0-041007	=Raw Data!H213	=(C19*2)/5
20	C0049878 Spk G	DF06-V01-RCP001-0-041007	=Raw Data!H209	=(C20*2)/5
21	C0049878 Spk H	DF06-V01-RCP001-0-041007	=Raw Data!H211	=(C21*2)/5
22				
23	C0049879	DF06-V02-AVP001-0-041007	=Raw Data!H222	=(C23*2)/5
24	C0049879 Rep	DF06-V02-AVP001-0-041007	=Raw Data!H223	=(C24*2)/5
25	C0049879 Spk I	DF06-V02-AVP001-0-041007	=Raw Data!H218	=(C25*2)/5
26				
27	C0049880	DF06-V02-PVP001-0-041007	=Raw Data!H228	=(C27*2)/5
28	C0049880 Rep	DF06-V02-PVP001-0-041007	=Raw Data!H229	=(C28*2)/5
29	C0049880 Spk K	DF06-V02-PVP001-0-041007	=Raw Data!H225	=(C29*2)/5
30	C0049880 Spk L	DF06-V02-PVP001-0-041007	=Raw Data!H227	=(C30*2)/5
31				

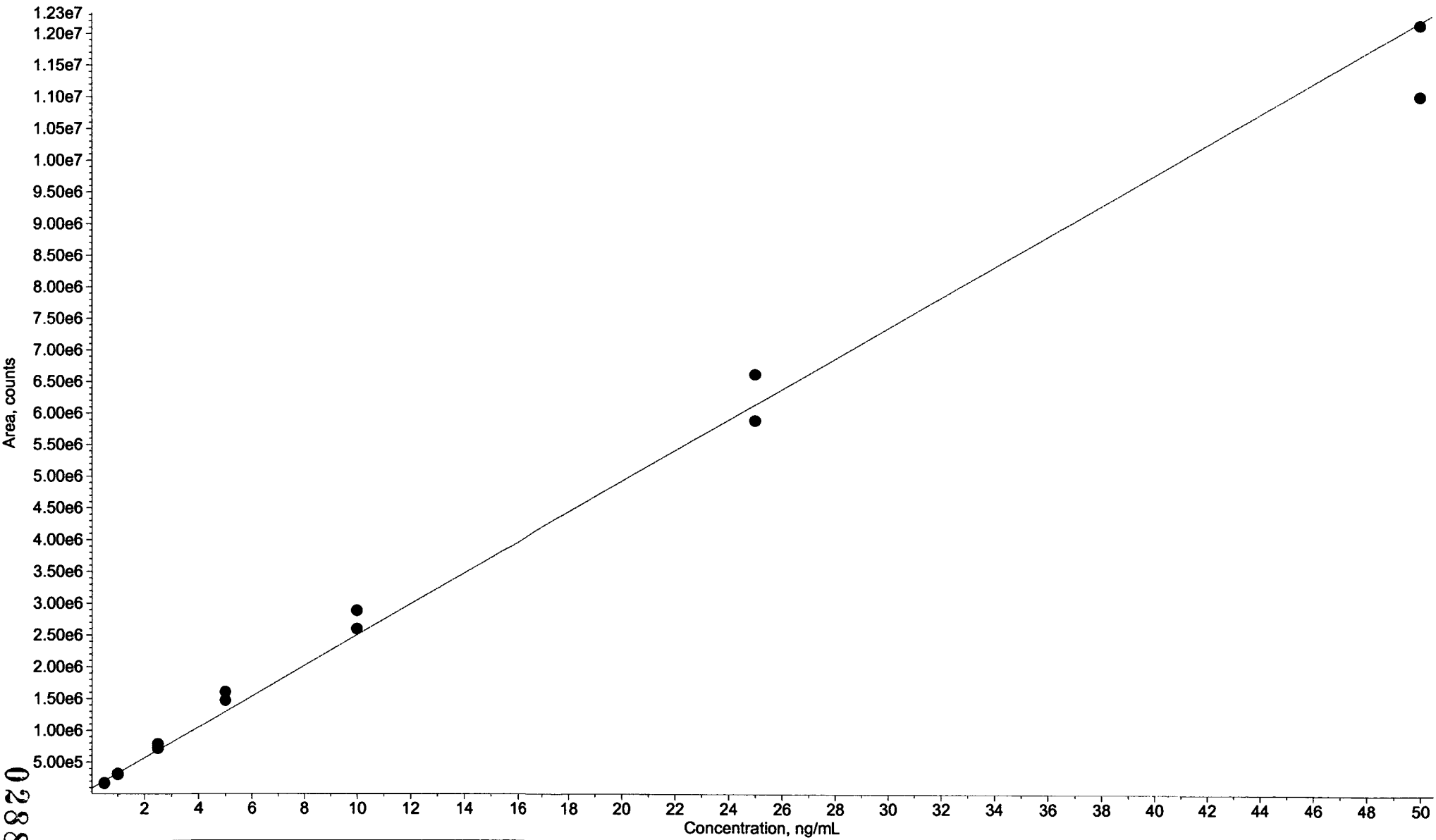
① RE PAP mLC 9/21/05

0287

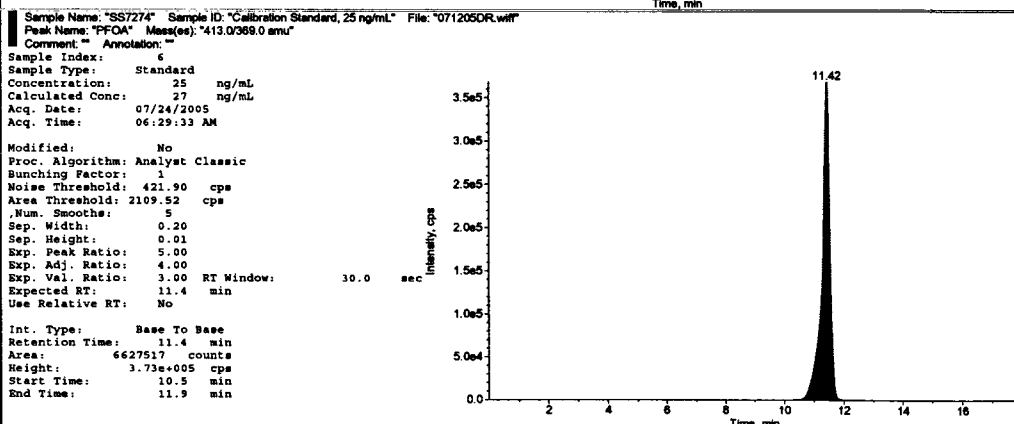
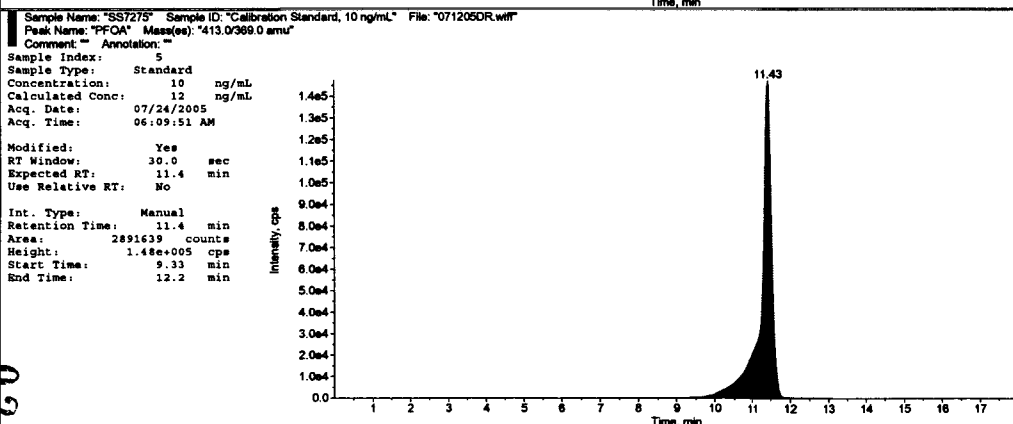
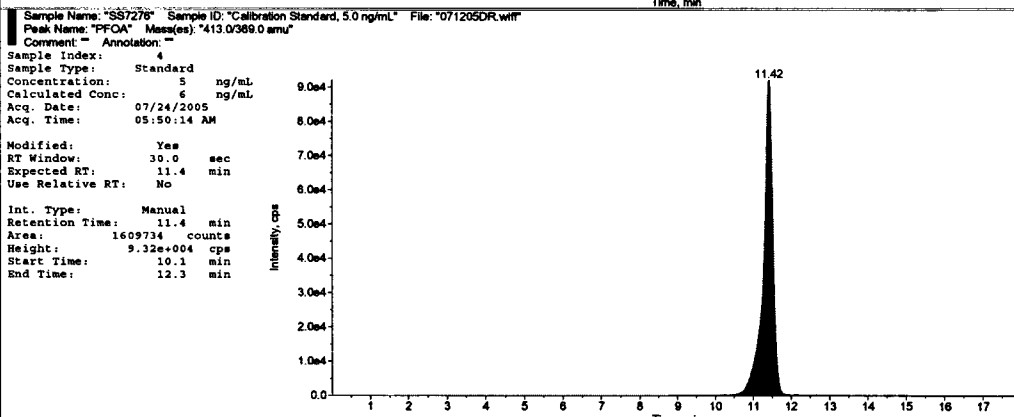
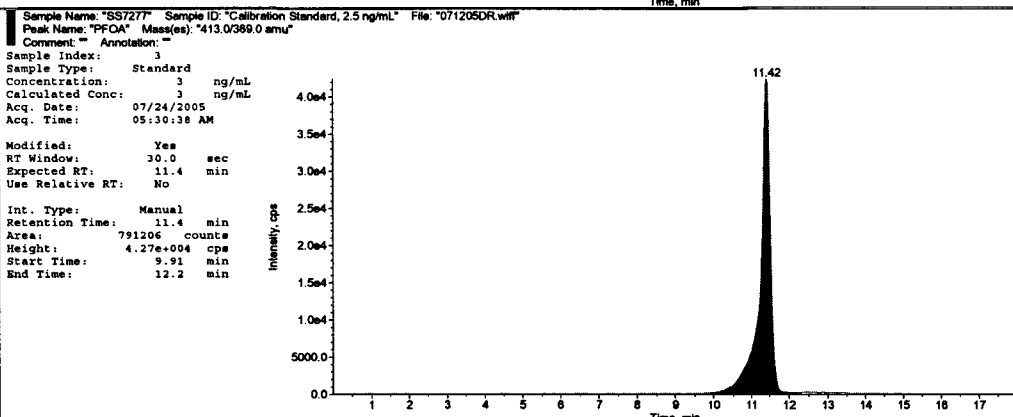
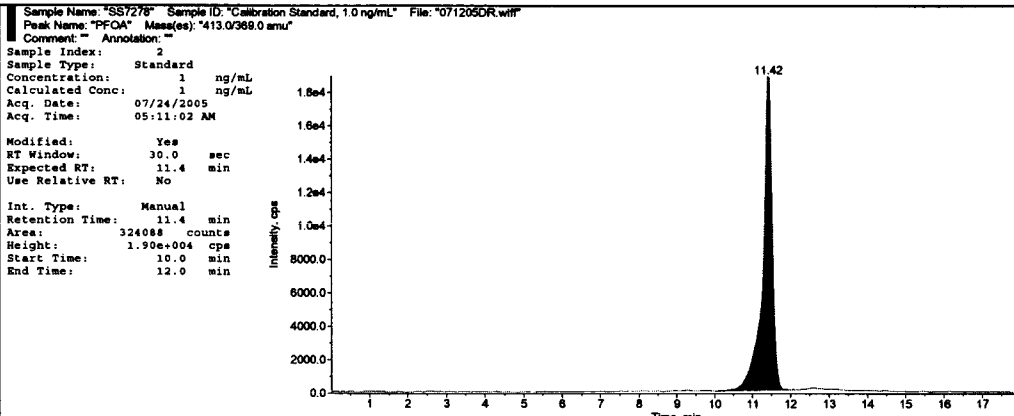
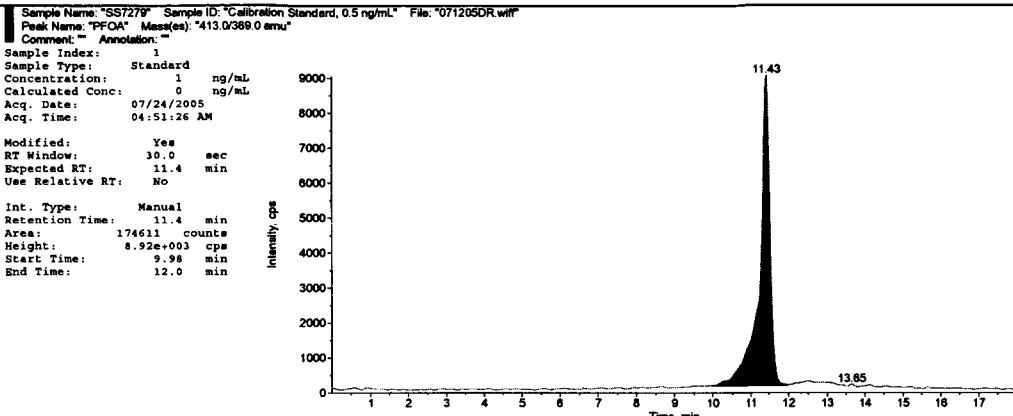
BANK 08/03/05

CEE 07/26/05

071205DR VEG.rdb (PFOA): "Linear" Regression ("1 / x" weighting): $y = 2.42e+005 x + 8.74e+004$ ($r = 0.9953$)

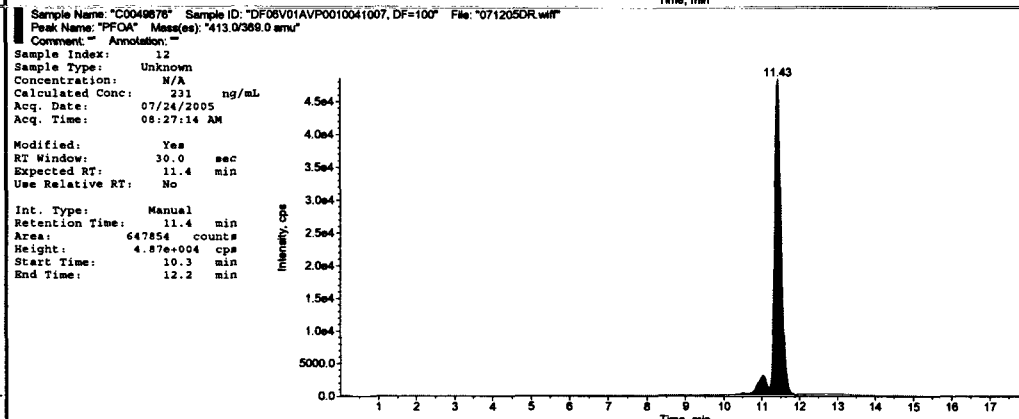
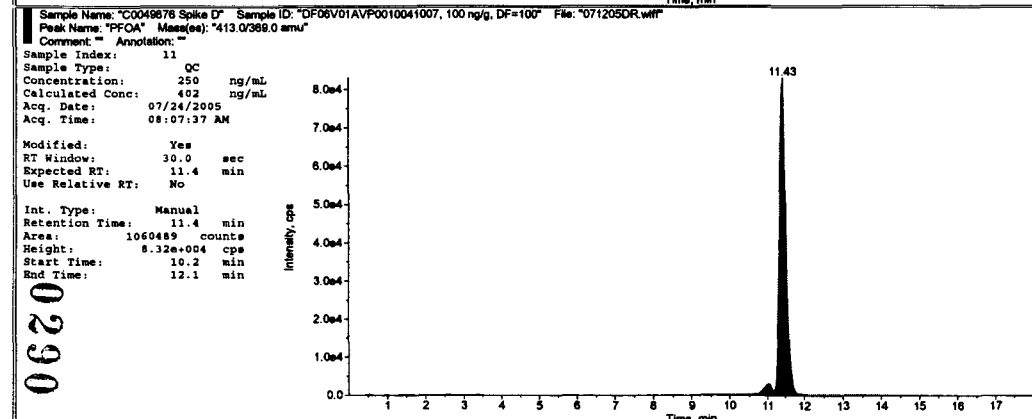
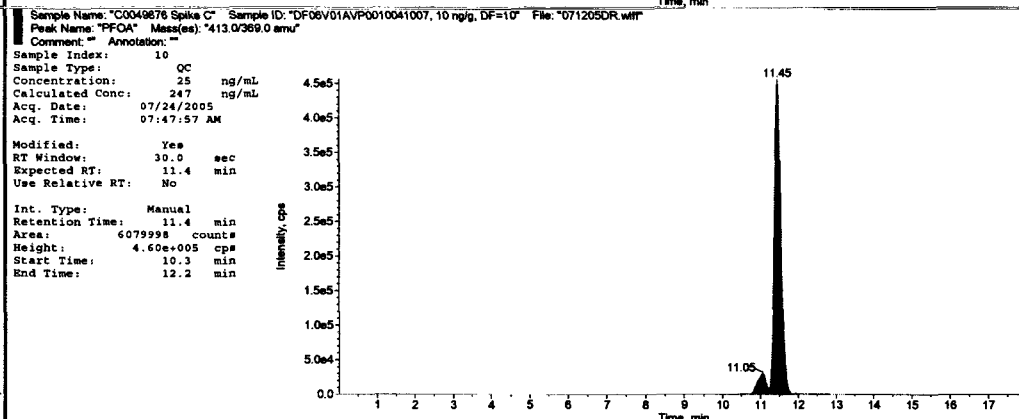
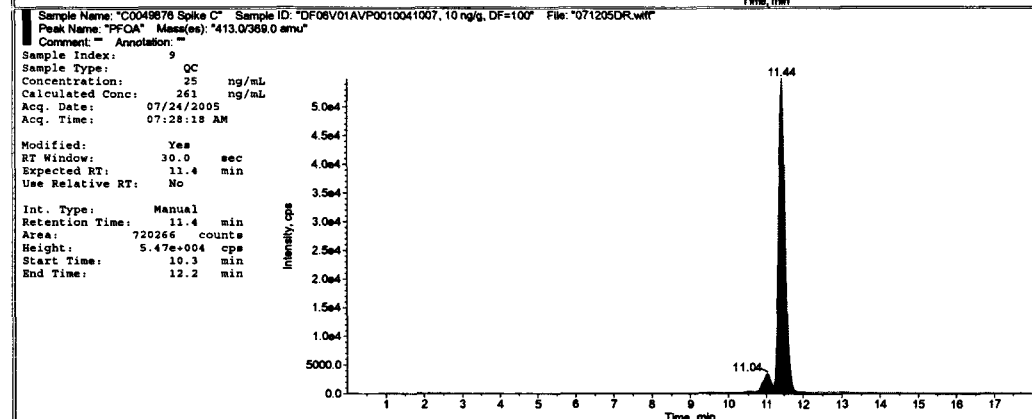
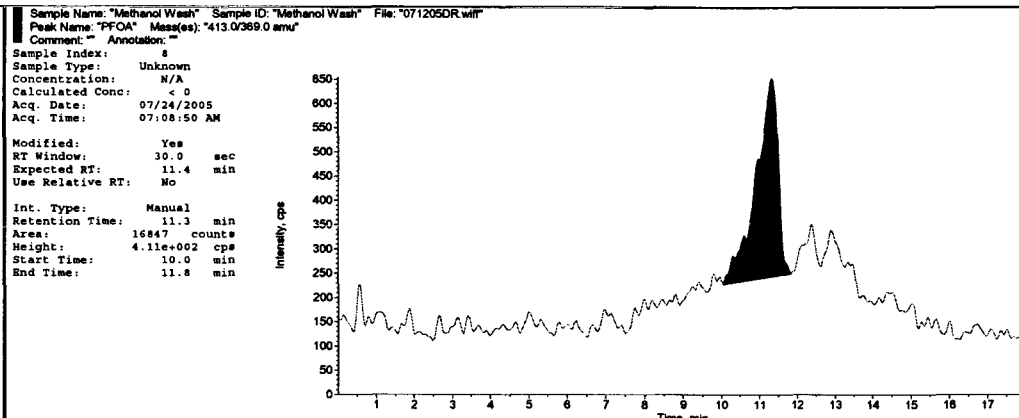
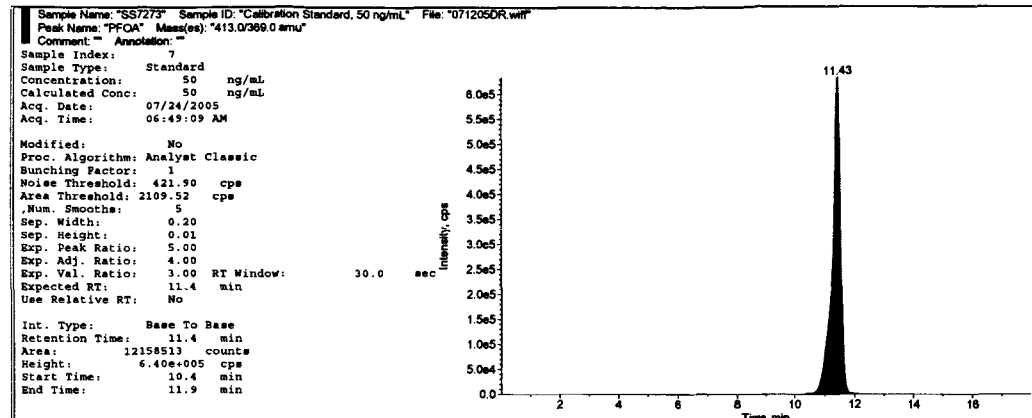


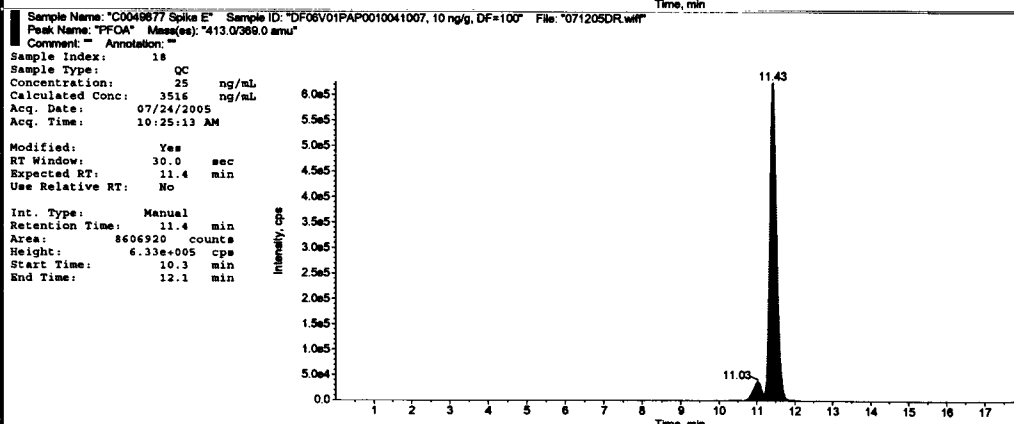
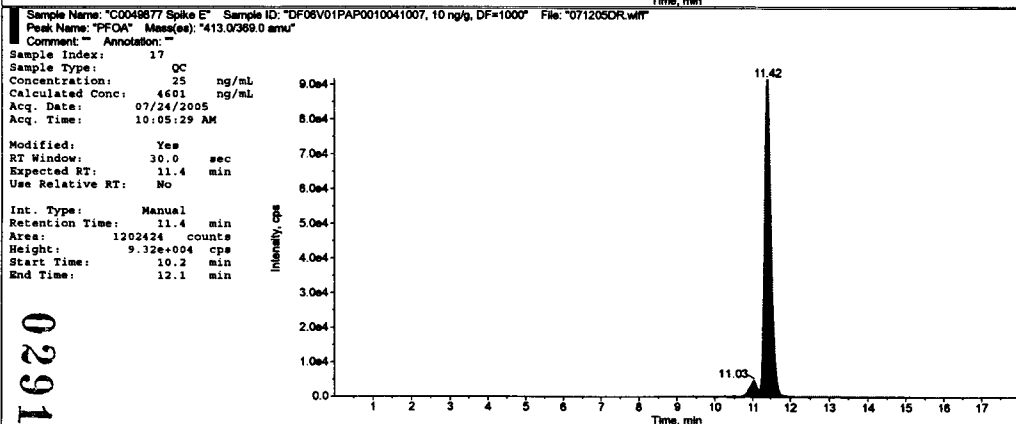
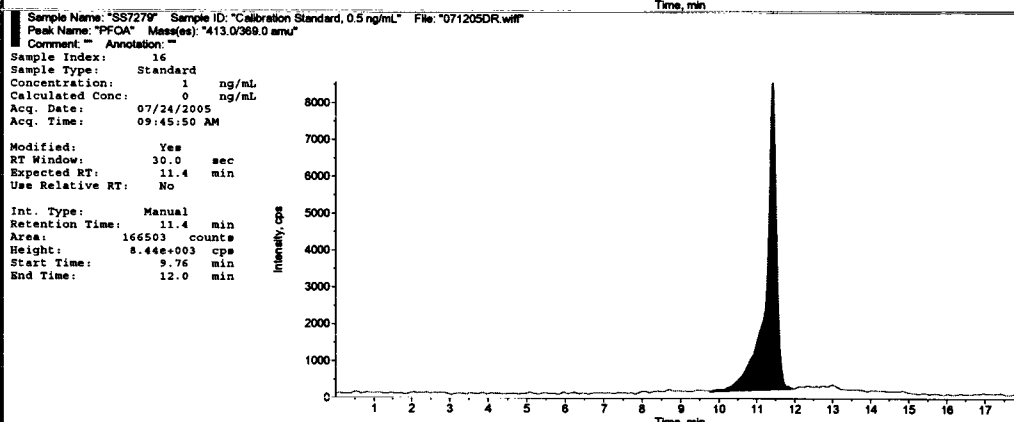
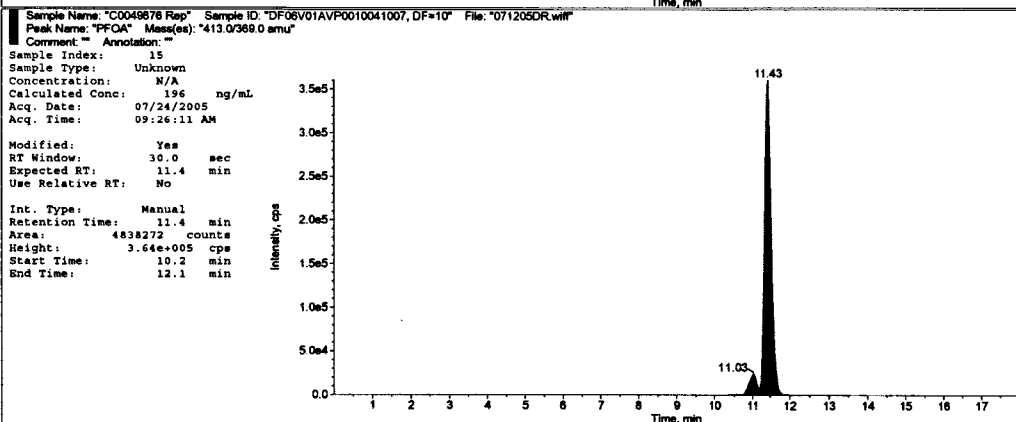
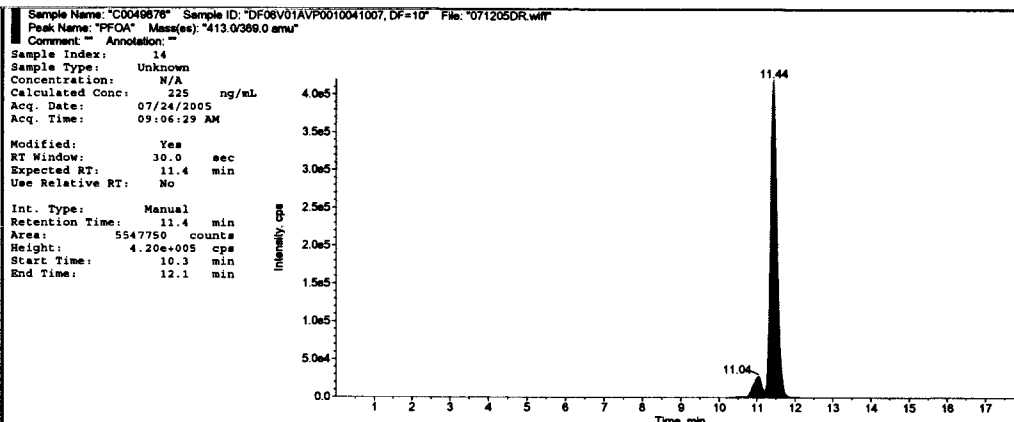
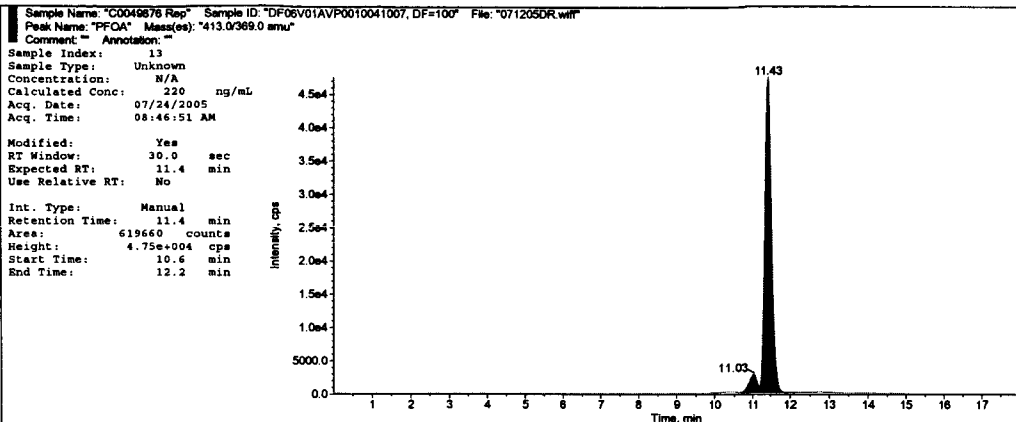
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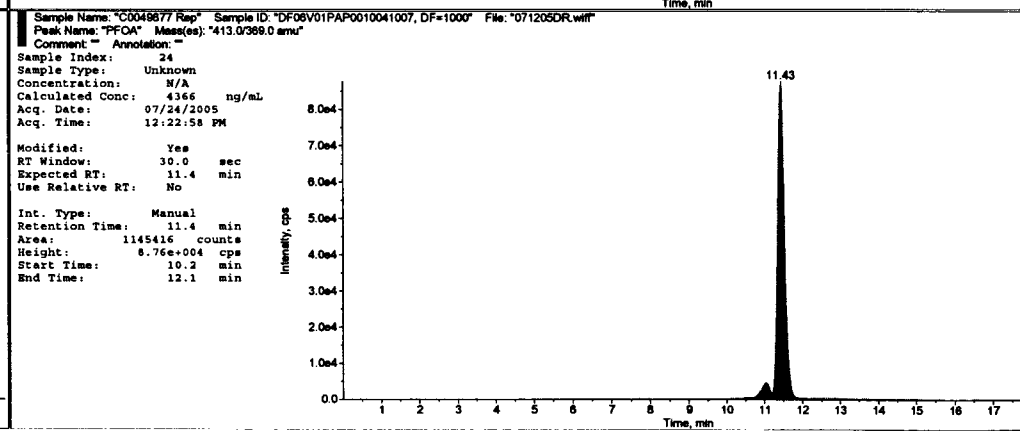
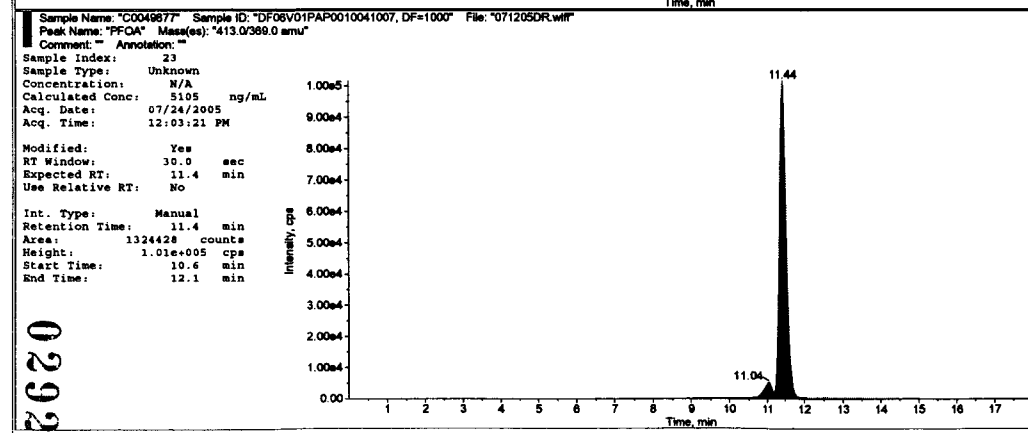
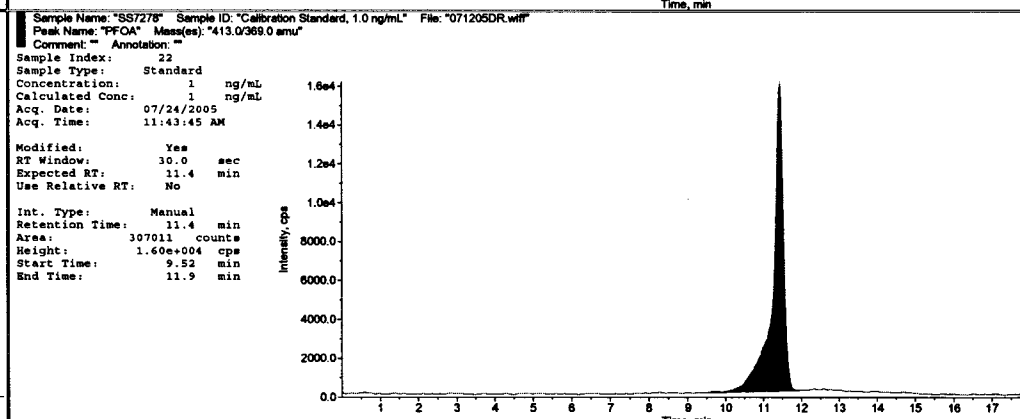
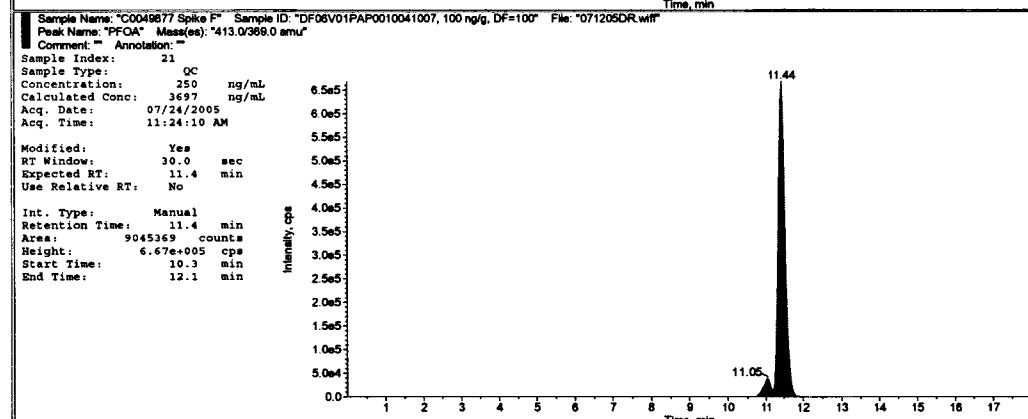
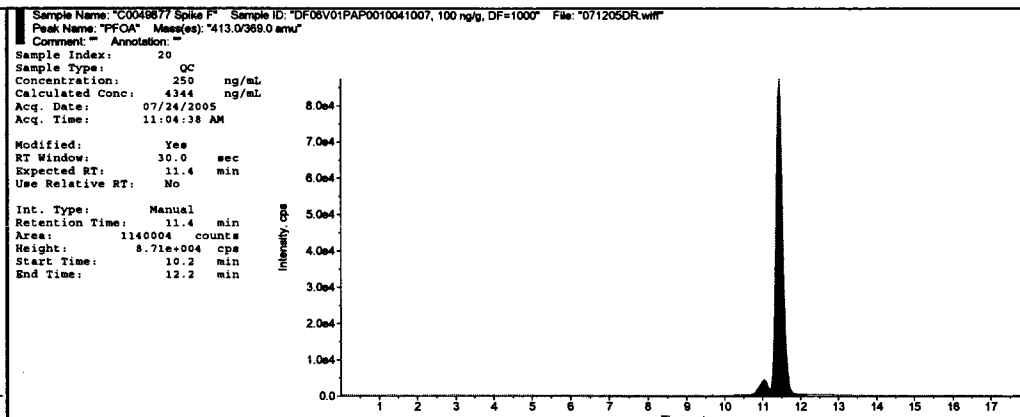
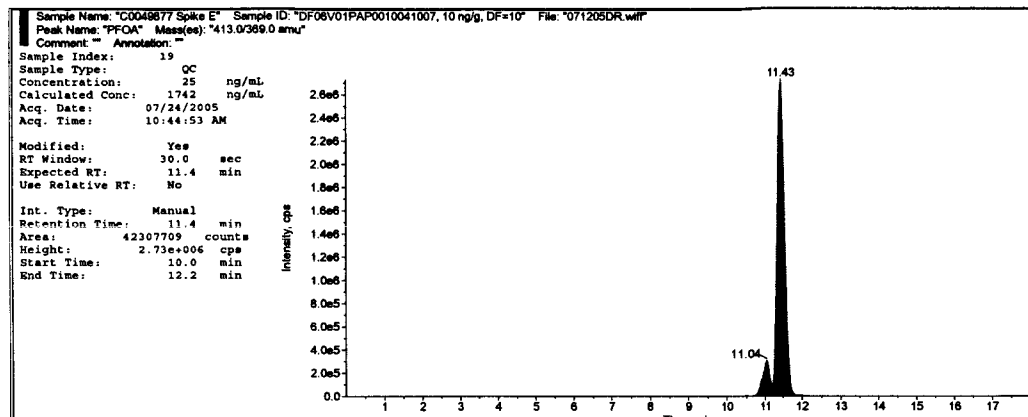
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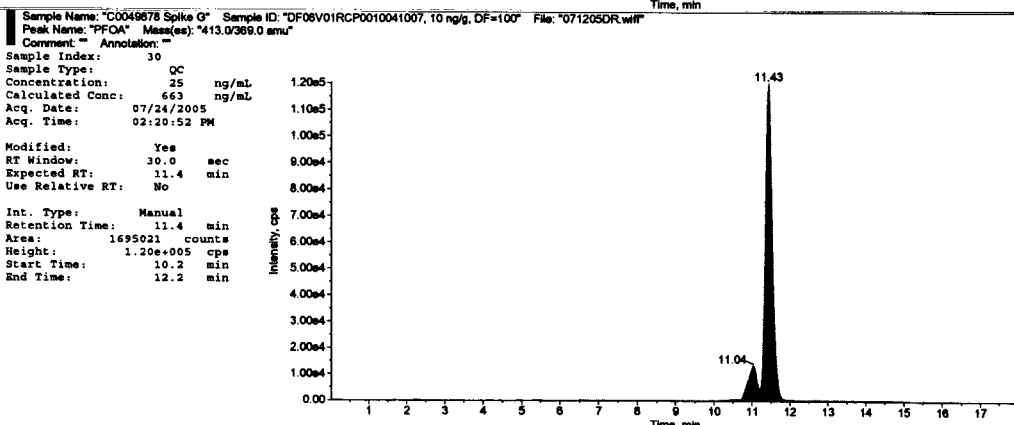
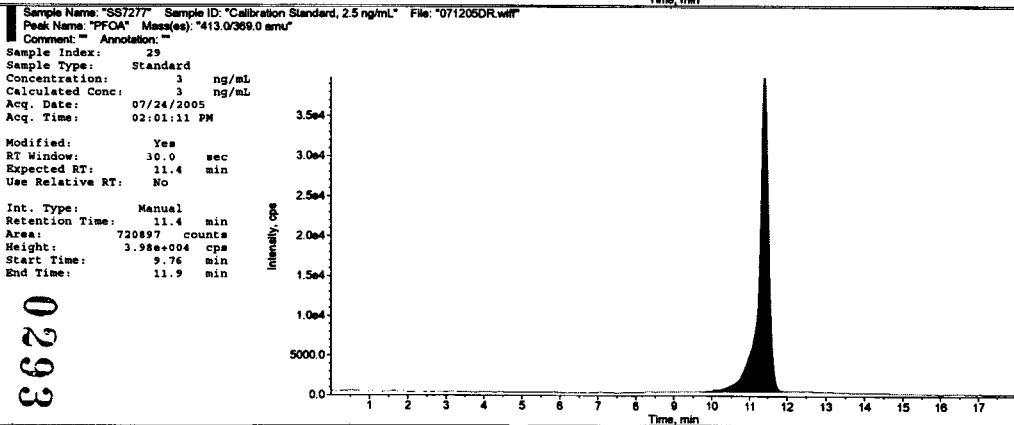
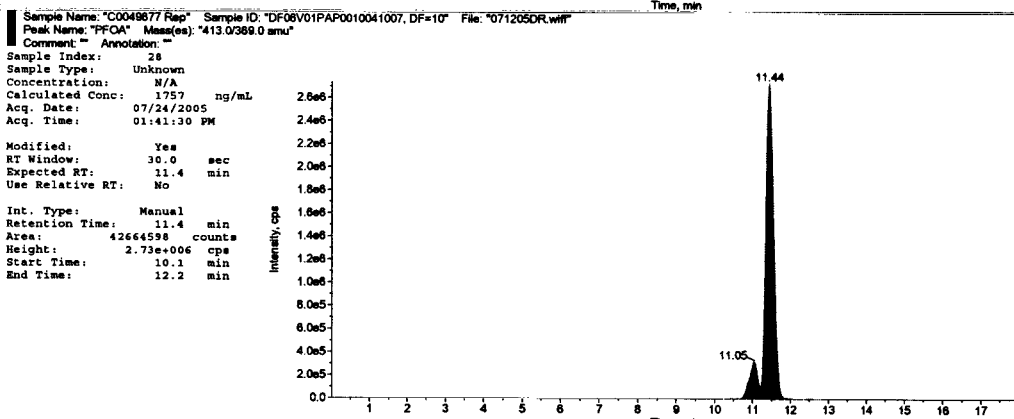
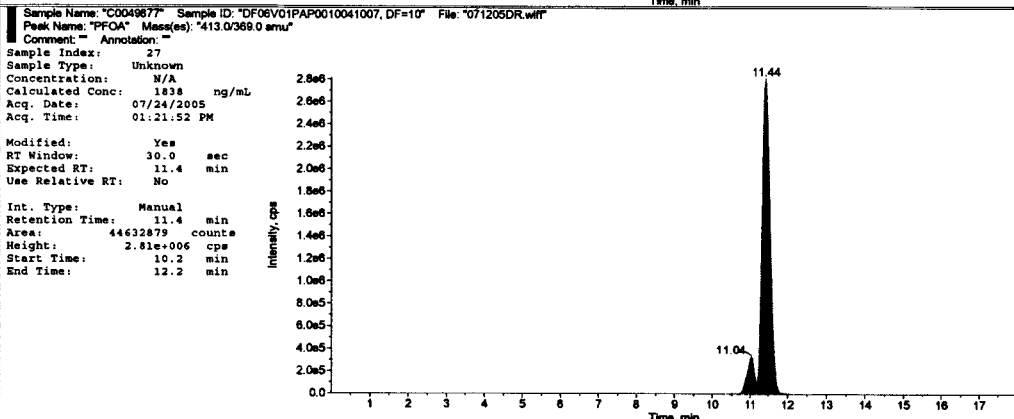
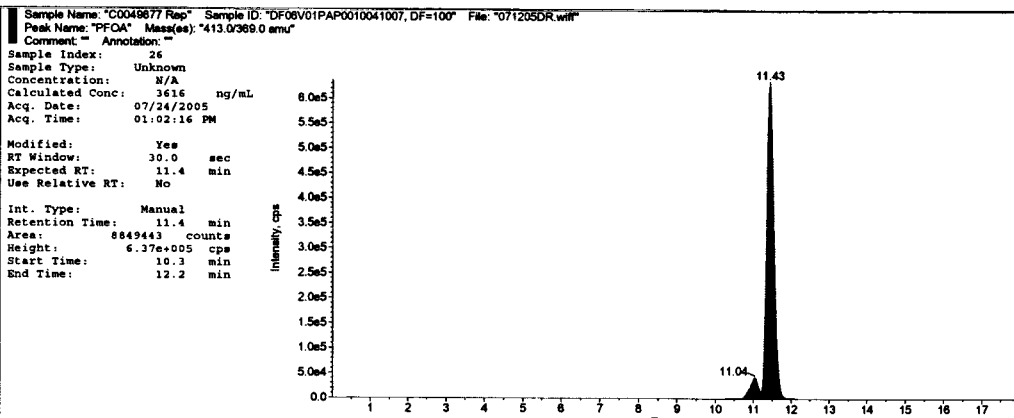
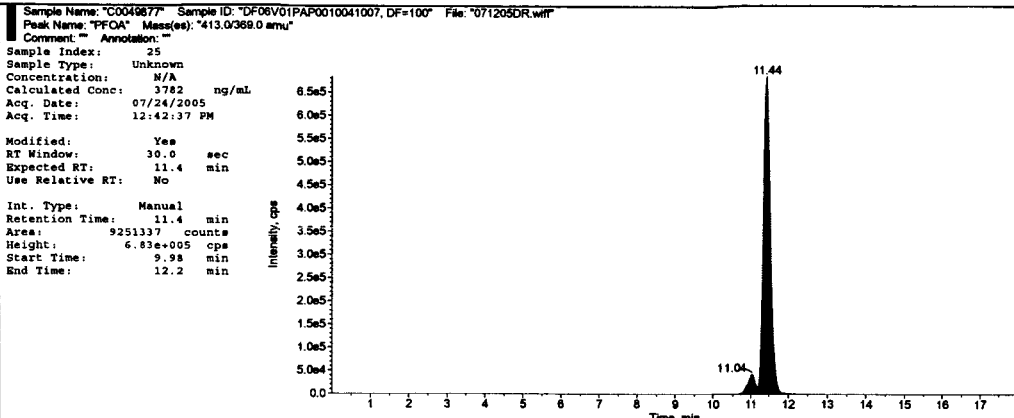


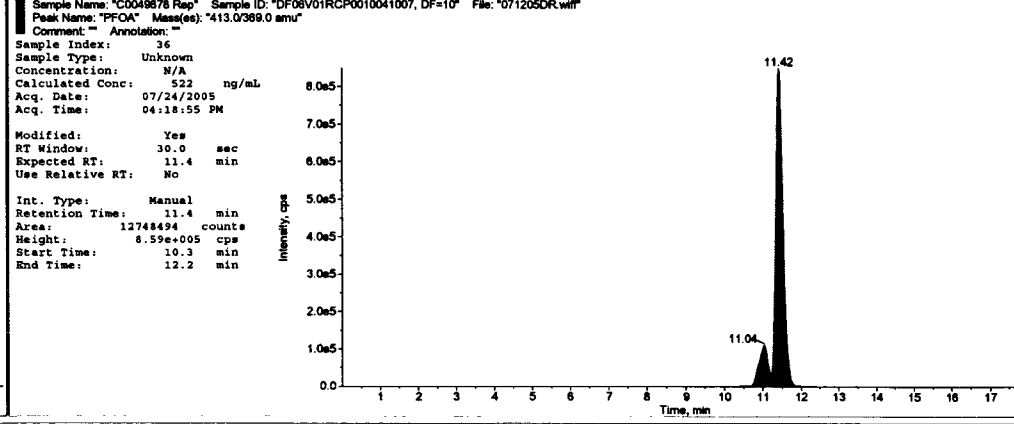
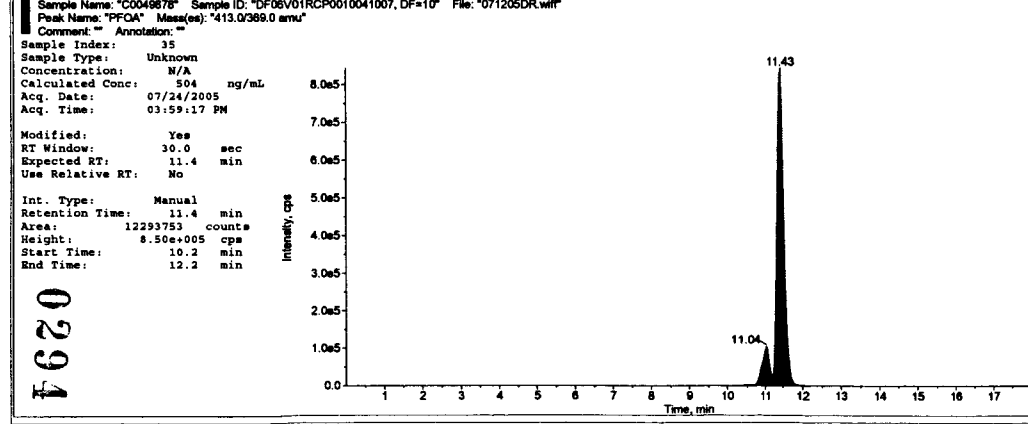
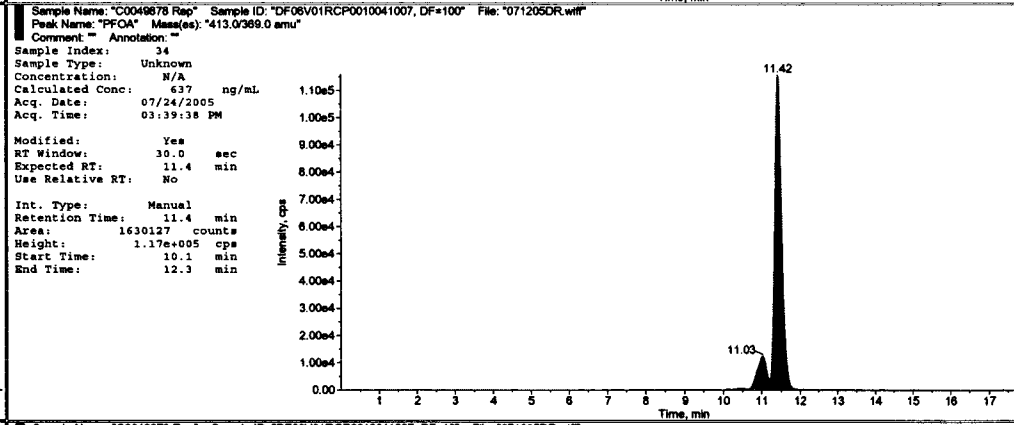
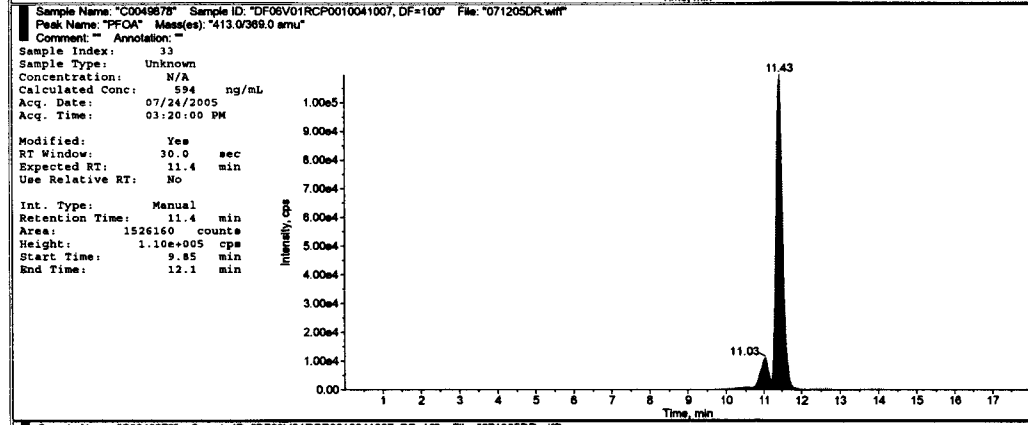
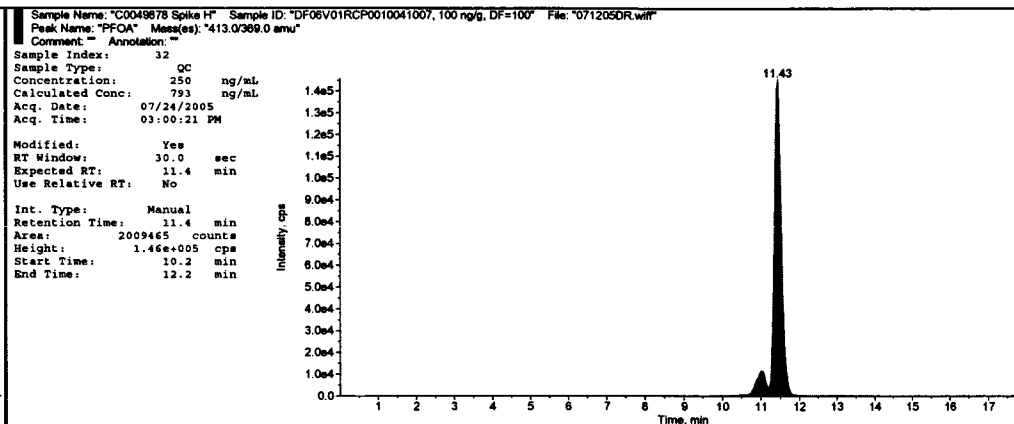
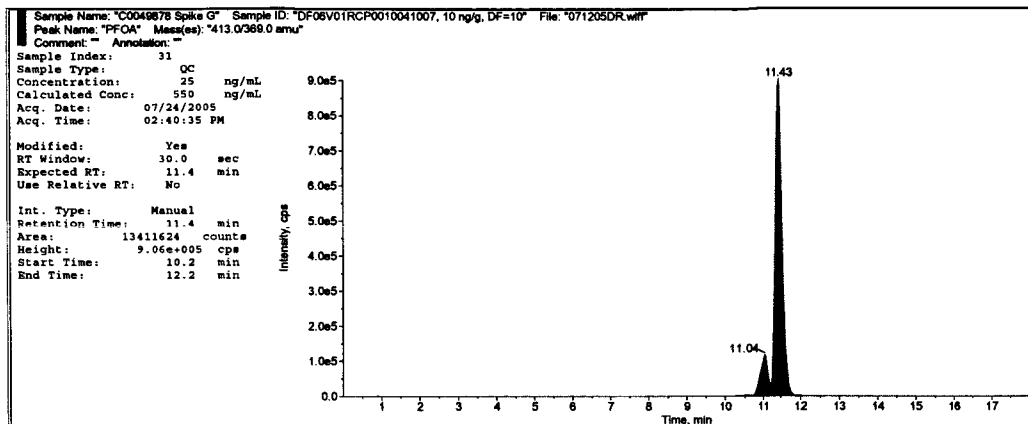


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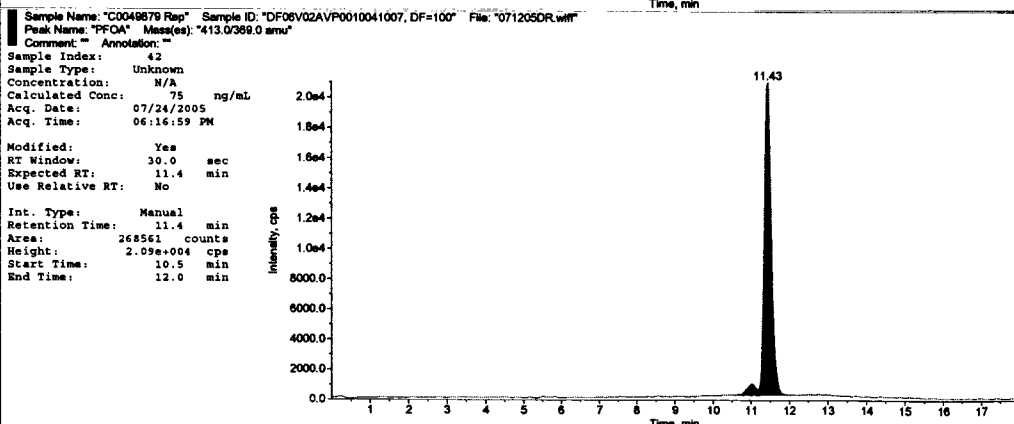
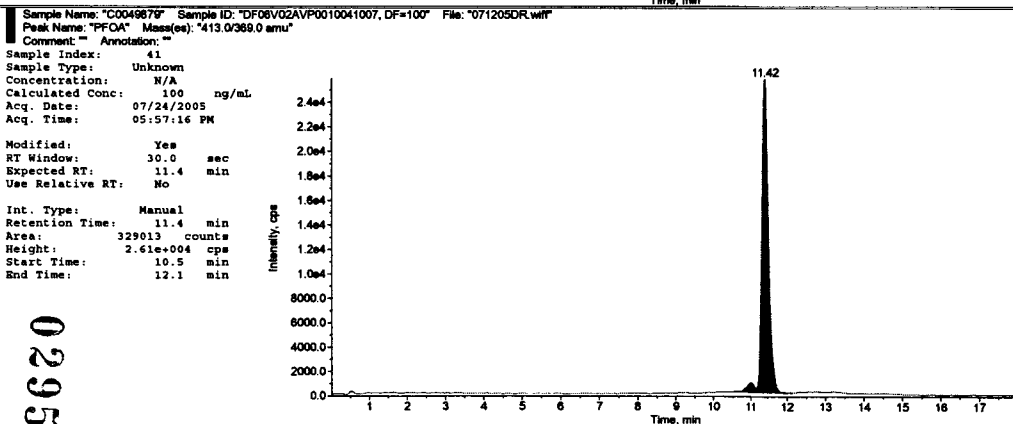
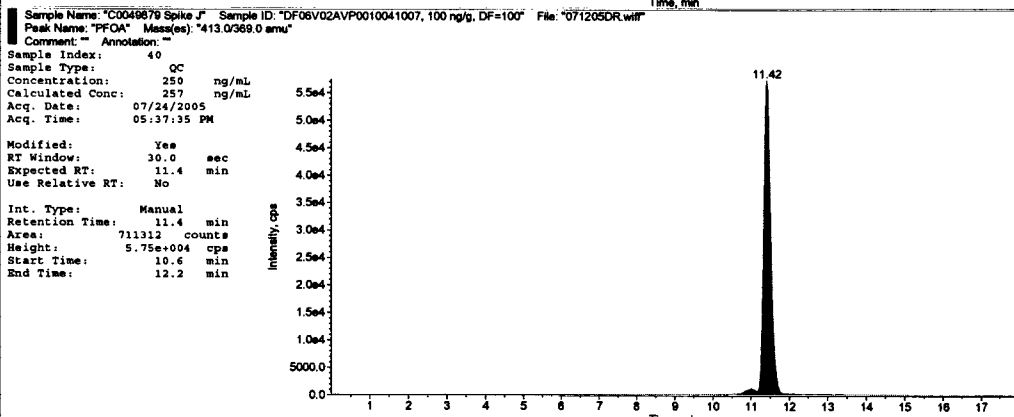
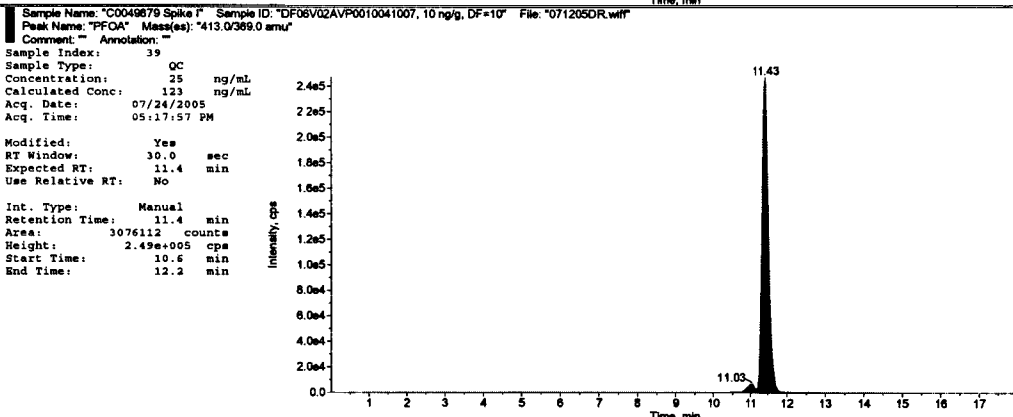
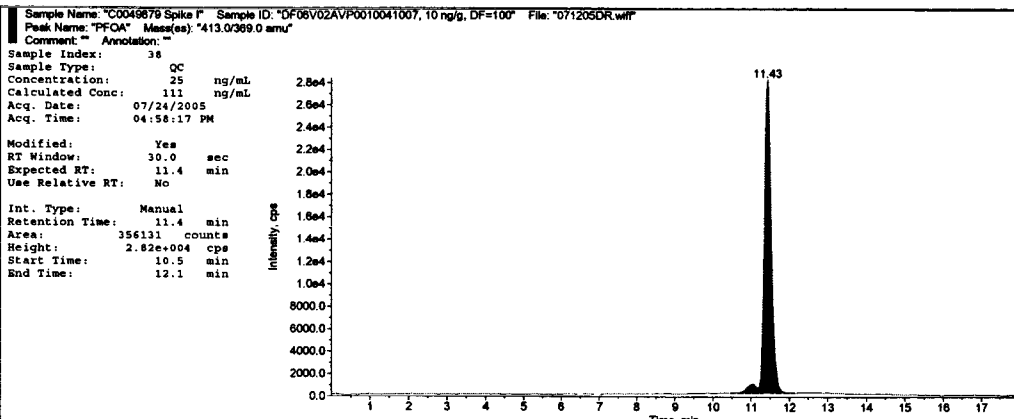
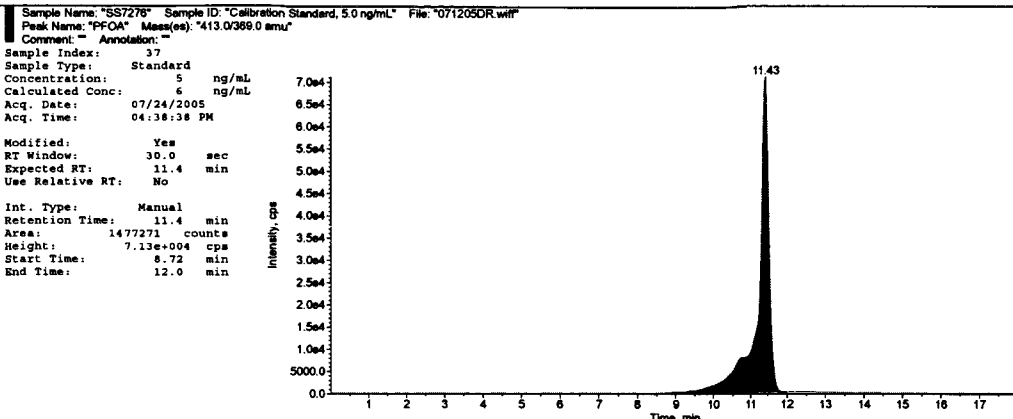


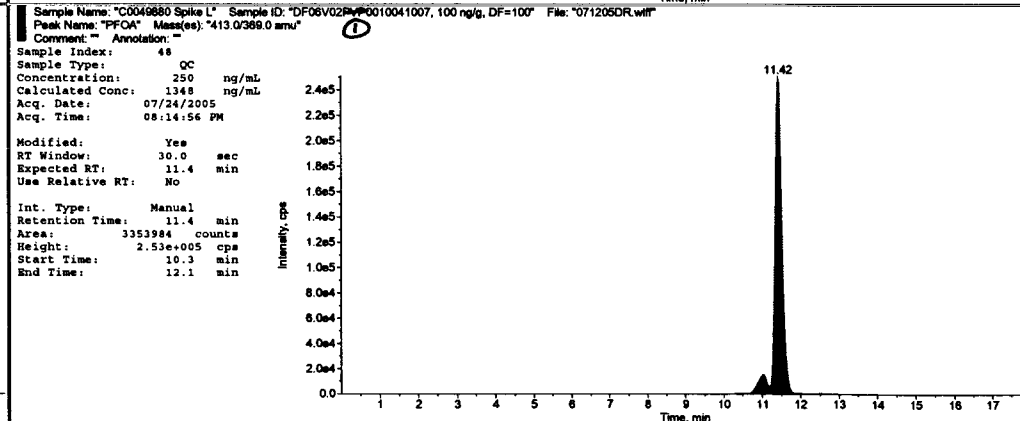
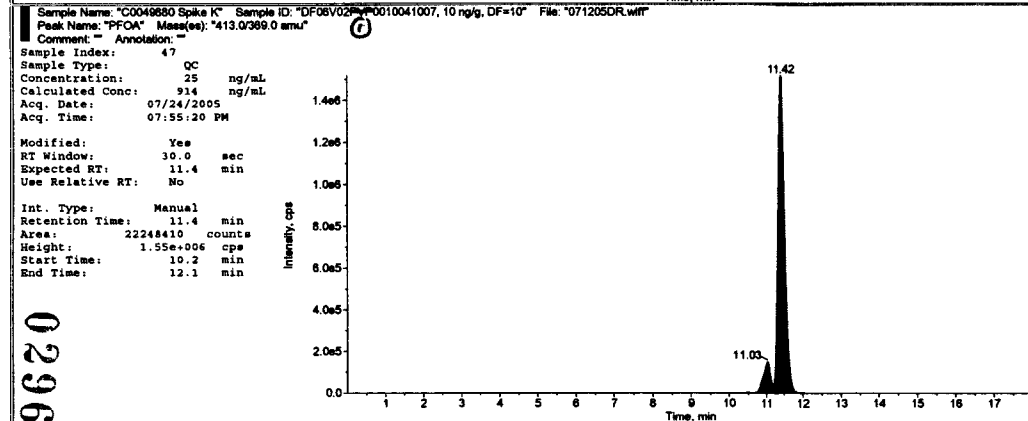
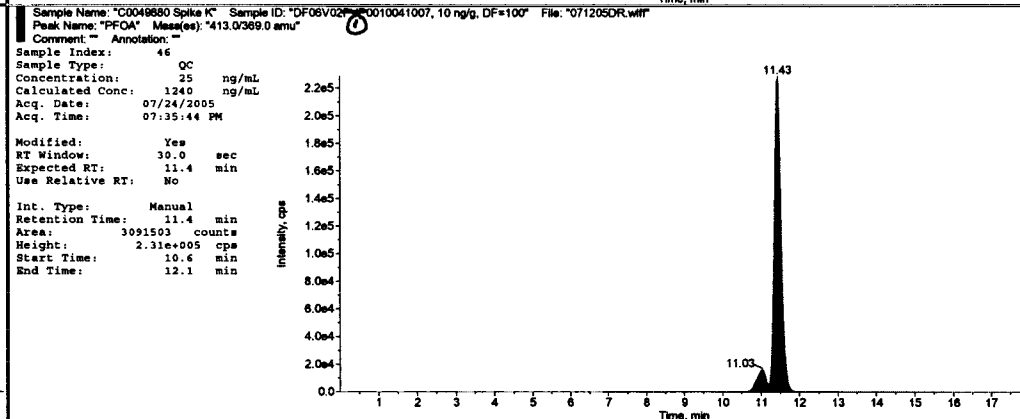
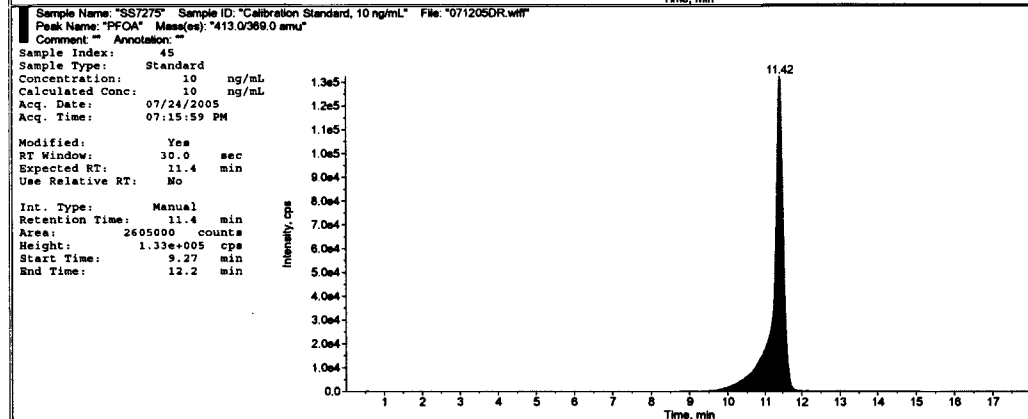
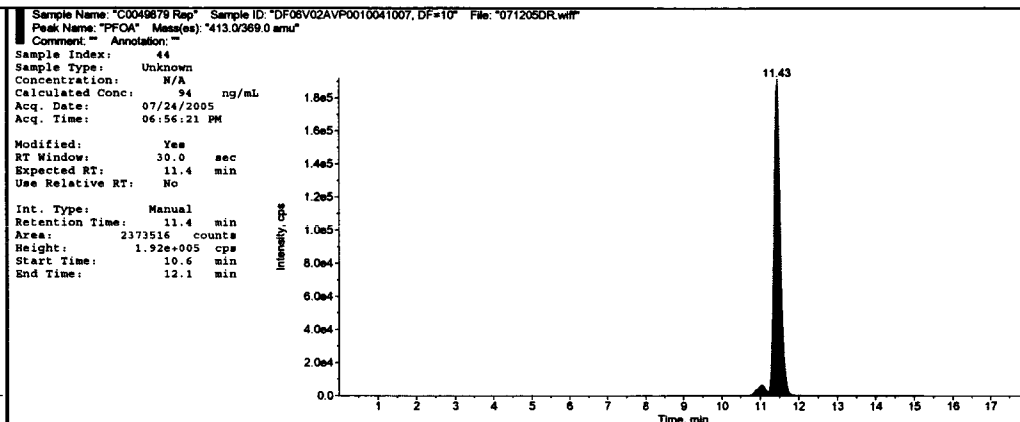
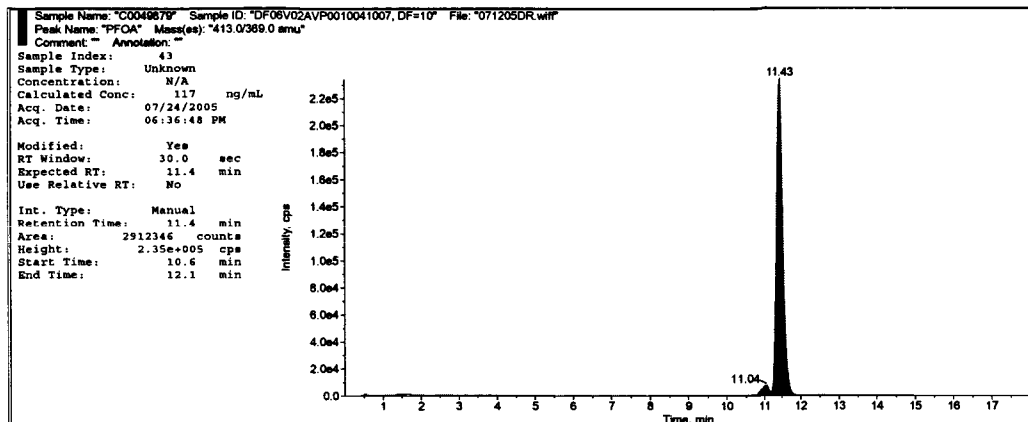
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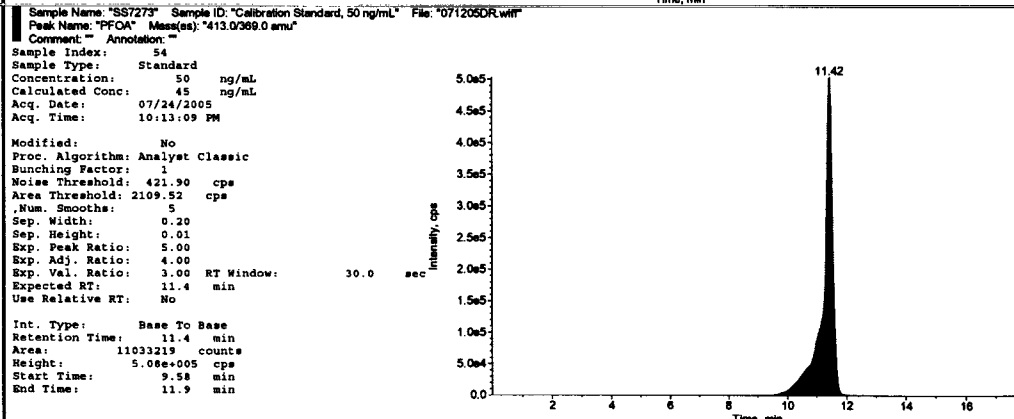
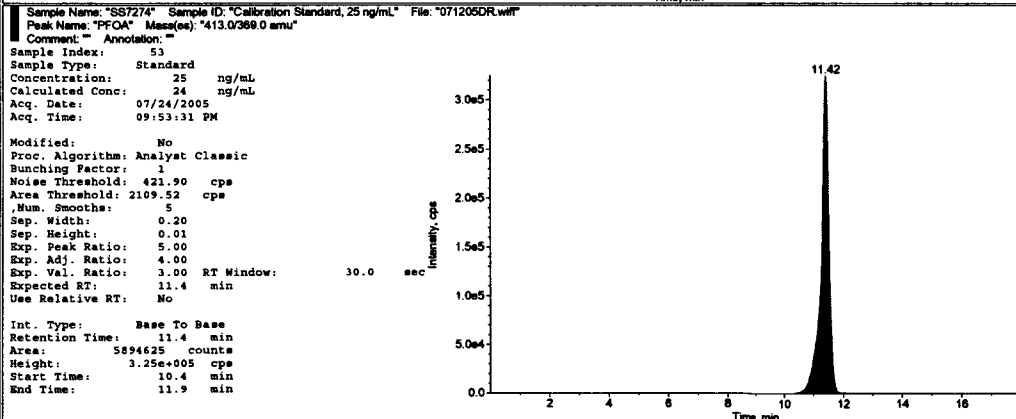
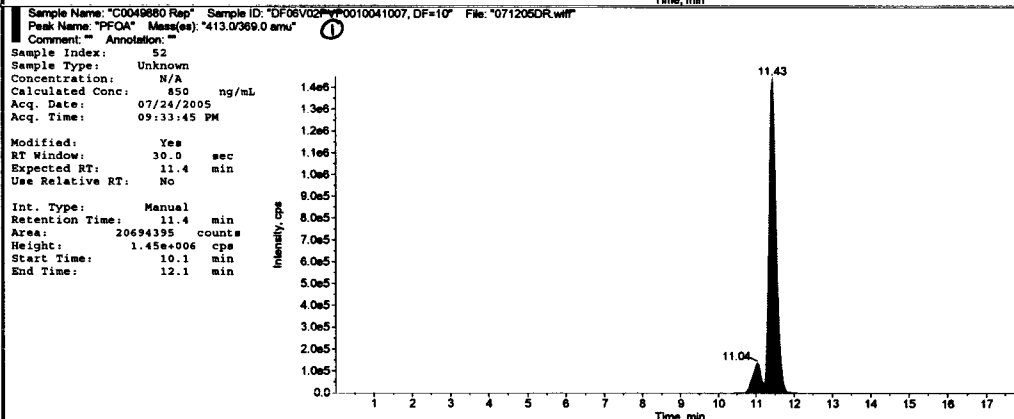
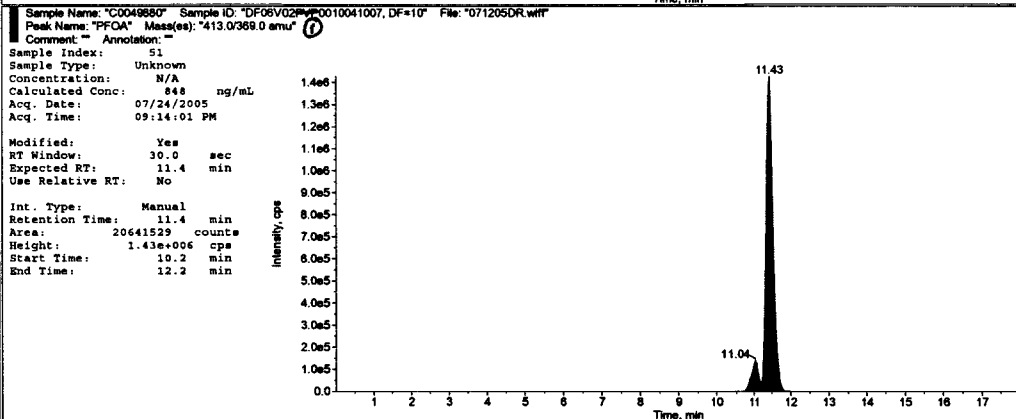
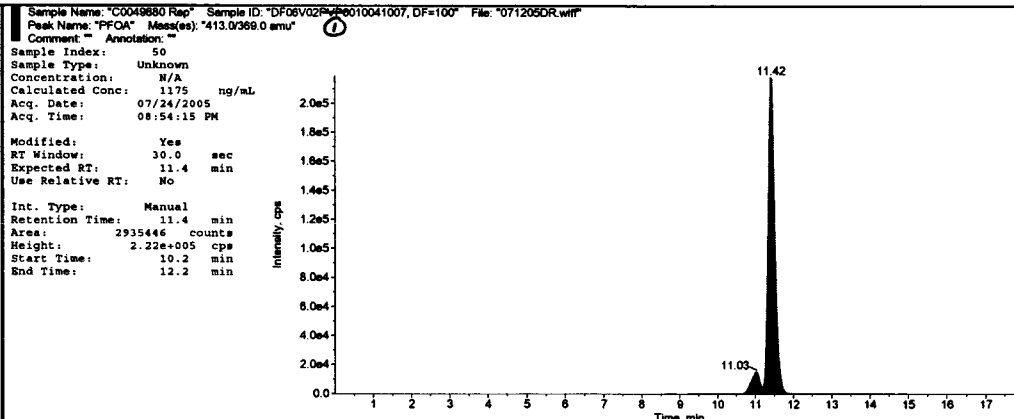
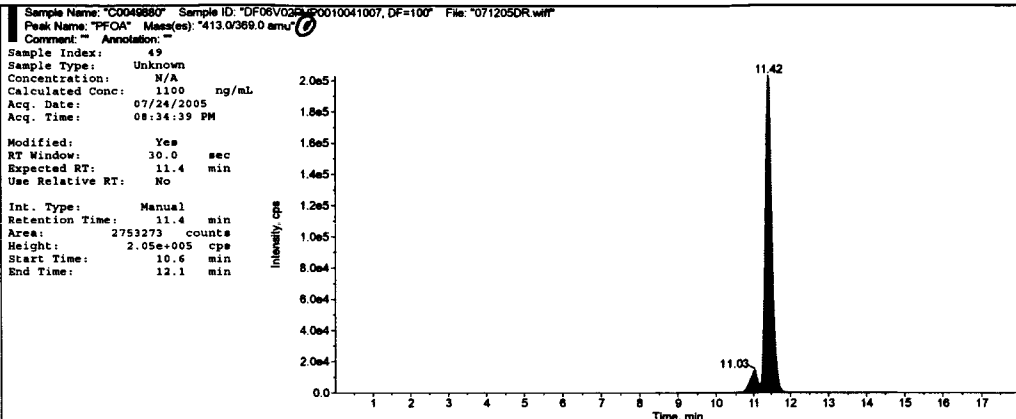


0294





0296
① (R) PAP MLC 9/21/05



0297
① RE PAP MLC 9/21/05

CEE 08/2/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
1 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5786	0.500	0.457	071205DR-101
2 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	9049	1.00	0.927	071205DR-102
3 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	22433	2.50	2.85	071205DR-103
4 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	47743	5.00	6.50	071205DR-104
5 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	82988	10.0	11.6	071205DR-105
6 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	189207	25.0	26.9	071205DR-106
7 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	363598	50.0	50.6	071205DR-107
8 Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	071205DR-108
9 C0049878 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	38544	* 25.0	518.	071205DR-109
10 C0049878 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	348878	25.0	496.	071205DR-110
11 C0049878 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	43203	* 250.	585.	071205DR-111
12 C0049878	DF06V01AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	34675	* N/A	462.	071205DR-112
13 C0049878 Rep	DF06V01AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	32294	* N/A	427.	071205DR-113
14 C0049878	DF06V01AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	321610	N/A	459.	071205DR-114
15 C0049878 Rep	DF06V01AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	289196	N/A	413.	071205DR-115
16 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5336	0.500	0.382	071205DR-116
17 C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=1000	Quality Control	1000	PFOA Confirm Ion	47822	* 25.0	6510.	071205DR-117
18 C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	373424	25.0	5340.	071205DR-118
19 C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	2471900	* 25.0	3560.	071205DR-119
20 C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=1000	Quality Control	1000	PFOA Confirm Ion	44545	* 250.	6040.	071205DR-120
21 C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	388797	250.	5560.	071205DR-121
22 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	8632	1.00	0.867	071205DR-122
23 C0049877	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOA Confirm Ion	54301	* N/A	7440.	071205DR-123
24 C0049877 Rep	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOA Confirm Ion	45887	* N/A	6230.	071205DR-124
25 C0049877	DF06V01PAP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	416026	N/A	5960.	071205DR-125
26 C0049877 Rep	DF06V01PAP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	401426	N/A	5740.	071205DR-126
27 C0049877	DF06V01PAP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	2668936	* N/A	3840.	071205DR-127
28 C0049877 Rep	DF06V01PAP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	2558259	* N/A	3680.	071205DR-128
29 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	19440	2.50	2.42	071205DR-129
30 C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	106039	25.0	1490.	071205DR-130
31 C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	974687	* 25.0	1400.	071205DR-131
32 C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	109450	250.	1540.	071205DR-132
33 C0049878	DF06V01RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	97681	N/A	1370.	071205DR-133
34 C0049878 Rep	DF06V01RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	107926	N/A	1520.	071205DR-134
35 C0049878	DF06V01RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	885379	* N/A	1270.	071205DR-135
36 C0049878 Rep	DF06V01RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	829218	* N/A	1330.	071205DR-136
37 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	37786	5.00	5.07	071205DR-137
38 C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	12691	* 25.0	145.	071205DR-138
39 C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	113265	25.0	159.	071205DR-139
40 C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	21866	* 250.	277.	071205DR-140
41 C0049879	DF06V02AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	12612	* N/A	144.	071205DR-141
42 C0049879 Rep	DF06V02AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	10362	* N/A	112.	071205DR-142
43 C0049879	DF06V02AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	109891	N/A	155.	071205DR-143
44 C0049879 Rep	DF06V02AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	82514	N/A	129.	071205DR-144
45 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	69858	10.0	9.69	071205DR-145
46 C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	155465	25.0	2200.	071205DR-146
47 C0049880 Spike K	DF06V02PVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	1346468	* 25.0	1940.	071205DR-147
48 C0049880 Spike L	DF06V02PVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	159517	250.	2260.	071205DR-148
49 C0049880	DF06V02PVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	140273	N/A	1980.	071205DR-149
50 C0049880 Rep	DF06V02PVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	148203	N/A	2070.	071205DR-150
51 C0049880	DF06V02PVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	1234391	* N/A	1770.	071205DR-151
52 C0049880 Rep	DF06V02PVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	1245540	* N/A	1790.	071205DR-152
53 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	168147	25.0	23.8	071205DR-153
54 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	321778	50.0	46.0	071205DR-154

* Analysis not required. BAK 08/02/05

① (P) PAP MLC 9/2/05

Vegetation Conversion

Compound: PFOA Confirm. Ion

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049876	DF06-V01-AVP001-0-041007	459	184
C0049876 Rep	DF06-V01-AVP001-0-041007	413	165
C0049876 Spk C	DF06-V01-AVP001-0-041007	496	198
C0049877	DF06-V01-PAP001-0-041007	5950	2380
C0049877 Rep	DF06-V01-PAP001-0-041007	5740	2296
C0049877 Spk E	DF06-V01-PAP001-0-041007	5340	2136
C0049877 Spk F	DF06-V01-PAP001-0-041007	5560	2224
C0049878	DF06-V01-RCP001-0-041007	1370	548
C0049878 Rep	DF06-V01-RCP001-0-041007	1520	608
C0049878 Spk G	DF06-V01-RCP001-0-041007	1490	596
C0049878 Spk H	DF06-V01-RCP001-0-041007	1540	616
C0049879	DF06-V02-AVP001-0-041007	155	62.0
C0049879 Rep	DF06-V02-AVP001-0-041007	129	51.6
C0049879 Spk I	DF06-V02-AVP001-0-041007	159	63.6
C0049880	DF06-V02-PVP001-0-041007 ^②	1980	792
C0049880 Rep	DF06-V02-PVP001-0-041007 ^②	2070	828
C0049880 Spk K	DF06-V02-PVP001-0-041007 ^②	2200	880
C0049880 Spk L	DF06-V02-PVP001-0-041007 ^②	2260	904

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = Peak Area AT OR ABOVE 0.2 ng/g ^①

NQ = Not quantifiable = Measured concentration below Limit of Quantitation (LOQ) which is 0.8 ng/g.
^① BETWEEN 0.2 ng/g AND ^②

① RE MLC 9/20/05

② RE PAP MLC 9/21/05

0299
 BML 09/03/05

	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound:	PFOA Confirm. Ion	Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049876	DF06-V01-AVP001-0-041007	=Raw Data!H253	=(C9*2)/5
10	C0049876 Rep	DF06-V01-AVP001-0-041007	=Raw Data!H254	=(C10*2)/5
11	C0049876 Spk C	DF06-V01-AVP001-0-041007	=Raw Data!H249	=(C11*2)/5
12				
13	C0049877	DF06-V01-PAP001-0-041007	=Raw Data!H264	=(C13*2)/5
14	C0049877 Rep	DF06-V01-PAP001-0-041007	=Raw Data!H265	=(C14*2)/5
15	C0049877 Spk E	DF06-V01-PAP001-0-041007	=Raw Data!H257	=(C15*2)/5
16	C0049877 Spk F	DF06-V01-PAP001-0-041007	=Raw Data!H260	=(C16*2)/5
17				
18	C0049878	DF06-V01-RCP001-0-041007	=Raw Data!H272	=(C18*2)/5
19	C0049878 Rep	DF06-V01-RCP001-0-041007	=Raw Data!H273	=(C19*2)/5
20	C0049878 Spk G	DF06-V01-RCP001-0-041007	=Raw Data!H269	=(C20*2)/5
21	C0049878 Spk H	DF06-V01-RCP001-0-041007	=Raw Data!H271	=(C21*2)/5
22				
23	C0049879	DF06-V02-AVP001-0-041007	=Raw Data!H282	=(C23*2)/5
24	C0049879 Rep	DF06-V02-AVP001-0-041007	=Raw Data!H283	=(C24*2)/5
25	C0049879 Spk I	DF06-V02-AVP001-0-041007	=Raw Data!H278	=(C25*2)/5
26				
27	C0049880	DF06-V02-PVP001-0-041007 (D)	=Raw Data!H288	=(C27*2)/5
28	C0049880 Rep	DF06-V02-PVP001-0-041007 (D)	=Raw Data!H289	=(C28*2)/5
29	C0049880 Spk K	DF06-V02-PVP001-0-041007 (D)	=Raw Data!H285	=(C29*2)/5
30	C0049880 Spk L	DF06-V02-PVP001-0-041007 (D)	=Raw Data!H287	=(C30*2)/5
31				

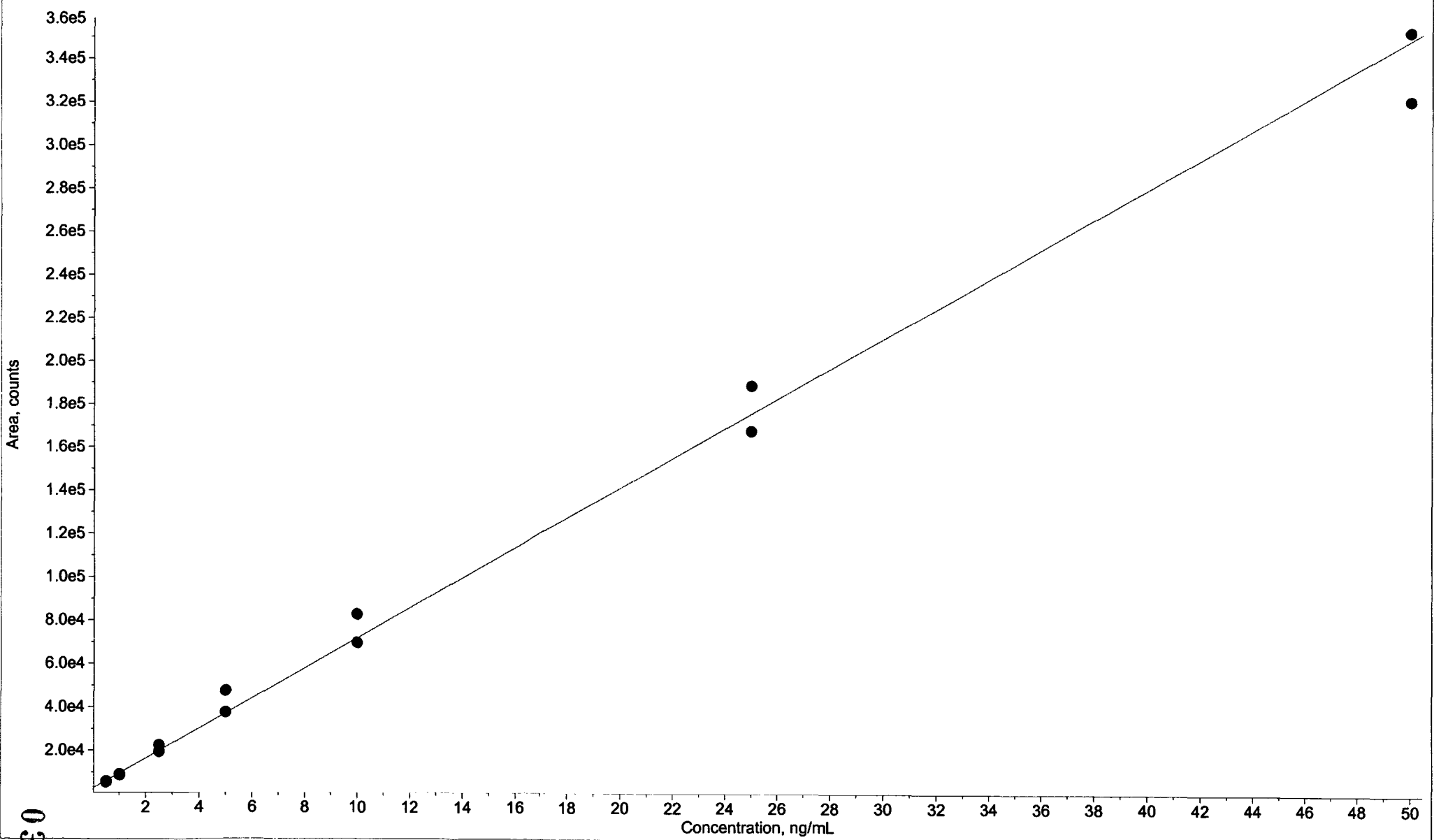
(D) RE PAP MLC 9/21/05

0300

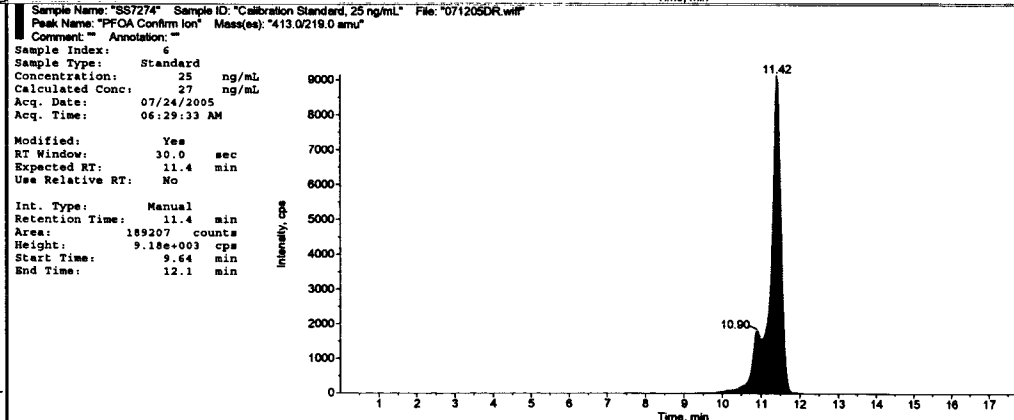
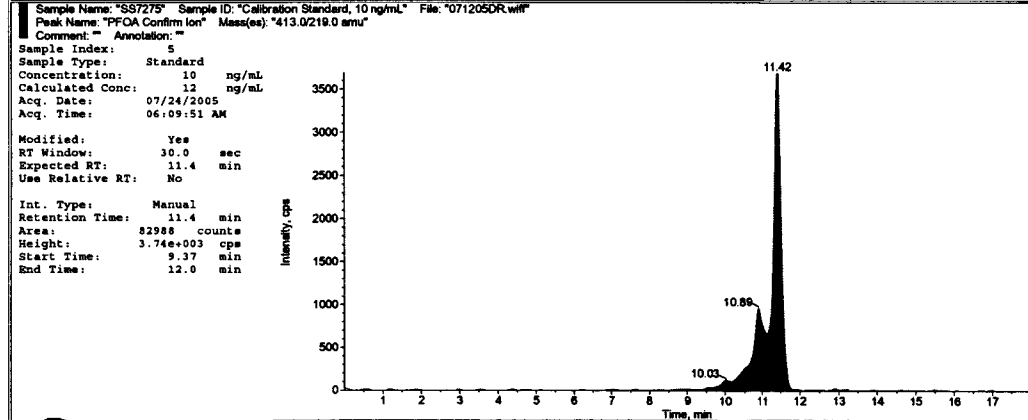
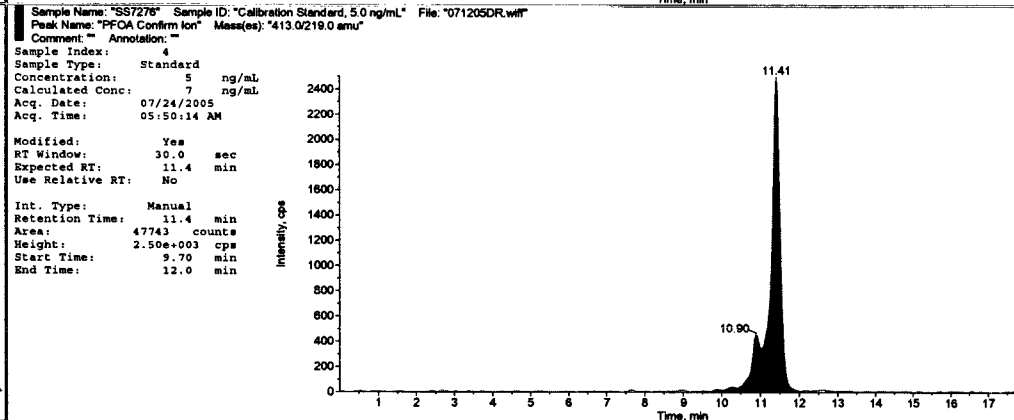
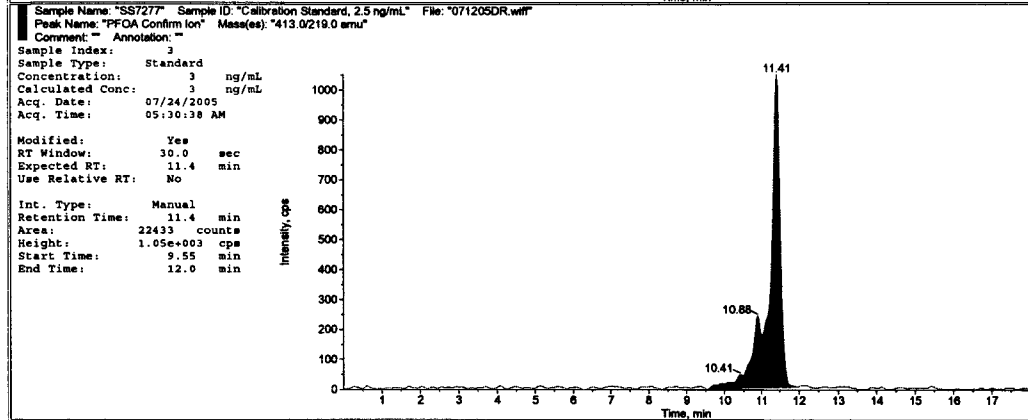
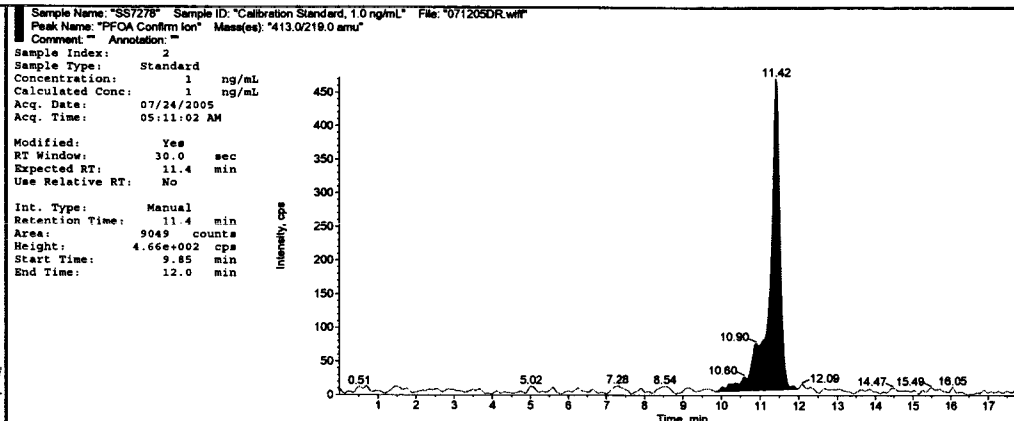
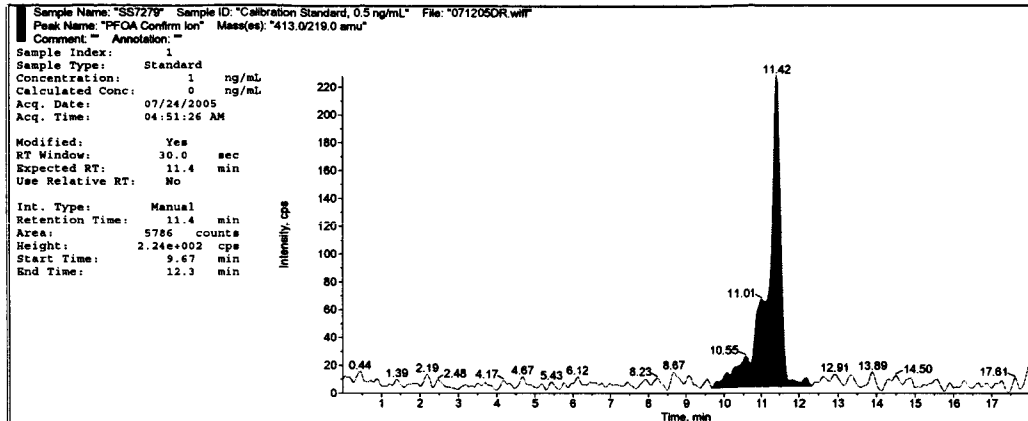
BAK 08/03/05

CEE 07/26/05

071205DR VEG.rdb (PFOA Confirm Ion): "Linear" Regression ("1 / x" weighting): $y = 6.94e+003 x + 2.62e+003$ ($r = 0.9959$)



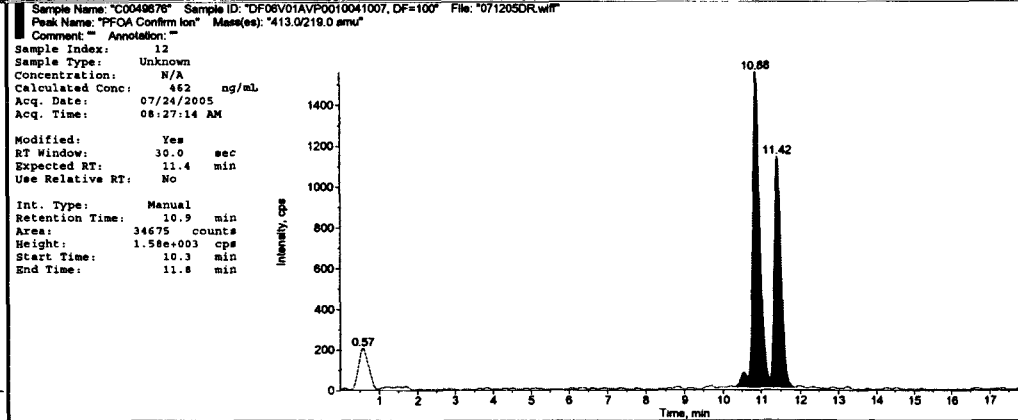
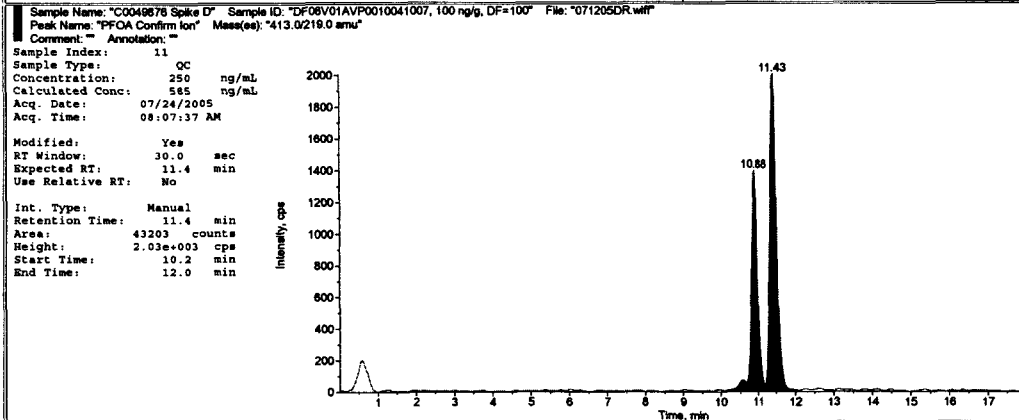
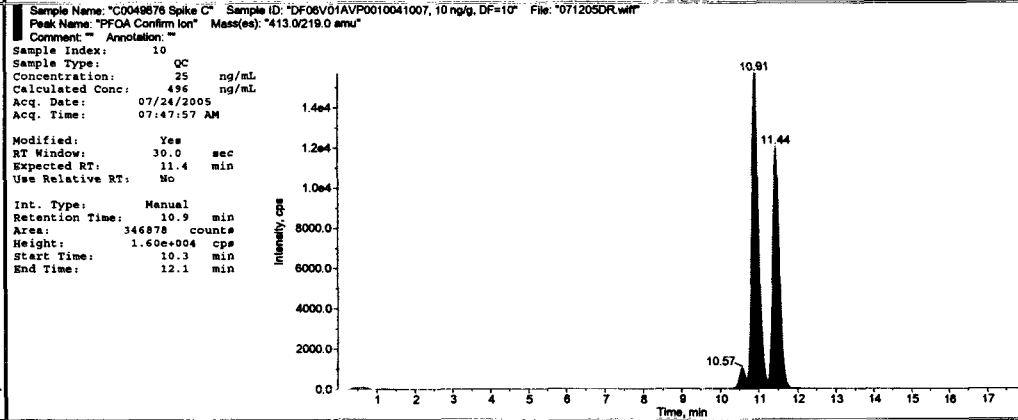
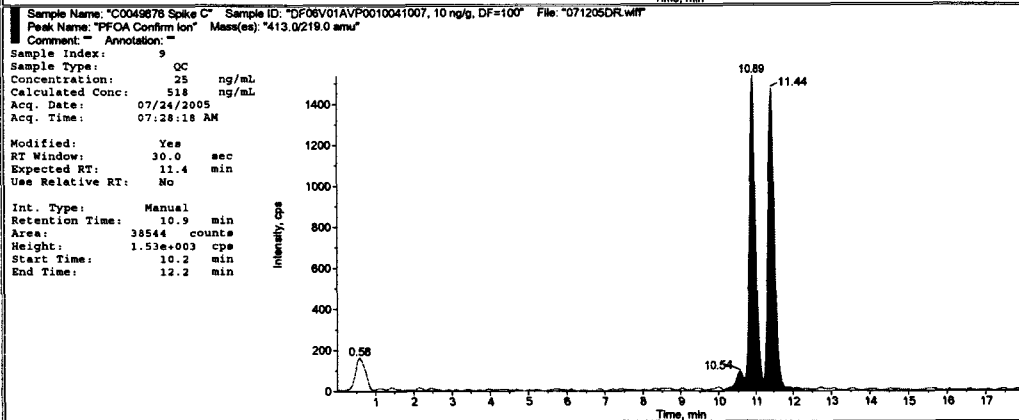
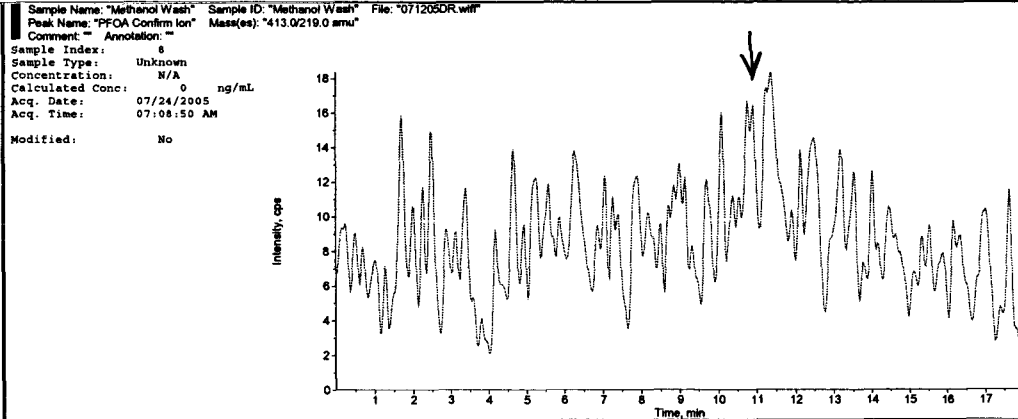
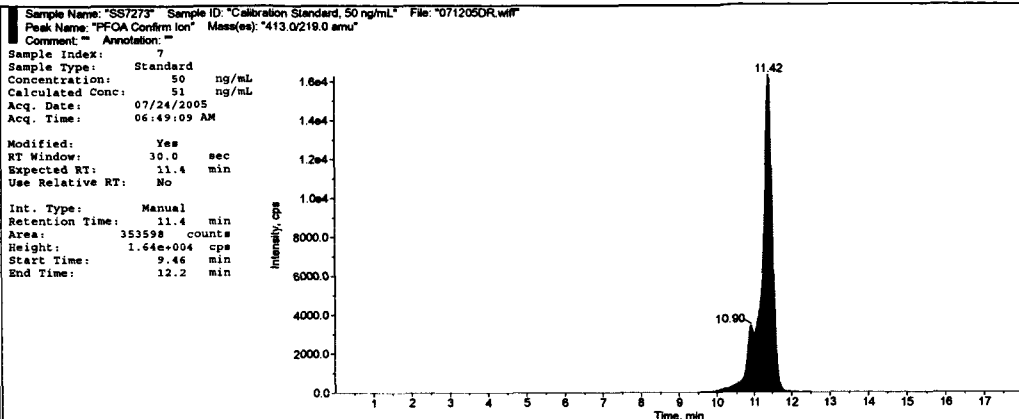
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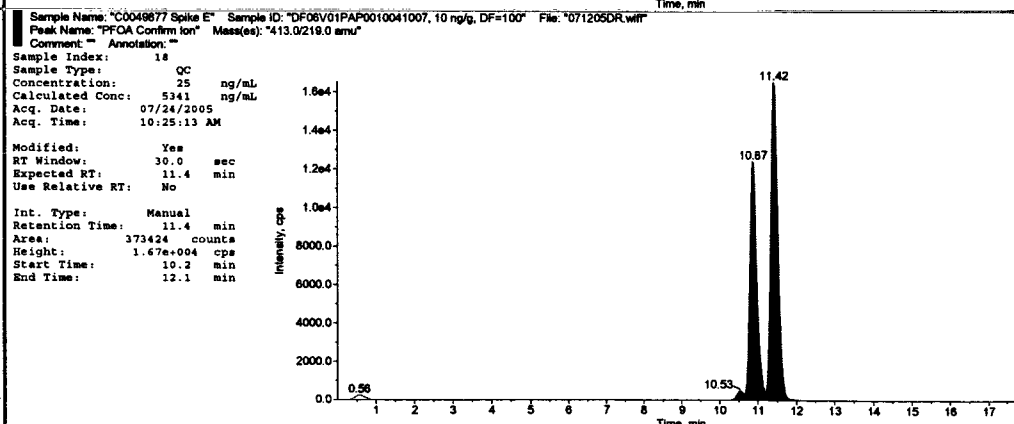
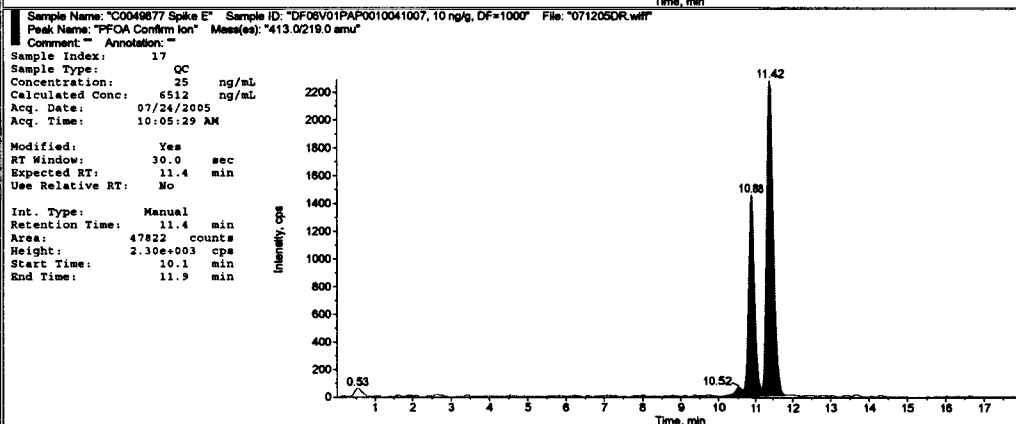
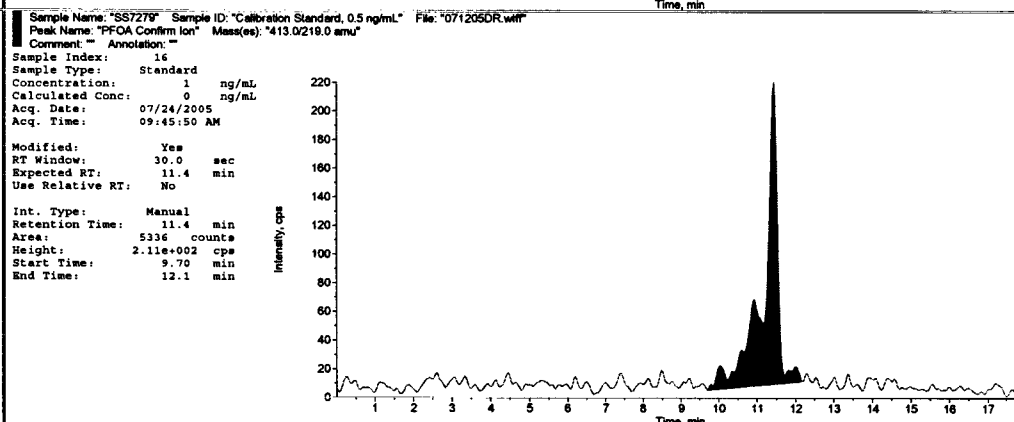
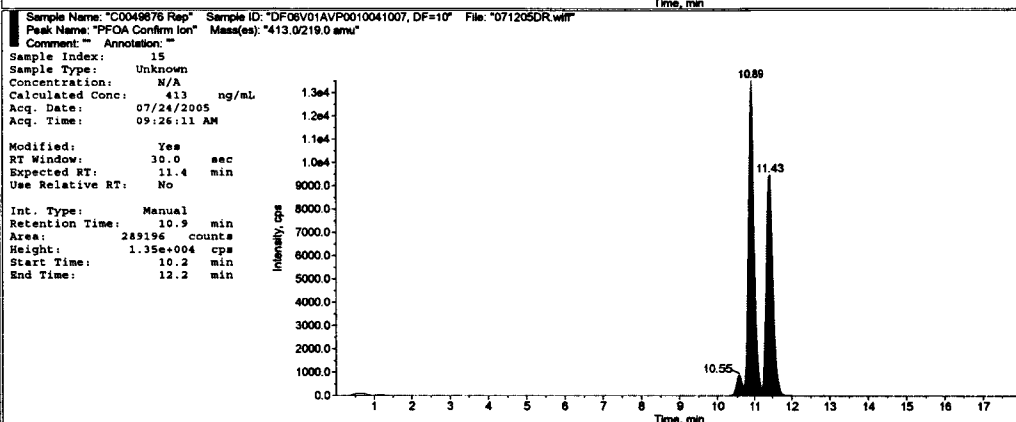
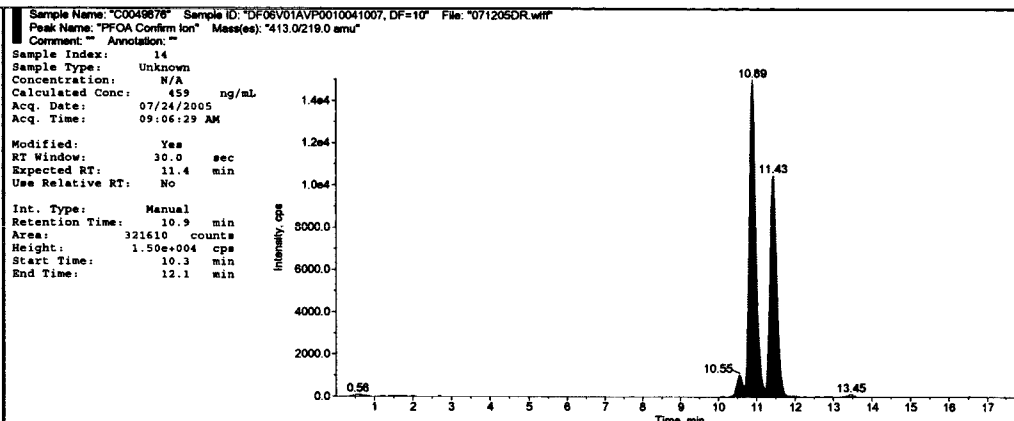
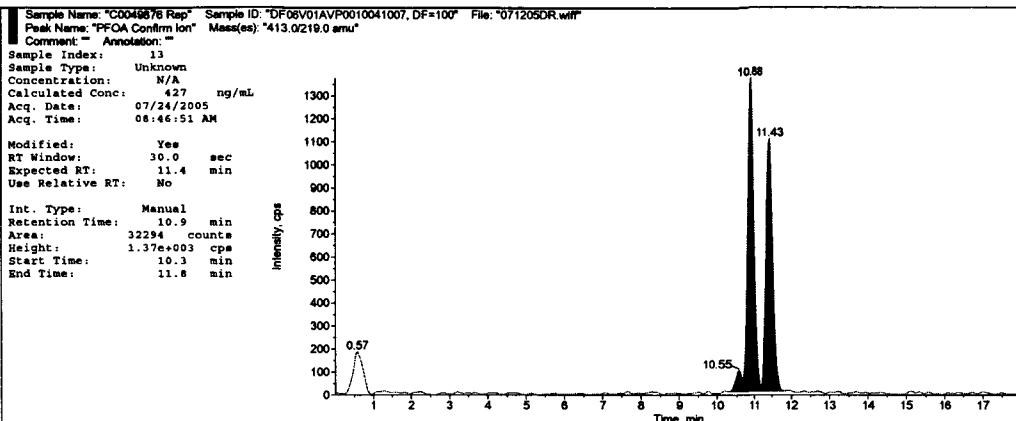


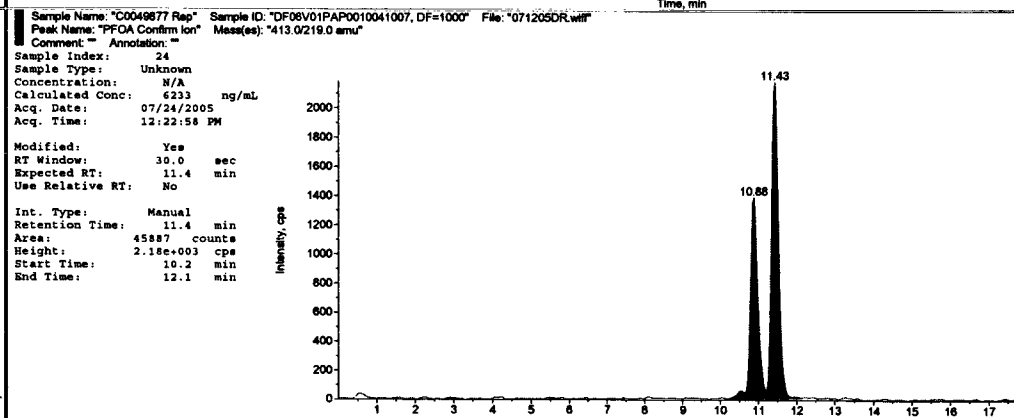
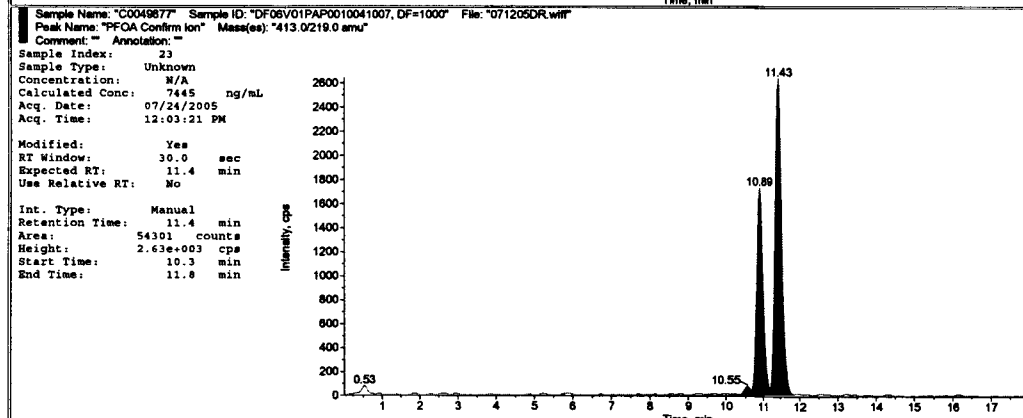
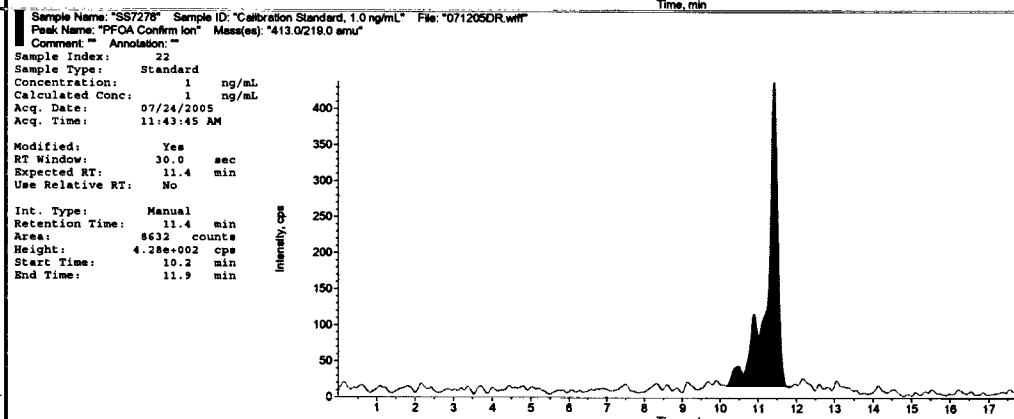
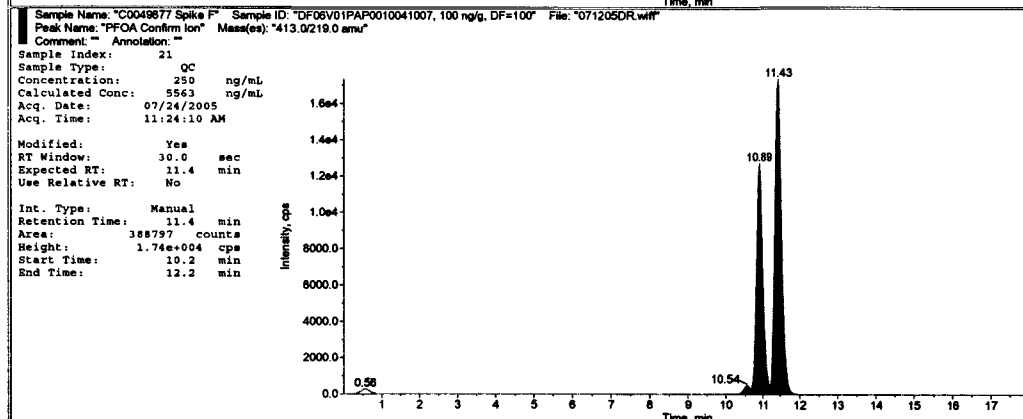
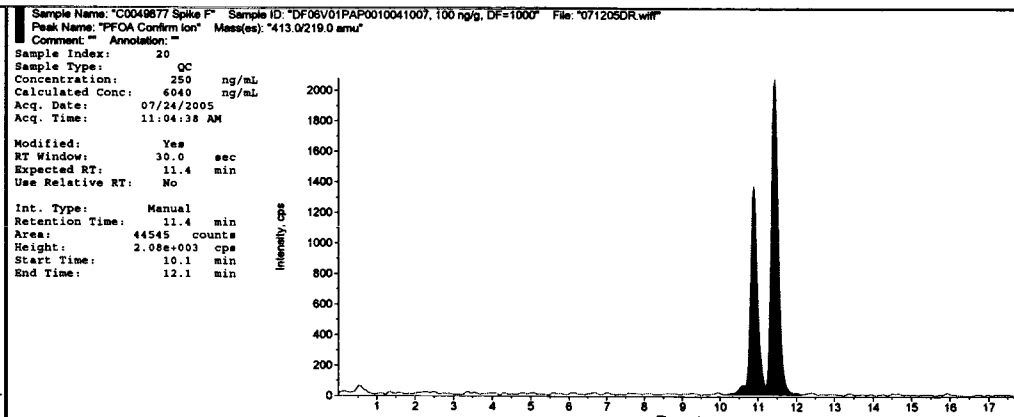
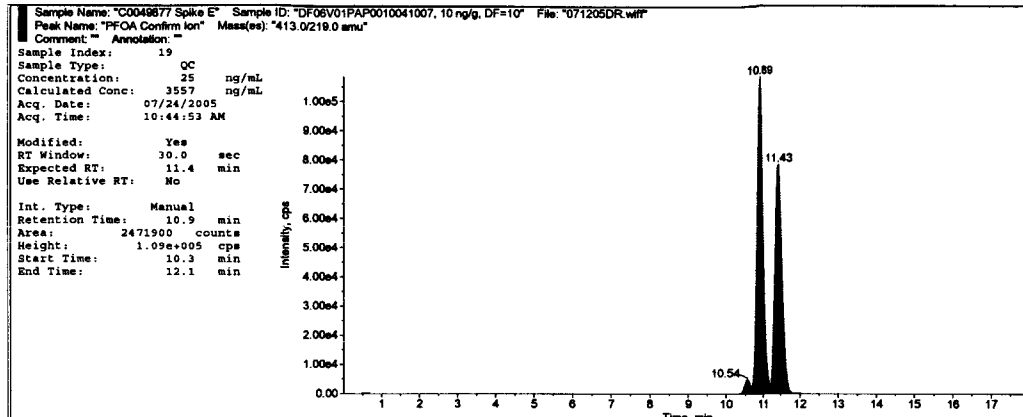
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Sample Run # 1 To 54
Index CEE 07/26/05

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Sample Index: 1 To: 54

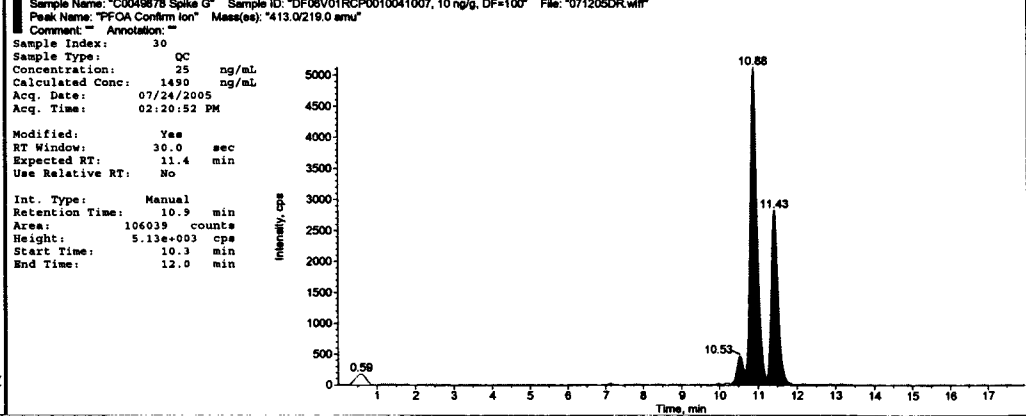
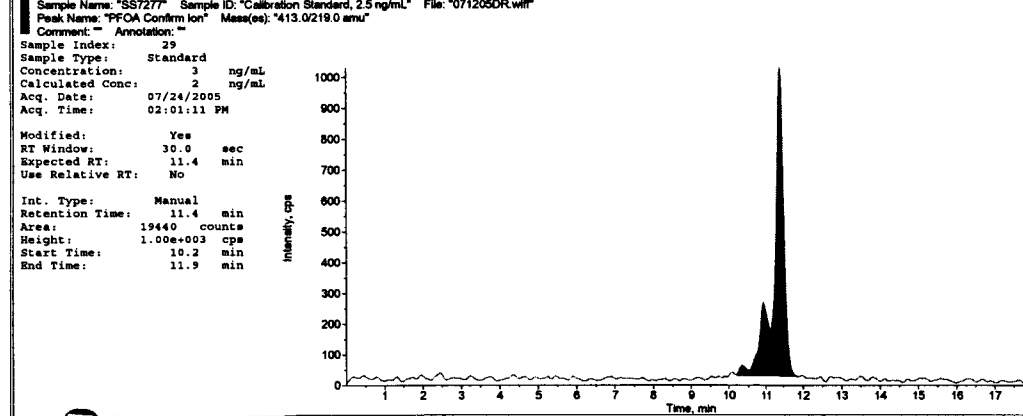
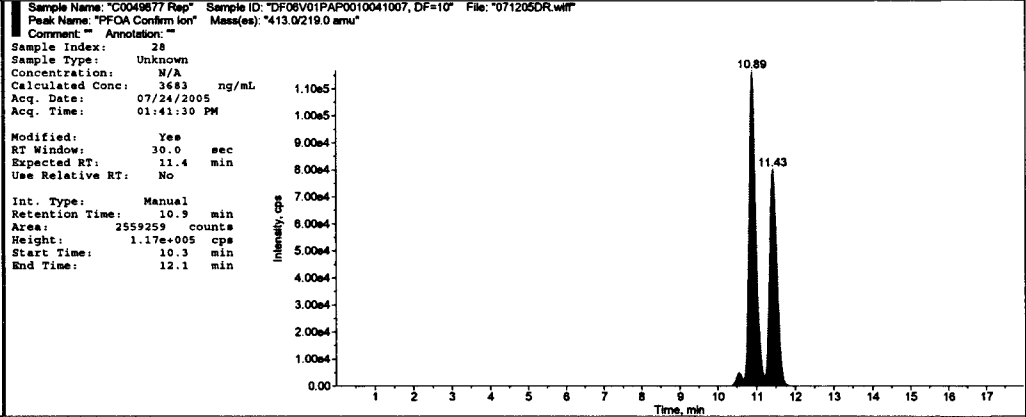
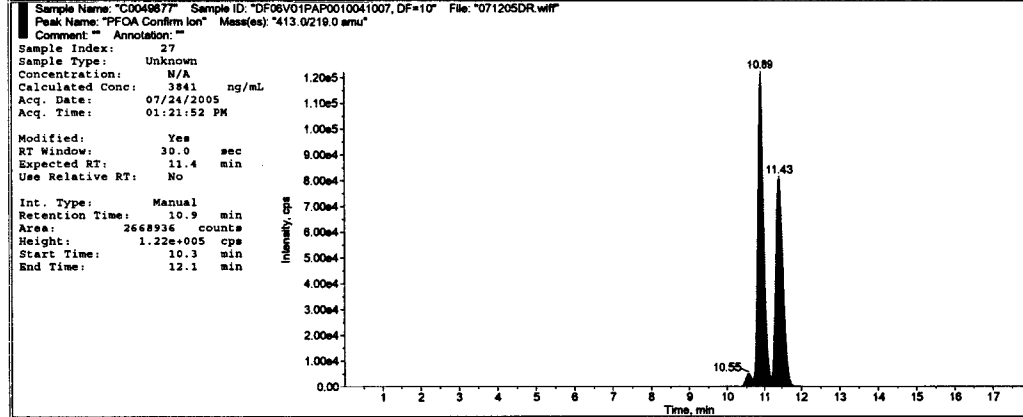
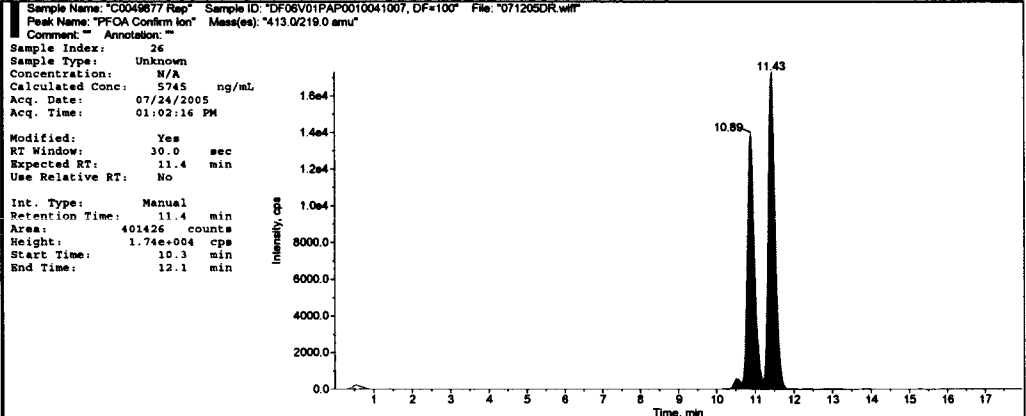
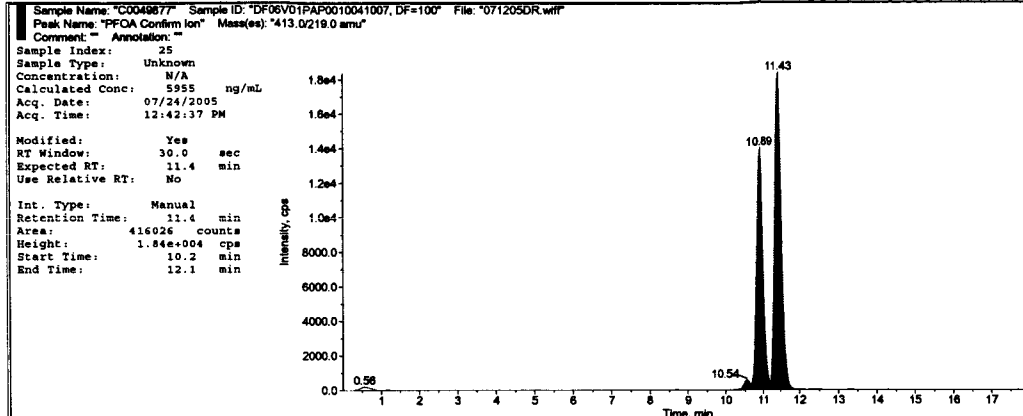
0302



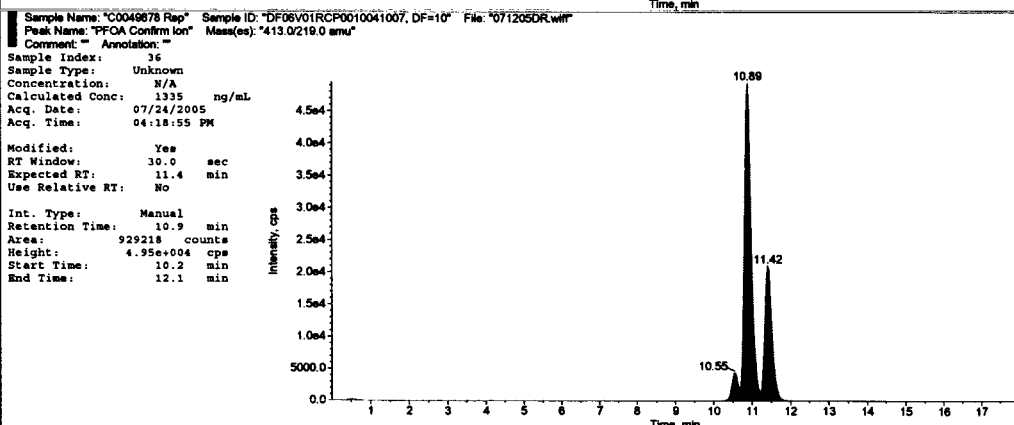
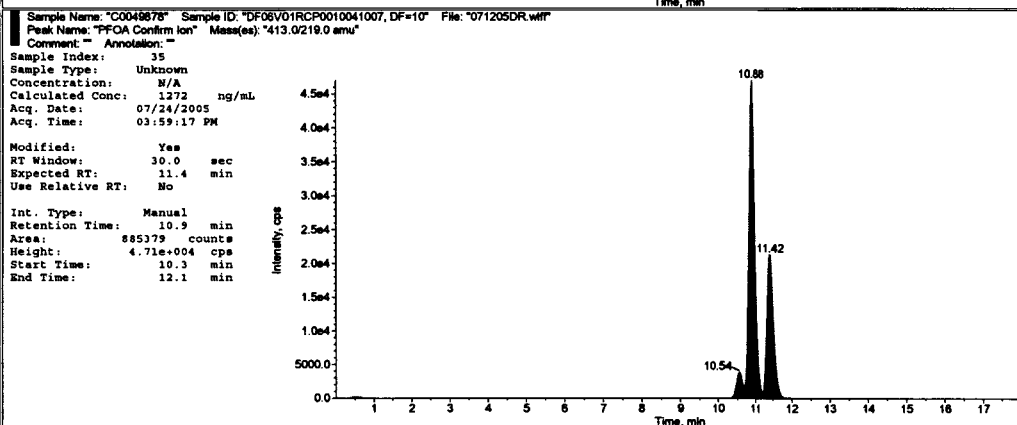
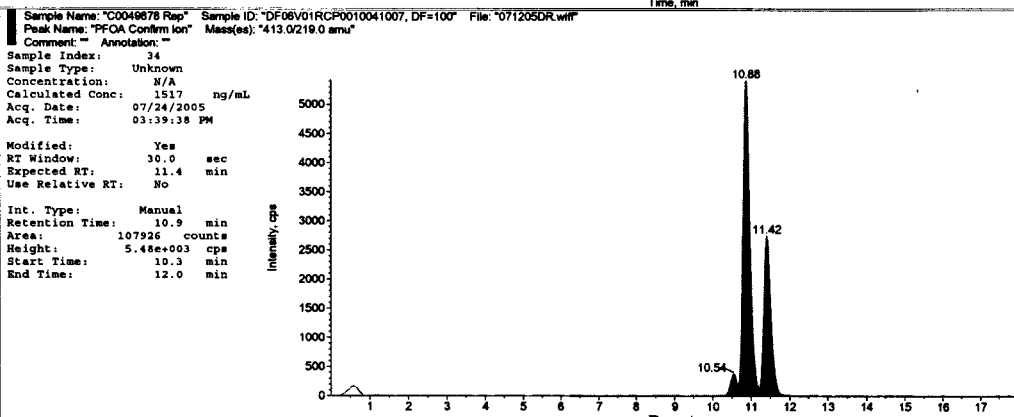
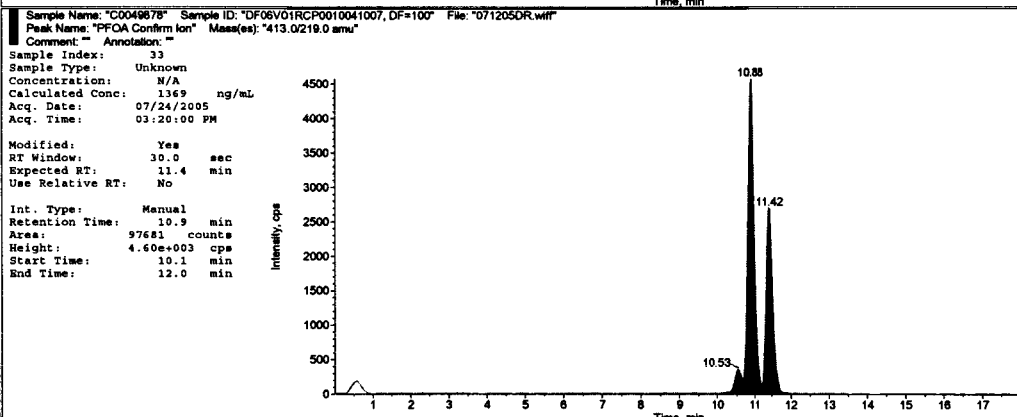
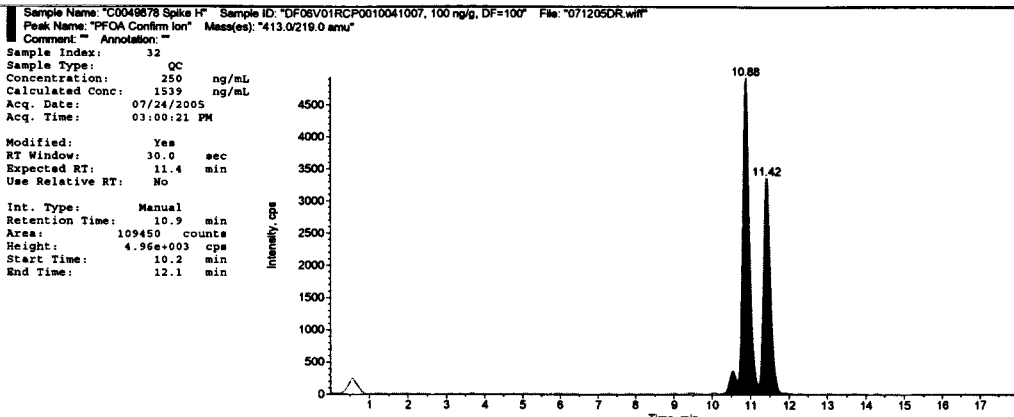
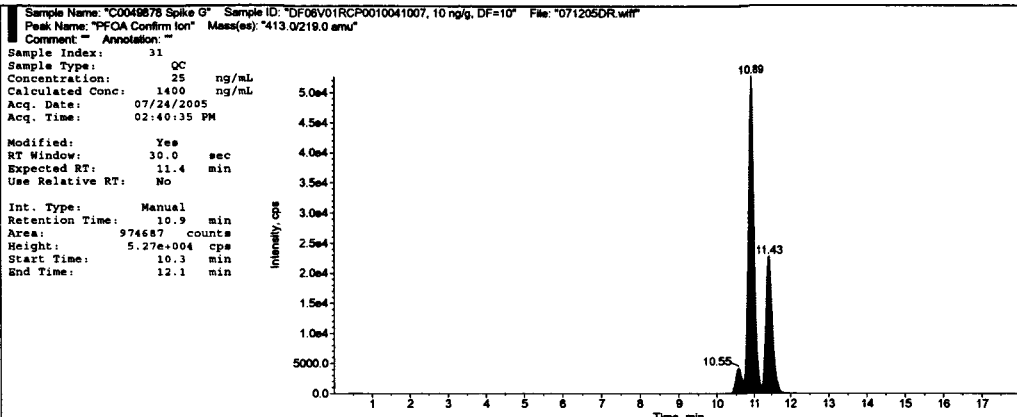




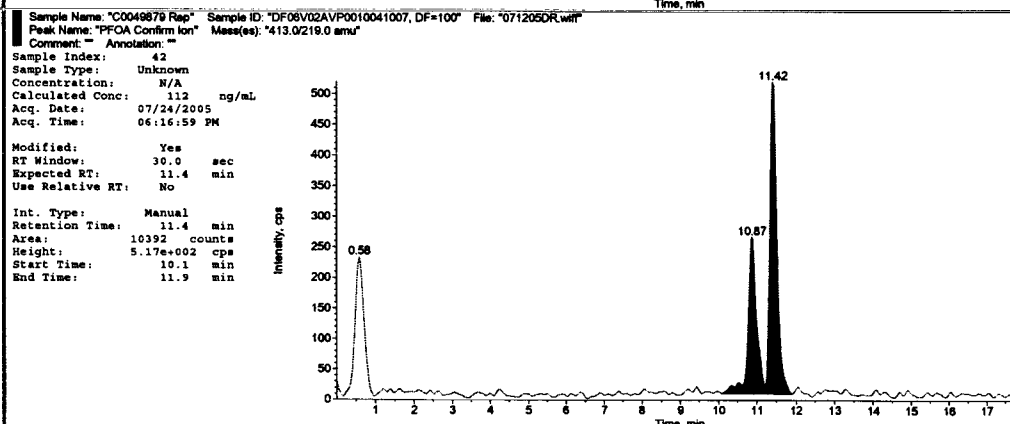
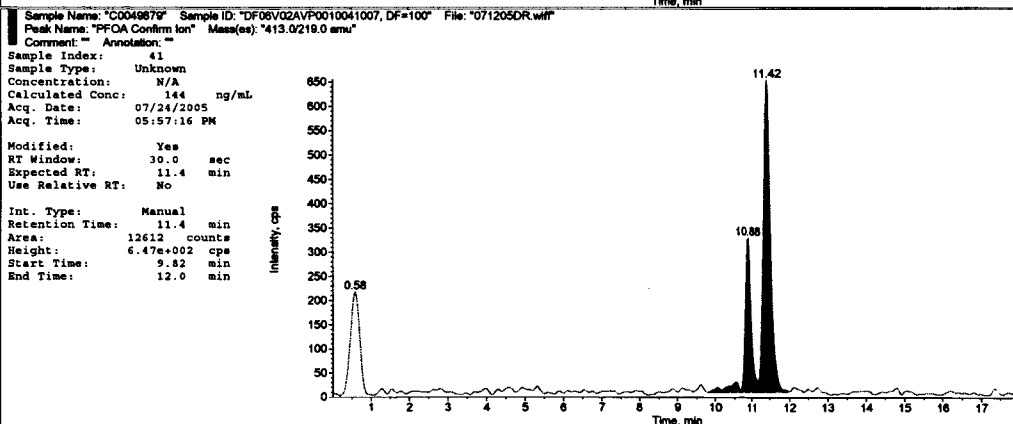
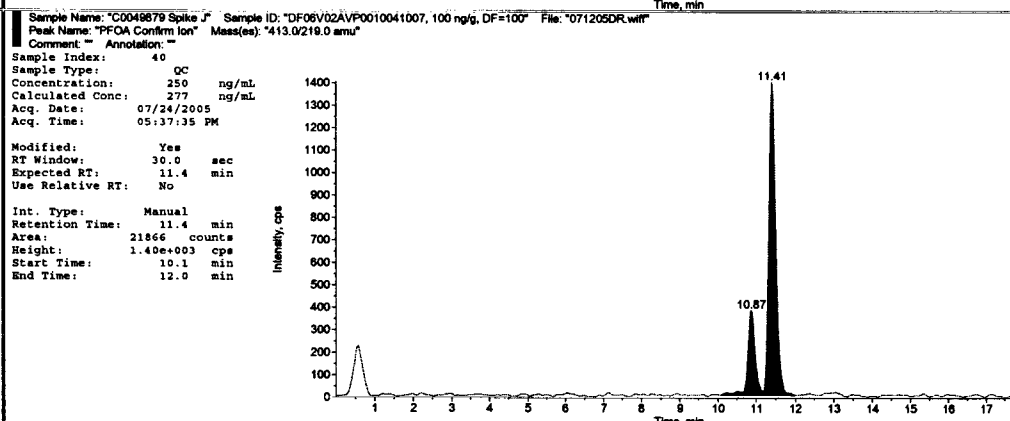
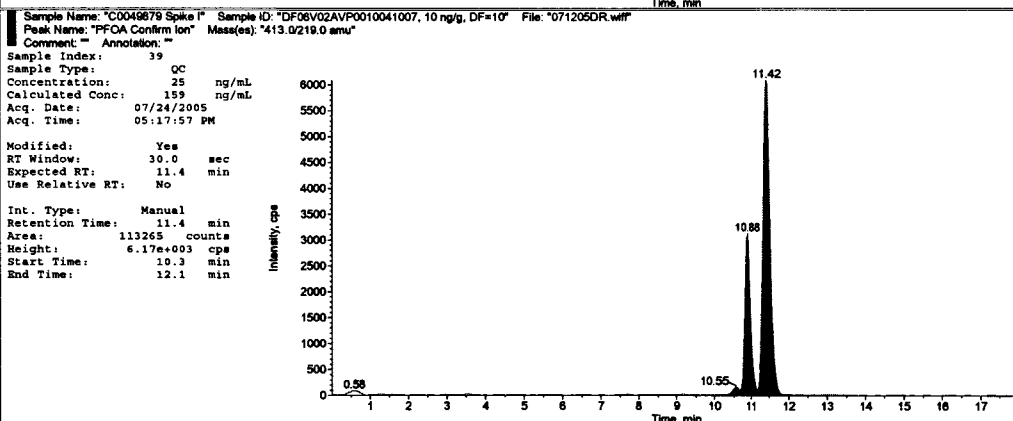
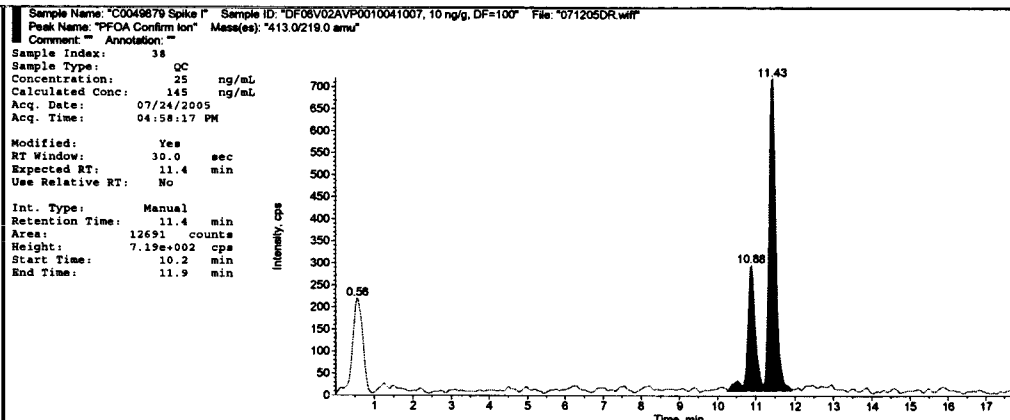
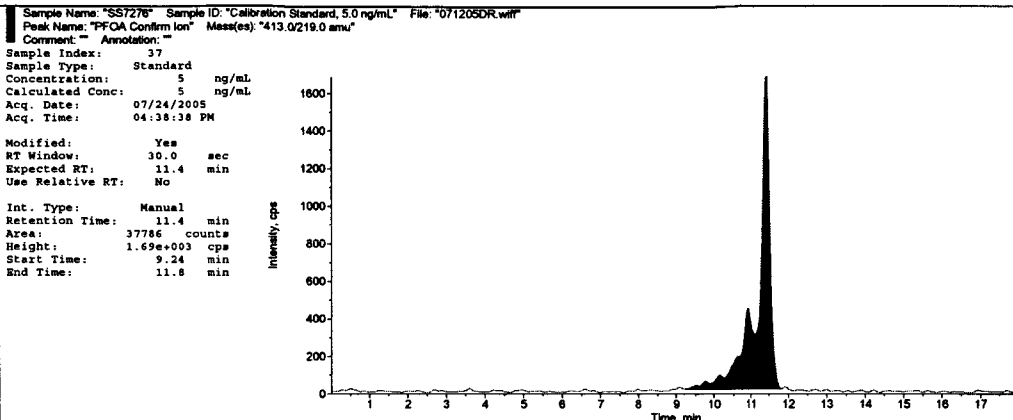
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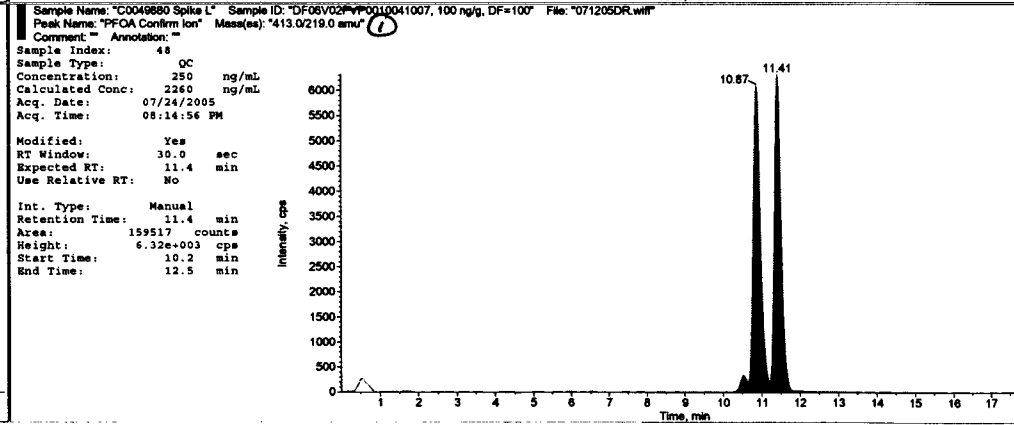
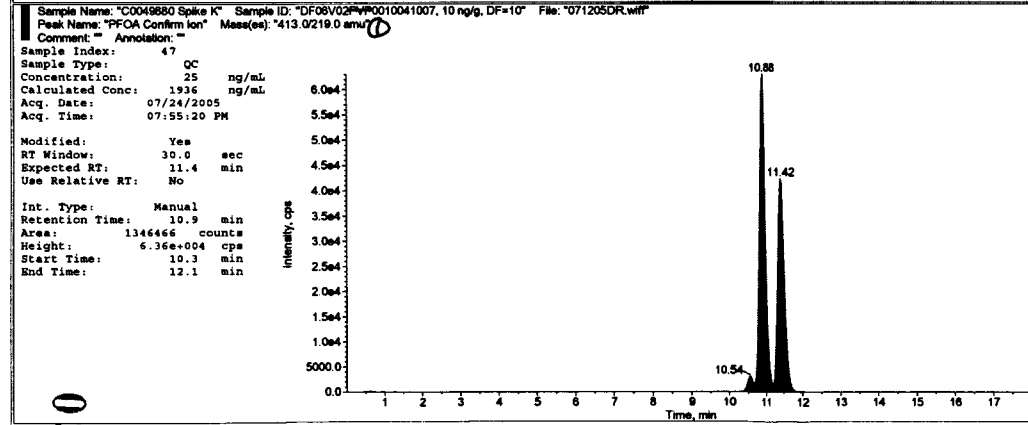
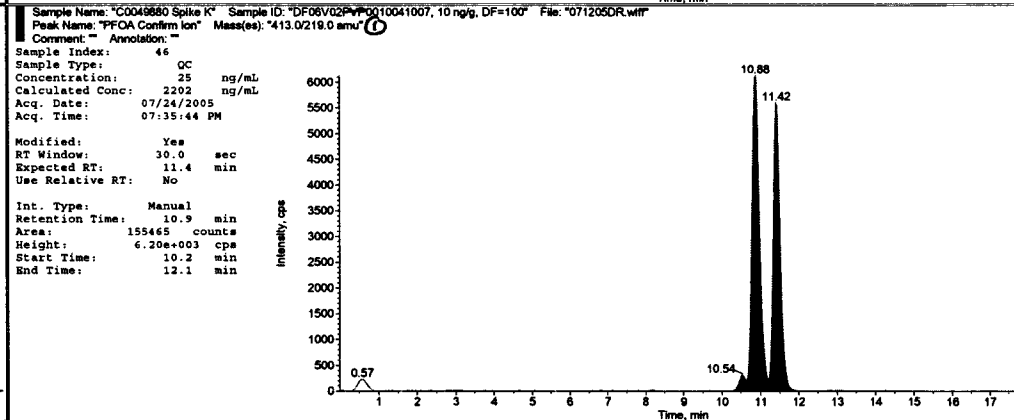
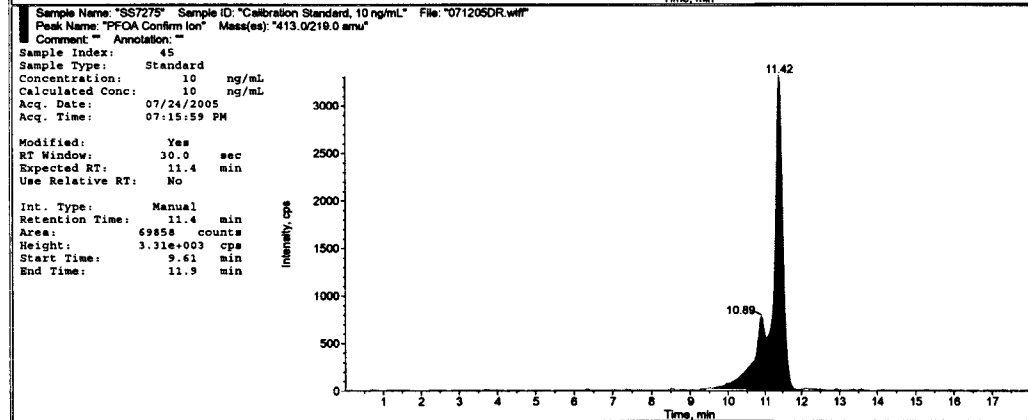
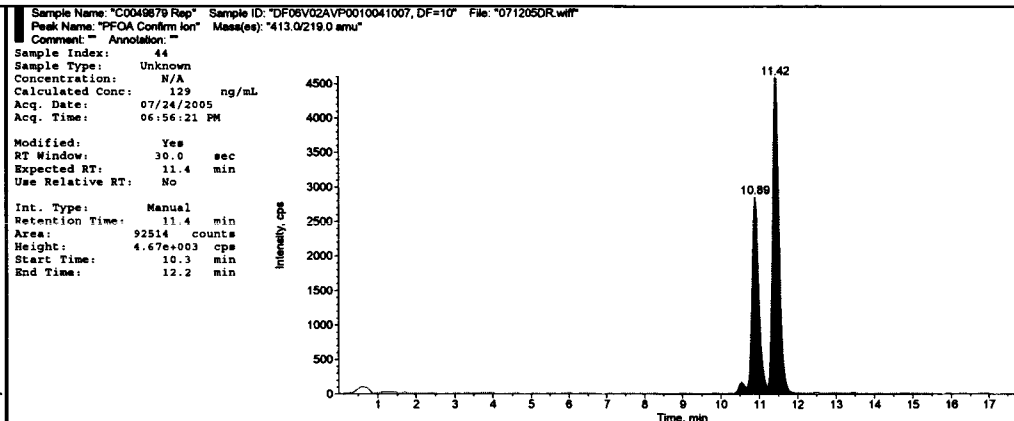
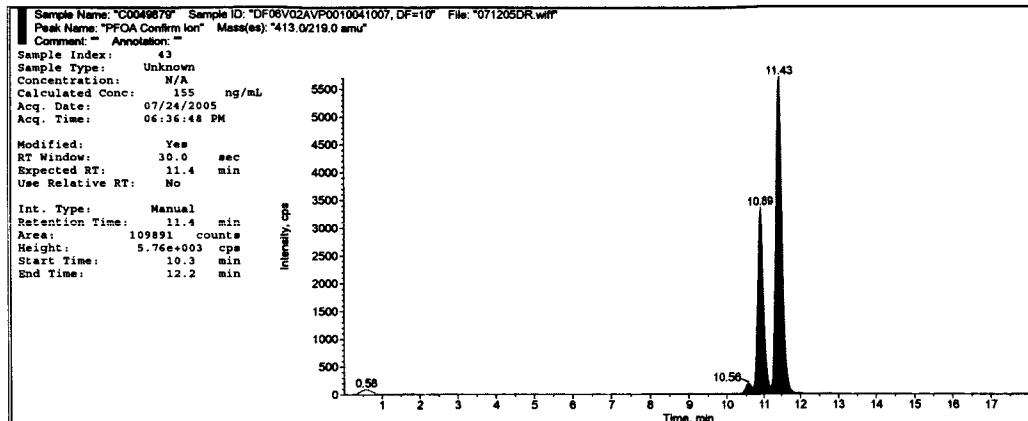


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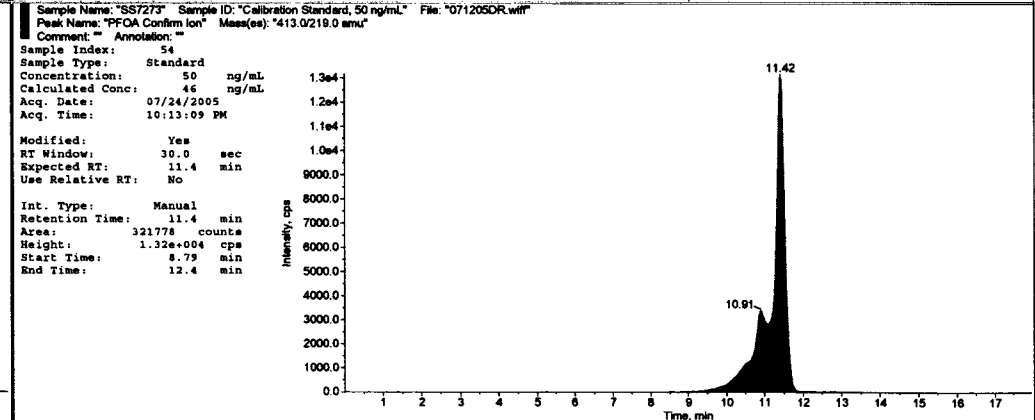
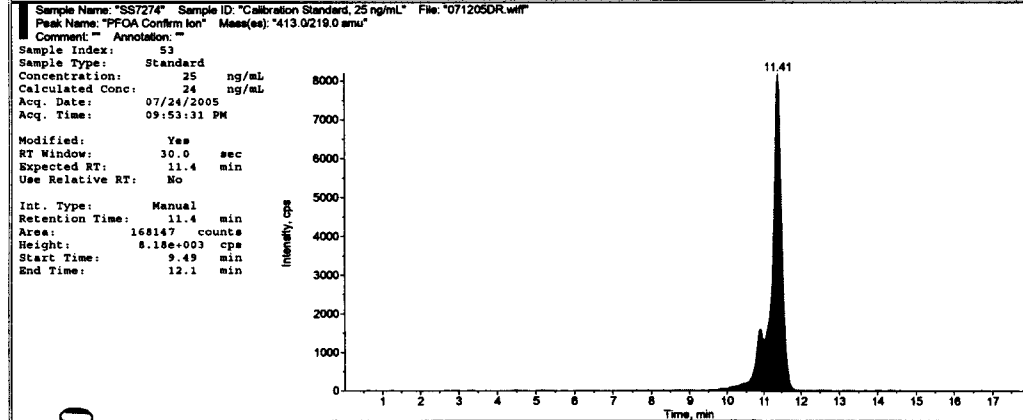
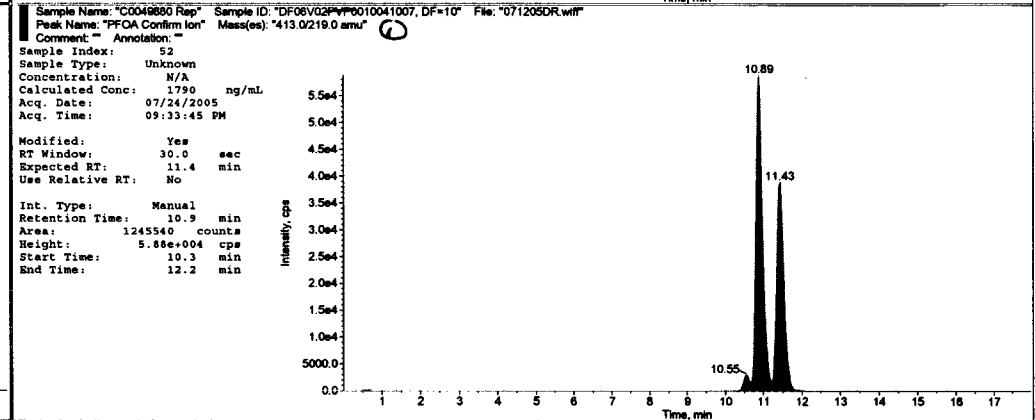
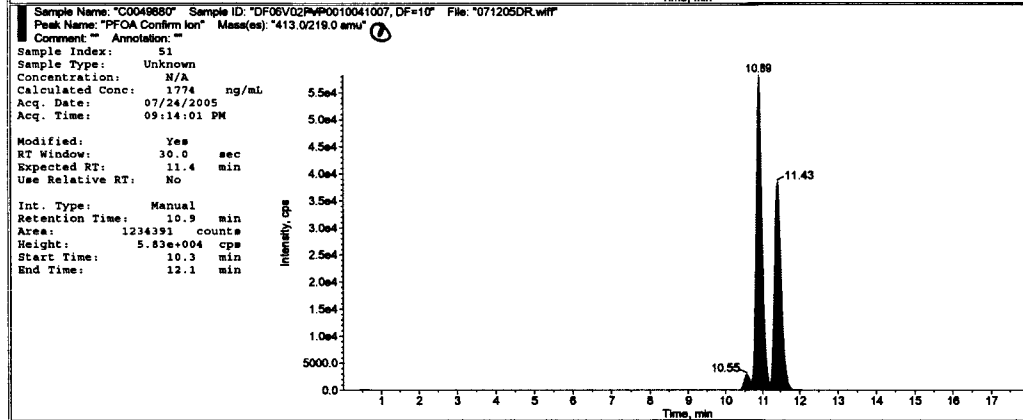
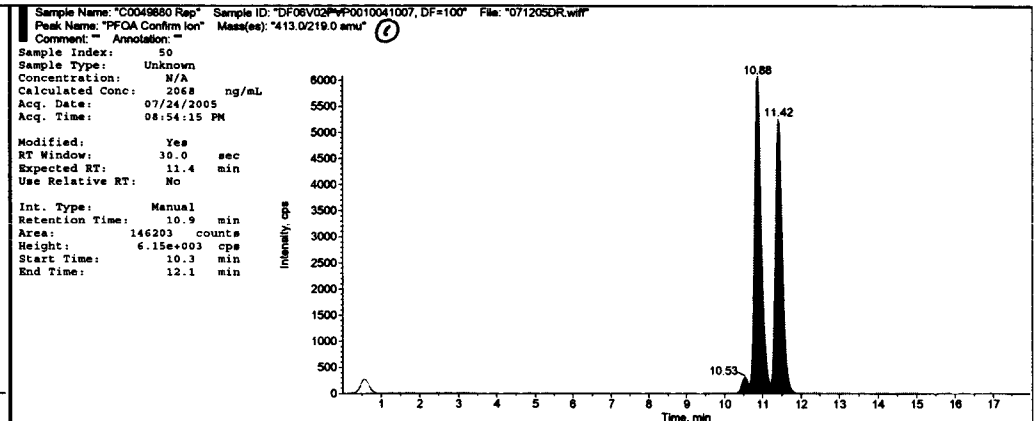
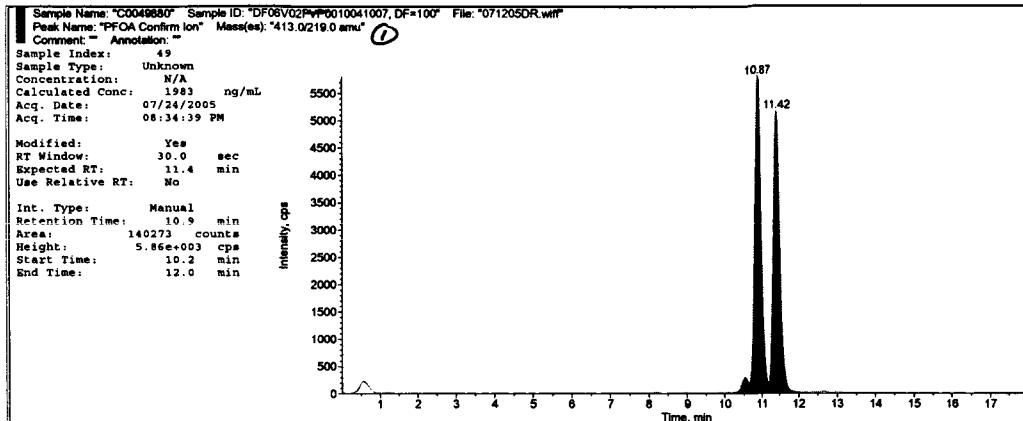


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0309
① (RE) PAP MLC 9/21/05



03
PAP MLC 9/2/05

A	B	C	D	E	F	G	H	I	J
PFBS									
Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Peak Area	Conc. Concentration (ng/L)	Calculated Concentration (ng/L)		Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFBS	16142	0.50	0.444		71205DR-101
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFBS	31198	1.00	0.978		71205DR-102
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFBS	79565	2.50	2.69		71205DR-103
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFBS	163696	5.00	5.68		71205DR-104
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFBS	285077	10.00	9.98		71205DR-105
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFBS	741489	25.00	26.2		71205DR-106
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFBS	1399742	50.00	49.5		71205DR-107
Methanol Wash	Methanol Wash	Unknown	1	PFBS	0	N/A	No Peak		71205DR-108
C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFBS	17141	25.00	47.9		71205DR-109
C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFBS	148908	25.00	51.5		71205DR-110
C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFBS	57626	250.00	192		71205DR-111
C0049876	DF06V01AVP0010041007, DF=100	Unknown	100	PFBS	10732	N/A	25.2		71205DR-112
C0049876 Rep	DF06V01AVP0010041007, DF=100	Unknown	100	PFBS	10722	N/A	25.2		71205DR-113
C0049876	DF06V01AVP0010041007, DF=10	Unknown	10	PFBS	91891	N/A	31.3		71205DR-114
C0049876 Rep	DF06V01AVP0010041007, DF=10	Unknown	10	PFBS	82469	N/A	28		71205DR-115
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFBS	15913	0.50	0.436		71205DR-116
C0049877 Spike E	DF06V01AP0010041007, 10 ng/g, DF=1000	Quality Control	1000	PFBS	5469	25.00	65.3		71205DR-117
C0049877 Spike E	DF06V01AP0010041007, 10 ng/g, DF=100	Quality Control	100	PFBS	30975	25.00	97		71205DR-118
C0049877 Spike E	DF06V01AP0010041007, 10 ng/g, DF=10	Quality Control	10	PFBS	264095	25.00	92.4		71205DR-119
C0049877 Spike F	DF06V01AP0010041007, 100 ng/g, DF=1000	Quality Control	1000	PFBS	11012	250.00	262		71205DR-120
C0049877 Spike F	DF06V01AP0010041007, 100 ng/g, DF=100	Quality Control	100	PFBS	80927	250.00	274		71205DR-121
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFBS	31459	1.00	0.987		71205DR-122
C0049877	DF06V01AP0010041007, DF=1000	Unknown	1000	PFBS	6404	N/A	98.5		71205DR-123
C0049877 Rep	DF06V01AP0010041007, DF=1000	Unknown	1000	PFBS	6097	N/A	87.6		71205DR-124
C0049877	DF06V01AP0010041007, DF=100	Unknown	100	PFBS	29069	N/A	90.2		71205DR-125
C0049877 Rep	DF06V01AP0010041007, DF=100	Unknown	100	PFBS	26267	N/A	80.3		71205DR-126
C0049877	DF06V01AP0010041007, DF=10	Unknown	10	PFBS	233561	N/A	81.5		71205DR-127
C0049877 Rep	DF06V01AP0010041007, DF=10	Unknown	10	PFBS	224709	N/A	78.4		71205DR-128
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.00E+00	PFBS	73556	2.50	2.48		71205DR-129
C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=100	Quality Control	1.00E+02	PFBS	11155	25.00	26.7		71205DR-130
C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=10	Quality Control	1.00E+01	PFBS	97516	25.00	33.3		71205DR-131
C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=100	Quality Control	1.00E+02	PFBS	61208	250.00	204		71205DR-132
C0049878	DF06V01RCP0010041007, DF=100	Unknown	1.00E+02	PFBS	5184	N/A	5.52		71205DR-133
C0049878 Rep	DF06V01RCP0010041007, DF=100	Unknown	1.00E+02	PFBS	5725	N/A	7.44		71205DR-134
C0049878	DF06V01RCP0010041007, DF=10	Unknown	1.00E+01	PFBS	35934	N/A	11.5		71205DR-135
C0049878 Rep	DF06V01RCP0010041007, DF=10	Unknown	1.00E+01	PFBS	36314	N/A	11.6		71205DR-136
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.00E+00	PFBS	156341	5.00	5.42		71205DR-137
C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=100	Quality Control	1.00E+02	PFBS	16390	25.00	45.3		71205DR-138
C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=10	Quality Control	1.00E+01	PFBS	131061	25.00	45.2		71205DR-139
C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=100	Quality Control	1.00E+02	PFBS	57090	250.00	190		71205DR-140
C0049879	DF06V02AVP0010041007, DF=100	Unknown	1.00E+02	PFBS	11771	N/A	28.9		71205DR-141
C0049879 Rep	DF06V02AVP0010041007, DF=100	Unknown	1.00E+02	PFBS	10226	N/A	23.4		71205DR-142
C0049879	DF06V02AVP0010041007, DF=10	Unknown	1.00E+01	PFBS	92705	N/A	31.6		71205DR-143
C0049879 Rep	DF06V02AVP0010041007, DF=10	Unknown	1.00E+01	PFBS	78045	N/A	26.4		71205DR-144
SS7275	Calibration Standard, 10 ng/mL	Standard	1.00E+00	PFBS	283482	10.00	9.93		71205DR-145
C0049880 Spike K	DF06V02VP0010041007, 10 ng/g, DF=100	Quality Control	1.00E+02	PFBS	15451	25.00	41.9		71205DR-146
C0049880 Spike K	DF06V02VP0010041007, 10 ng/g, DF=10	Quality Control	1.00E+01	PFBS	141858	25.00	49		71205DR-147
C0049880 Spike L	DF06V02VP0010041007, 100 ng/g, DF=100	Quality Control	1.00E+02	PFBS	66016	250.00	221		71205DR-148
C0049880	DF06V02VP0010041007, DF=100	Unknown	1.00E+02	PFBS	9708	N/A	21.6		71205DR-149
C0049880 Rep	DF06V02VP0010041007, DF=100	Unknown	1.00E+02	PFBS	9369	N/A	20.4		71205DR-150
C0049880	DF06V02VP0010041007, DF=10	Unknown	1.00E+01	PFBS	73914	N/A	24.9		71205DR-151
C0049880 Rep	DF06V02VP0010041007, DF=10	Unknown	1.00E+01	PFBS	75281	N/A	25.4		71205DR-152
SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PFBS	700346	25.00	24.7		71205DR-153
SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PFBS	1373607	50.00	48.6		71205DR-154
PFHS									
Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Peak Area	Conc. Concentration (ng/L)	Calculated Concentration (ng/L)		Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFHS	27416	0.50	0.453		71205DR-101
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFHS	58625	1.00	1.07		71205DR-102
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFHS	140460	2.50	2.68		71205DR-103
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFHS	304418	5.00	5.91		71205DR-104
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFHS	525913	10.00	10.3		71205DR-105
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFHS	1338928	25.00	26.3		71205DR-106
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFHS	2609290	50.00	51.3		71205DR-107
Methanol Wash	Methanol Wash	Unknown	1	PFHS	0	N/A	No Peak		71205DR-108
C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFHS	71367	25.00	132		71205DR-109
C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFHS	703539	25.00	138		71205DR-110
C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFHS	152296	250.00	291		71205DR-111
C0049876	DF06V01AVP0010041007, DF=100	Unknown	100	PFHS	53326	N/A	100		71205DR-112
C0049876 Rep	DF06V01AVP0010041007, DF=100	Unknown	100	PFHS	52947	N/A	95.6		71205DR-113
C0049876	DF06V01AVP0010041007, DF=10	Unknown	10	PFHS	524664	N/A	103		71205DR-114
C0049876 Rep	DF06V01AVP0010041007, DF=10	Unknown	10	PFHS	459307	N/A	89.6		71205DR-115
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFHS	25560	0.50	0.417		71205DR-116
C0049877 Spike E	DF06V01AP0010041007, 10 ng/g, DF=1000	Quality Control	1000	PFHS	82239	25.00	1530		71205DR-117
C0049877 Spike E	DF06V01AP0010041007, 10 ng/g, DF=100	Quality Control	100	PFHS	700720	25.00	1370		71205DR-118
C0049877 Spike E	DF06V01AP0010041007, 10 ng/g, DF=10	Quality Control	10	PFHS	4968960	25.00	978		71205DR-119
C0049877 Spike F	DF06V01AP0010041007, 100 ng/g, DF=1000	Quality Control	1000	PFHS	87105	250.00	1630		71205DR-120
C0049877 Spike F	DF06V01AP0010041007, 100 ng/g, DF=100	Quality Control	100	PFHS	821062	250.00	1610		71205DR-121
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFHS	50862	1.00	0.915		71205DR-122
C0049877	DF06V01AP0010041007, DF=1000	Unknown	1000	PFHS	97569	N/A	1840		71205DR-123
C0049877 Rep	DF06V01AP0010041007, DF=1000	Unknown	1000	PFHS	81999	N/A	1530		71205DR-124
C0049877	DF06V01AP0010041007, DF=100	Unknown	100	PFHS	793587	N/A	1560		71205DR-125
C0049877 Rep	DF06V01AP0010041007, DF=100	Unknown	100	PFHS	776901	N/A	1520		71205DR-126
C0049877	DF06V01AP0010041007, DF=10	Unknown	10	PFHS	5334327	N/A	1050		71205DR-127
C0049877 Rep	DF06V01AP0010041007, DF=10	Unknown	10	PFHS	5108394	N/A	1010		71205DR-128
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFHS	131643	2.50	2.51		71205DR-129
C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFHS	44439	25.00	78.9		71205DR-130
C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFHS	438565	25.00	85.5		71205DR-131
C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFHS	147505	250.00	282		71205DR-132
C0049878	DF06V01RCP0010041007, DF=100	Unknown	100	PFHS	26559	N/A	43.6		71205DR-133
C0049878 Rep	DF06V01RCP0010041007, DF=100	Unknown	100	PFHS	30818	N/A	52		71205DR-134
C0049878	DF06V01RCP0010041007, DF=10	Unknown	10	PFHS	269505	N/A	52.2		71205DR-135
C0049878 Rep	DF06V01RCP0010041007, DF=10	Unknown	10	PFHS	305463	N/A	59.3		71205DR-136
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFHS	267714	5.00	5.19		71205DR-137
C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFHS	34529	25.00	59.4		71205DR-138
C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFHS	334190	25.00	65		71205DR-139
C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFHS	113667	250.00	215		71205DR-140
C0049879	DF06V02AVP0010041007, DF=100	Unknown	100	PFHS	26468	N/A	43.5		71205DR-141
C0049879 Rep	DF06V02AVP0010041007, DF=100	Unknown	100	PFHS	22544	N/A	35.7		71205DR-142

	A	B	C	D	E	F	G	H	I	J
103	C0049879	DF06V02AVP0010041007, DF=10	Unknown	10	PFHS	253863	N/A	49.2	71205DR-143	
104	C0049879 Rep	DF06V02AVP0010041007, DF=10	Unknown	10	PFHS	203310	N/A	39.2	71205DR-144	
105	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFHS	494015	10.00	9.65	71205DR-145	
106	C0049880 Spike K	DF06V02VPV0010041007, 10 ng/g, DF=100	Quality Control	100	PFHS	89208	25.00	167	71205DR-146	
107	C0049880 Spike K	DF06V02VPV0010041007, 10 ng/g, DF=10	Quality Control	10	PFHS	875260	25.00	172	71205DR-147	
108	C0049880 Spike L	DF06V02VPV0010041007, 100 ng/g, DF=100	Quality Control	100	PFHS	198013	250.00	381	71205DR-148	
109	C0049880	DF06V02VPV0010041007, DF=100	Unknown	100	PFHS	70445	N/A	130	71205DR-149	
110	C0049880 Rep	DF06V02VPV0010041007, DF=100	Unknown	100	PFHS	73903	N/A	137	71205DR-150	
111	C0049880	DF06V02VPV0010041007, DF=10	Unknown	10	PFHS	707313	N/A	139	71205DR-151	
112	C0049880 Rep	DF06V02VPV0010041007, DF=10	Unknown	10	PFHS	717502	N/A	141	71205DR-152	
113	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFHS	1232291	25.00	24.2	71205DR-153	
114	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFHS	2395693	50.00	47.1	71205DR-154	
115										
116										
117										
118										
119										
PFOS										
120	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Peak Area (octe)	Concentration (calculated)	Concentration (ng/mL)	Run #	
121	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOS	28095	0.50	0.329	71205DR-101	
122	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOS	57014	1.00	0.914	71205DR-102	
123	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOS	136980	2.50	2.53	71205DR-103	
124	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOS	289620	5.00	5.62	71205DR-104	
125	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOS	512236	10.00	10.1	71205DR-105	
126	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOS	1301449	25.00	26.1	71205DR-106	
127	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOS	2531380	50.00	51	71205DR-107	
128	Methanol Wash	Methanol Wash	Unknown	1	PFOS	1200	N/A	< 0	71205DR-108	
129	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOS	2289301	25.00	4610	71205DR-109	
130	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOS	14162296	25.00	2860	71205DR-110	
131	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOS	2018351	250.00	4060	71205DR-111	
132	C0049876	DF06V01AVP0010041007, DF=100	Unknown	100	PFOS	2211930	N/A	4450	71205DR-112	
133	C0049876 Rep	DF06V01AVP0010041007, DF=100	Unknown	100	PFOS	2166435	N/A	4360	71205DR-113	
134	C0049876	DF06V01AVP0010041007, DF=10	Unknown	10	PFOS	13727393	N/A	2780	71205DR-114	
135	C0049876 Rep	DF06V01AVP0010041007, DF=10	Unknown	10	PFOS	12867088	N/A	2600	71205DR-115	
136	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOS	33090	0.50	0.43	71205DR-116	
137	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=1000	Quality Control	1000	PFOS	2208807	25.00	44500	71205DR-117	
138	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOS	11971040	25.00	24200	71205DR-118	
139	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOS	44119964	25.00	8930	71205DR-119	
140	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=1000	Quality Control	1000	PFOS	2081750	250.00	41900	71205DR-120	
141	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOS	12512878	250.00	25500	71205DR-121	
142	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOS	66749	1.00	1.11	71205DR-122	
143	C0049877	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOS	2542469	N/A	51200	71205DR-123	
144	C0049877 Rep	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOS	2177760	N/A	43800	71205DR-124	
145	C0049877	DF06V01PAP0010041007, DF=100	Unknown	100	PFOS	13147793	N/A	26600	71205DR-125	
146	C0049877 Rep	DF06V01PAP0010041007, DF=100	Unknown	100	PFOS	12693014	N/A	25700	71205DR-126	
147	C0049877	DF06V01PAP0010041007, DF=10	Unknown	10	PFOS	46169528	N/A	9340	71205DR-127	
148	C0049877 Rep	DF06V01PAP0010041007, DF=10	Unknown	10	PFOS	44700710	N/A	9040	71205DR-128	
149	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOS	174021	2.50	3.28	71205DR-129	
150	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOS	1329233	25.00	2670	71205DR-130	
151	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOS	9176554	25.00	1850	71205DR-131	
152	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOS	1303611	250.00	2610	71205DR-132	
153	C0049878	DF06V01RCP0010041007, DF=100	Unknown	100	PFOS	1186632	N/A	2380	71205DR-133	
154	C0049878 Rep	DF06V01RCP0010041007, DF=100	Unknown	100	PFOS	1310441	N/A	2630	71205DR-134	
155	C0049878	DF06V01RCP0010041007, DF=10	Unknown	10	PFOS	8345287	N/A	1690	71205DR-135	
156	C0049878 Rep	DF06V01RCP0010041007, DF=10	Unknown	10	PFOS	9119843	N/A	1840	71205DR-136	
157	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOS	269965	5.00	5.22	71205DR-137	
158	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOS	288031	25.00	559	71205DR-138	
159	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOS	2555750	25.00	515	71205DR-139	
160	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOS	333167	250.00	650	71205DR-140	
161	C0049879	DF06V02AVP0010041007, DF=100	Unknown	100	PFOS	289414	N/A	562	71205DR-141	
162	C0049879 Rep	DF06V02AVP0010041007, DF=100	Unknown	100	PFOS	255697	N/A	494	71205DR-142	
163	C0049879	DF06V02AVP0010041007, DF=10	Unknown	10	PFOS	2653418	N/A	535	71205DR-143	
164	C0049879 Rep	DF06V02AVP0010041007, DF=10	Unknown	10	PFOS	2276917	N/A	458	71205DR-144	
165	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOS	483521	10.00	9.55	71205DR-145	
166	C0049880 Spike K	DF06V02VPV0010041007, 10 ng/g, DF=100	Quality Control	100	PFOS	3259111	25.00	6570	71205DR-146	
167	C0049880 Spike K	DF06V02VPV0010041007, 10 ng/g, DF=10	Quality Control	10	PFOS	18319932	25.00	3710	71205DR-147	
168	C0049880 Spike L	DF06V02VPV0010041007, 100 ng/g, DF=100	Quality Control	100	PFOS	3169886	250.00	6390	71205DR-148	
169	C0049880	DF06V02VPV0010041007, DF=100	Unknown	100	PFOS	2967381	N/A	5980	71205DR-149	
170	C0049880 Rep	DF06V02VPV0010041007, DF=100	Unknown	100	PFOS	3119477	N/A	6290	71205DR-150	
171	C0049880	DF06V02VPV0010041007, DF=10	Unknown	10	PFOS	17110744	N/A	3460	71205DR-151	
172	C0049880 Rep	DF06V02VPV0010041007, DF=10	Unknown	10	PFOS	1796483	N/A	3630	71205DR-152	
173	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOS	1228884	25.00	24.6	71205DR-153	
174	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOS	2341872	50.00	47.2	71205DR-154	
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PFOA										
179	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Peak Area (octe)	Concentration (calculated)	Concentration (ng/mL)	Run #	
180	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	174611	0.50	0.36	71205DR-101	
181	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	324088	1.00	0.977	71205DR-102	
182	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	791206	2.50	2.9	71205DR-103	
183	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1609734	5.00	6.28	71205DR-104	
184	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2891639	10.00	11.6	71205DR-105	
185	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	6627517	25.00	27	71205DR-106	
186	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	12158513	50.00	49.8	71205DR-107	
187	Methanol Wash	Methanol Wash	Unknown	1	PFOA	16847	N/A	< 0	71205DR-108	
188	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	720266	25.00	261	71205DR-109	
189	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	6079998	25.00	247	71205DR-110	
190	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	1060489	250.00	402	71205DR-111	
191	C0049876	DF06V01AVP0010041007, DF=100	Unknown	100	PFOA	647854	N/A	231	71205DR-112	
192	C0049876 Rep	DF06V01AVP0010041007, DF=100	Unknown	100	PFOA	619660	N/A	220	71205DR-113	
193	C0049876	DF06V01AVP0010041007, DF=10	Unknown	10	PFOA	5547750	N/A	225	71205DR-114	
194	C0049876 Rep	DF06V01AVP0010041007, DF=10	Unknown	10	PFOA	4838272	N/A	196	71205DR-115	
195	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	166503	0.50	0.326	71205DR-116	
196	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=1000	Quality Control	1000	PFOA	1202424	25.00	4600	71205DR-117	
197	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	8606920	25.00	3520	71205DR-118	
198	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	42307709	25.00	1740	71205DR-119	
199	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=1000	Quality Control	1000	PFOA	1140004	250.00	4340	71205DR-120	
200	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	9045369	250.00	3700	71205DR-121	
201	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	307011	1.00	0.906	71205DR-122	
202	C0049877	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOA	1324428	N/A	5100	71205DR-123	
203	C0049877 Rep	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOA	1145416	N/A	4370	71205DR-124	
204	C0049877	DF06V01PAP0010041007, DF=100	Unknown	100	PFOA	9251337	N/A	3780	71205DR-125	

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BK 09/03/05

Raw Data

A	B	C	D	E	F	G	H	I	J
205	C0049877 Rep	DF06V01PAP0010041007, DF=100	Unknown	100	PFOA	8849443	N/A	3620	71205DR-126
206	C0049877	DF06V01PAP0010041007, DF=10	Unknown	10	PFOA	44632879	N/A	1840	71205DR-127
207	C0049877 Rep	DF06V01PAP0010041007, DF=10	Unknown	10	PFOA	42664598	N/A	1760	71205DR-128
208	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	720897		2.61	71205DR-129
209	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	1695021	25.00	663	71205DR-130
210	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	13411624	25.00	550	71205DR-131
211	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	2009465	250.00	793	71205DR-132
212	C0049878	DF06V01RCP0010041007, DF=100	Unknown	100	PFOA	1526160	N/A	594	71205DR-133
213	C0049878 Rep	DF06V01RCP0010041007, DF=100	Unknown	100	PFOA	1630127	N/A	637	71205DR-134
214	C0049878	DF06V01RCP0010041007, DF=10	Unknown	10	PFOA	12293753	N/A	504	71205DR-135
215	C0049878 Rep	DF06V01RCP0010041007, DF=10	Unknown	10	PFOA	12748494	N/A	522	71205DR-136
216	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1477271	5.00	5.74	71205DR-137
217	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	356131	25.00	111	71205DR-138
218	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	3076112	25.00	123	71205DR-139
219	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	711312	250.00	257	71205DR-140
220	C0049879	DF06V02AVP0010041007, DF=100	Unknown	100	PFOA	329013	N/A	99.7	71205DR-141
221	C0049879 Rep	DF06V02AVP0010041007, DF=100	Unknown	100	PFOA	268561	N/A	74.8	71205DR-142
222	C0049879	DF06V02AVP0010041007, DF=10	Unknown	10	PFOA	2912346	N/A	117	71205DR-143
223	C0049879 Rep	DF06V02AVP0010041007, DF=10	Unknown	10	PFOA	2373516	N/A	94.3	71205DR-144
224	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2605000	10.00	10.4	71205DR-145
225	C0049880 Spike K	DF06V02VP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	3091503	25.00	1240	71205DR-146
226	C0049880 Spike K	DF06V02VP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	2248410	25.00	914	71205DR-147
227	C0049880 Spike L	DF06V02VP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	3353984	250.00	1350	71205DR-148
228	C0049880	DF06V02VP0010041007, DF=100	Unknown	100	PFOA	2753273	N/A	1100	71205DR-149
229	C0049880 Rep	DF06V02VP0010041007, DF=100	Unknown	100	PFOA	2935446	N/A	1180	71205DR-150
230	C0049880	DF06V02VP0010041007, DF=10	Unknown	10	PFOA	20641529	N/A	848	71205DR-151
231	C0049880 Rep	DF06V02VP0010041007, DF=10	Unknown	10	PFOA	20694395	N/A	850	71205DR-152
232	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5894625	25.00	24	71205DR-153
233	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	11033219	50.00	45.2	71205DR-154
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PFOA Confirm Ion

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Peak Area	Conc (ug/L)	Calculated Concentration (ng/L)	Run #
240	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5786	0.50	0.457
241	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	9049	1.00	0.927
242	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	22433	2.50	2.85
243	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	47743	5.00	6.5
244	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	82988	10.00	11.6
245	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	189207	25.00	26.9
246	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	353598	50.00	50.6
247	Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak
248	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	38544	25.00	518
249	C0049876 Spike C	DF06V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	346878	25.00	496
250	C0049876 Spike D	DF06V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	43203	250.00	585
251	C0049876	DF06V01AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	34675	N/A	462
252	C0049876 Rep	DF06V01AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	32294	N/A	427
253	C0049876	DF06V01AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	321610	N/A	459
254	C0049876 Rep	DF06V01AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	289196	N/A	413
255	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5336	0.50	0.392
256	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=1000	Quality Control	1000	PFOA Confirm Ion	47822	25.00	6510
257	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	373424	25.00	5340
258	C0049877 Spike E	DF06V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	2471900	25.00	3560
259	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=1000	Quality Control	1000	PFOA Confirm Ion	44545	250.00	6040
260	C0049877 Spike F	DF06V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	388797	250.00	5560
261	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	8632	1.00	0.867
262	C0049877	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOA Confirm Ion	54301	N/A	7440
263	C0049877 Rep	DF06V01PAP0010041007, DF=1000	Unknown	1000	PFOA Confirm Ion	45887	N/A	6230
264	C0049877	DF06V01PAP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	416026	N/A	5950
265	C0049877 Rep	DF06V01PAP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	401426	N/A	5740
266	C0049877	DF06V01PAP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	2668936	N/A	3840
267	C0049877 Rep	DF06V01PAP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	2559259	N/A	3680
268	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	19440	2.50	2.42
269	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	106039	25.00	1490
270	C0049878 Spike G	DF06V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	974687	25.00	1400
271	C0049878 Spike H	DF06V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	109450	250.00	1540
272	C0049878	DF06V01RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	97681	N/A	1370
273	C0049878 Rep	DF06V01RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	107926	N/A	1520
274	C0049878	DF06V01RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	885379	N/A	1270
275	C0049878 Rep	DF06V01RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	929218	N/A	1330
276	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	37786	5.00	5.07
277	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	12691	25.00	145
278	C0049879 Spike I	DF06V02AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	113265	25.00	159
279	C0049879 Spike J	DF06V02AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	21866	250.00	277
280	C0049879	DF06V02AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	12612	N/A	144
281	C0049879 Rep	DF06V02AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	10392	N/A	112
282	C0049879	DF06V02AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	109891	N/A	155
283	C0049879 Rep	DF06V02AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	92514	N/A	129
284	SS7275	Calibration Standard, 10 ng/mL	Standard	1.0	PFOA Confirm Ion	69858	10.00	9.69
285	C0049880 Spike K	DF06V02VP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm Ion	155465	25.00	2200
286	C0049880 Spike K	DF06V02VP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PFOA Confirm Ion	1346466	25.00	1940
287	C0049880 Spike L	DF06V02VP0010041007, 100 ng/g, DF=100	Quality Control	100.0	PFOA Confirm Ion	159517	250.00	2260
288	C0049880	DF06V02VP0010041007, DF=100	Unknown	100.0	PFOA Confirm Ion	140273	N/A	1980
289	C0049880 Rep	DF06V02VP0010041007, DF=100	Unknown	100.0	PFOA Confirm Ion	146203	N/A	2070
290	C0049880	DF06V02VP0010041007, DF=10	Unknown	10.0	PFOA Confirm Ion	1234391	N/A	1770
291	C0049880 Rep	DF06V02VP0010041007, DF=10	Unknown	10.0	PFOA Confirm Ion	1245540	N/A	1790
292	SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PFOA Confirm Ion	168147	25.00	23.8
293	SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PFOA Confirm Ion	321778	50.00	46

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BAL 08/03/05

Vegetation Set #2:
Internal Chain of Custody/Fortification
Sheets

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.	Matrix:
P760 and P 1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	0829	07/06/05	CLB

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0073483 Control	5.0	—
—	C0073483 Spike A	5.0	2.5 ng/g
—	C0073483 Spike B	5.0	10 ng/g
Blank			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	^{Micropipet} Syringe [Ⓟ] Volume (μL)	Initials/Date
Spike A	SS0007404 (0.1 μg/mL)	125	25/50	BAK 07/12/05
Spike B	SS0007404 (0.1 μg/mL)	500	100/200	BAK 07/12/05
Spike —	—	—	—	—
Spike —	—	—	—	—

All samples were measured on:			
Balance ID:	Time:	Date:	Initials:
BALANCE 20	1030	07/06/05	CLB

After measuring samples were returned to:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1225	07/06/05	CLB

Comments: ① ② BAK 07/12/05

Analysis Summary	Data Set ID:	071205E	Initials/Date:	AMB 10/14/05
	Data Set ID:	—	Initials/Date:	— / —
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:
	BAK 10/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	00073483 Control	↓									---	↓	---	---	Methanol	RE0020201
---	00073483 Spk A	↓									---	↓	---	---	Acetonitrile	RE0020234
---	00073483 Spk B	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	---	---	Carbon	RE0019743
															Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019762
															2% Ascorbic Acid in Methanol	SL0014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640
															Toluene	RE0007267
Initials/Date	-	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/13/05	---	AMB 07/14/05	---	---	Extraction Items Initials/Date	BAK 07/12/05
															Analysis Items Initials/Date	* AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes. Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/12/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes. Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

After this step, samples were covered w/ foil and stored in Refrigerator 32 (IN0000708) overnight.
BAK 07/12/05

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0376

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ : FREEZER 34	Time: 1001	Date: 07/06/05	Initials: <i>CLB</i>

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
_____	C0049881 Spike C	5.0	10 ^{ng/g}
_____	C0049881 Spike D	5.0	100 ^{ng/g}
_____	C0049881	5.0	10 ^{ng/g} *
_____	C0049881 Rep.	5.0	10 ^{ng/g} *

BAK
07/12/05

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	-Syringe ① Volume (μL)	Initials/Date
Spike C	SS0007404 (0.1 ^{ng/mL})	500	100/200	<i>BAK</i> 07/12/05
Spike D	SS0007403 (1.0 ^{ng/mL})	500	100/200	<i>BAK</i> 07/12/05
Spike *	SS0007505 (0.1 ^{ng/mL} 13C)	500	100/200	<i>BAK</i> 07/12/05
Spike _____	_____	_____	_____	_____

Microject

All samples were measured on:			
Balance ID: BALANCE 20	Time: 1030	Date: 07/06/05	Initials: <i>CLB</i>

After measuring samples were returned to:			
CED ID ¹ : FREEZER 34	Time: 1225	Date: 07/06/05	Initials: <i>CLB</i>

Comments: ① ② *BAK* 07/12/05

Analysis Summary	Data Set ID:	071205E	Initials/Date:	<i>AMB</i> / 07/14/05
	Data Set ID:	071205R	Initials/Date:	<i>AMB</i> / 07/20/05
	Data Set ID:	_____	Initials/Date:	_____ / _____

Set extraction/analysis data verified by	Initials/Date:
<i>JP</i>	10/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Oxygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Oxygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	00049881 Spk C	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	0.1 1.0 0.1 1.0	↓	Methanol	RE0020201
---	00049881 Spk D	↓	↓	↓	↓	↓	↓	↓	↓	↓	0.1/1.0	↓	0.1 1.0	↓	Acetonitrile	RE0020234
---	00049881	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	0.1 1.0 0.1 1.0	↓	Carbon	RE0019743
---	00049881 Rip.	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	0.1 1.0 0.1 1.0	↓	Silica gel	RE0000639
Florisil RE0000641																
LC-NH2 RE0019762																
2% Ascorbic Acid in Methanol SLC014702																
1-Octanol RE0016118																
30% Dimethyldichlorosilane in Toluene Solution SLC014640																
Toluene RE0007267																
Initials/Date	---	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	AMB 07/14/05	AMB 07/14/05	AMB 07/26/05	AMB 07/26/05	Extraction Items Initials/Date	BAK 07/12/05
															Analysis Items Initials/Date	* AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

DF=10,100 AMB 07/26/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605
- STEP 3 = Pack and Condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/12/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

After this step, samples were covered w/ foil and stored in Refrigerator 32 (IN0000708) overnight.
BAK 07/12/05

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0318

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	PC000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1001	07/06/05	CEB

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0049882 Spike E	5.0	10 ng/g
—	C0049882 Spike F	5.0	100 ng/g
—	C0049882	5.0	10 ng/g*
—	C0049882 Rep.	5.0	10 ng/g*
Blank 07/12/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	Micro pipet Syringe ① Volume (μL)	Initials/Date
Spike E	SS0007404 (0.1 μg/mL)	500	100/200	BRK 07/12/05
Spike F	SS0007403 (1.0 μg/mL)	500	100/200	BRK 07/12/05
Spike *	SS0007505 (0.1 μg/mL) ^{13C}	500	100/200	BRK 07/12/05
Spike —	—	—	—	—

All samples were measured on:			
Balance ID:	Time:	Date:	Initials:
BALANCE RD	1030	07/06/05	CEB

After measuring samples were returned to:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1225	07/06/05	CEB

Comments: ① ② BRK 07/12/05

Analysis Summary	Data Set ID:	071205E	Initials/Date:	AMB 107/14/05
	Data Set ID:	071205R	Initials/Date:	AMB 107/26/05
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:
—	— 108/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	C0049882 SPK F										---		0.1/1.0 0.1/10		Methanol	RE0020201
---	C0049882 SPK F										0.1/1.0		0.1/10		Acetonitrile	RE0020234
---	C0049882										---		0.1/1.0 0.1/10		Carbon	RE0019743
---	C0049882 Rep.	✓	✓	✓	✓	✓	✓	✓	✓	✓	---	↓	0.1/1.0 0.1/10	↓	Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019762
															2% Ascorbic Acid in Methanol	SL0014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640
															Toluene	RE0007267
Initials/Date	-	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	AMB 07/14/05	AMB 07/14/05	AMB 07/20/05	AMB 07/20/05	Extraction Items Initials/Date	BAK 07/12/05
															Analysis Items Initials/Date	* AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

DTF=10.100 AMB 07/20/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
 STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605
 STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
 STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/05/05
 STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605
 STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182
 STEP 7 = Repeat steps 5 through 6 again.
 STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
 STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
 STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
 STEP 11 = Perform LC/MS/MS re-analysis.

After this step, samples were stored in Refrigerator 32 (IN000708) (after being covered w/ foil) overnight.
BAK 07/12/05

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0320

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ : FREEZER 34	Time: 1002	Date: 07/06/05	Initials: <i>CLB</i>

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0049883 Spike G	5.0	10 ^{ng/g}
—	C0049883 Spike H	5.0	100 ^{ng/g}
—	C0049883	5.0	10 ^{ng/g} *
—	C0049883 Rep.	5.0	10 ^{ng/g} *
Blank <i>BAK 07/12/05</i>			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	Micropipet - Syringe Volume (μL)	Initials/Date
Spike G	SS0007404 (0.1 ^{ng/mL})	500	100/200	<i>BAK 07/12/05</i>
Spike H	SS0007403 (1.0 ^{ng/mL})	500	100/200	<i>BAK 07/12/05</i>
Spike *	SS0007505 (0.1 ^{ng/mL})	500	100/200	<i>BAK 07/12/05</i>
Spike —	—	—	—	—

All samples were measured on:			
Balance ID: BALANCE 20	Time: 1030	Date: 07/06/05	Initials: <i>CLB</i>

After measuring samples were returned to:			
CED ID ¹ : FREEZER 34	Time: 1225	Date: 07/06/05	Initials: <i>CLB</i>

Comments: ① (CC) *BAK 07/12/05*

Analysis Summary	Data Set ID:	071205E	Initials/Date:	<i>AMB 1 07/14/05</i>
	Data Set ID:	071205ER	Initials/Date:	<i>AMB 107/26/05</i>
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:	<i>BAK 1 08/22/05</i>
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¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: _____ Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	C0049883 Spk G	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	① 0.1 0.1 1.0 1.0	↓	Methanol	RE0020201
---	C0049883 Spk H	↓	↓	↓	↓	↓	↓	↓	↓	↓	0.1/1.0	↓	0.1 0.1 1.0 1.0	↓	Acetonitrile	RE0020234
---	C0049883	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	① 0.1 0.1 1.0 1.0	↓	Carbon	RE0019743
---	C0049883 Rep.	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	① 0.1 0.1 1.0 1.0	↓	Silica gel	RE0000639
BAK 07/12/05																
FLORISIL																
LC-NH2																
2% ASCORBIC ACID IN METHANOL																
1-OCTANOL																
30% DIMETHYLDICHLOROSILANE IN TOLUENE SOLUTION																
TOLUENE																
Initials/Date	-	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/13/05	BAK 07/13/05	AMB 07/14/05	AMB 07/14/05	AMB 07/26/05	AMB 07/26/05	Extraction Items Initials/Date	BAK 07/12/05
															Analysis Items Initials/Date	* AMB 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

① DF = 10, 100 AMB 07/26/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: 1N0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: 1N0000182 Glassware silanized: initials/Date BAK 07/05/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: 1N0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: 1N0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

→ After this step, samples were covered w/ foil and stored in Refrigerator 32 (1N0000708) overnight - BAK 07/12/05

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0322

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.:	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1002	07/06/05	CLB

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
_____	C0049884 Spike I	5.0	10 ^{ng} /g
_____	C0049884 Spike J	5.0	100 ^{ng} /g
_____	C0049884	5.0	10 ^{ng} /g*
_____	C0049884 Rep.	5.0	10 ^{ng} /g*

BAK
07/12/05

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	Micropipet Syringe ^① Volume (μL)	Initials/Date
Spike I	SS0007404 (0.1 ^{μg} /mL)	500	100/200	BAK 07/12/05
Spike J	SS0007403 (1.0 ^{μg} /mL)	500	100/200	BAK 07/12/05
Spike *	SS0007505 (0.1 ^{μg} /mL ^{1c})	500	100/200	BAK 07/12/05
Spike _____	_____	_____	_____	_____

All samples were measured on:			
Balance ID:	Time:	Date:	Initials:
BALANCE 20	1030	07/06/05	CLB

After measuring samples were returned to:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1225	07/06/05	CLB

Comments: ① ② BAK 07/12/05

Analysis Summary	Data Set ID:	071205E	Initials/Date:	AMB 107/14/05
	Data Set ID:	071205R	Initials/Date:	AMB 107/26/05
	Data Set ID:	-	Initials/Date:	- / -

Set extraction/analysis data verified by	Initials/Date:
	BAK 108/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Oxygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Oxygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	C0049224 Spk 1	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	① 0.1 1.0 0.1 1.0	↓	Methanol	RE0020201
---	C0049224 Spk 2	↓	↓	↓	↓	↓	↓	↓	↓	↓	0.1/1.0	↓	① 0.1 1.0 0.1 1.0	↓	Acetonitrile	RE0020234
---	C0049224 Spk 3	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	① 0.1 1.0 0.1 1.0	↓	Carbon	RE0019743
---	C0049224 Spk 4	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	① 0.1 1.0 0.1 1.0	↓	Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019762
															2% Ascorbic Acid in Methanol	SL0014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640
															Toluene	RE0007267
Initials/Date	-	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	AMBS 07/14/05	AMBS 07/14/05	AMBS 07/26/05	AMBS 07/26/05	Extraction Items Initials/Date	BAK 07/12/05
			BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	AMBS 07/14/05	AMBS 07/14/05	AMBS 07/26/05	AMBS 07/26/05	Analysis Items Initials/Date	* AMBS 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

① DF = 10, 100 AMBS 07/26/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: 1N0000665
- STEP 3 = Pack and Condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: 1N0000182 Glassware silanized: initials/Date BAK 07/07/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: 1N0000665
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: 1N0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

→ After this step, samples were covered w/ foil and stored in Refrigerator 32 (1N0000708) overnight.
BAK 07/12/05

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0324

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ : FREEZER 34	Time: 1002	Date: 07/06/05	Initials: CLB

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0049885 Spike K	5.0	10 ^{mg/g}
—	C0049885 Spike L	5.0	100 ^{mg/g}
—	C0049885	5.0	10 ^{mg/g} *
—	C0049885 Rep.	5.0	10 ^{mg/g} *
BAK 07/12/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	Micropipet Syringe [Ⓢ] Volume (μL)	Initials/Date
Spike K	SS0007404 (0.1 ^{μg/mL})	500	100/200	BAK 07/12/05
Spike L	SS0007403 (1.0 ^{μg/mL})	500	100/200	BAK 07/12/05
Spike *	SS0007505 (0.1 ^{μg/mL})	500	100/200	BAK 07/12/05
Spike —	—	—	—	—

All samples were measured on:			
Balance ID: BALANCE 20	Time: 1030	Date: 07/06/05	Initials: CLB

After measuring samples were returned to:			
CED ID ¹ : FREEZER 34	Time: 1225	Date: 07/06/05	Initials: CLB

Comments: (1) (CC) BAK 07/12/05

Analysis Summary	Data Set ID:	071205E	Initials/Date:	AMB / 07/14/05
	Data Set ID:	071205R	Initials/Date:	AMB / 07/26/05
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:	BAK / 08/22/05
--	----------------	----------------

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	00049085 Spk K										---		① 0.1 10 0.1 10		Methanol	RE0020201
---	00049085 Spk L										0.1/1.0		① 0.1 10 0.1 10		Acetonitrile	RE0020284
---	00049085 Spk M										---		① 0.1 10 0.1 10		Carbon	RE0019743
---	00049085 Spk P	✓	✓	✓	✓	✓	✓	✓	✓	✓	---	✓	① 0.1 10 0.1 10	✓	Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019762
															2% Ascorbic Acid in Methanol	SLO014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SLO014640
															Toluene	RE0007267
Initials/Date	-	See ICOC page.	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/12/05	BAK 07/13/05	BAK 07/13/05	AMBS 07/14/05	AMBS 07/14/05	AMBS 07/20/05	AMBS 07/20/05	Extraction Items Initials/Date	BAK 07/12/05
															Analysis Items Initials/Date	* AMBS 07/14/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

① DF = 10,100 AMBS 07/20/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
 STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: 1N0000605
 STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
 STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: 1N0000182 Glassware silanized: initials/Date BAK 07/05/05
 STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: 1N0000605
 STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: 1N0000182
 STEP 7 = Repeat steps 5 through 6 again. → After this step, samples were covered w/ foil and stored in Refrigerator 32 (1N0000708) overnight.
 STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
 STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
 STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
 STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/13/05

0326

Vegetation Set #2

Data Set 071205E

Extracted on 07/12/05

(Analyzed on 07/15/05)

AMB 07/14/05

\\Abbeyroad\PE Sciex Data\Projects\P1131 Decatur Monitoring Study Batch:071205E VEG P760 Tab:Sample Set:SET1 AcqMethod:P760-1131 Isomer Separation

Sample	Sample Name	Sample ID	Vial Position	Data File	Run #
1	SS7279	Calibration Standard, 0.5 ng/mL	1	08_071205E071205E	071205E-201
2	SS7278	Calibration Standard, 1.0 ng/mL	2	08_071205E071205E	071205E-202
3	SS7277	Calibration Standard, 2.5 ng/mL	3	08_071205E071205E	071205E-203
4	SS7276	Calibration Standard, 5.0 ng/mL	4	08_071205E071205E	071205E-204
5	SS7275	Calibration Standard, 10 ng/mL	5	08_071205E071205E	071205E-205
6	SS7274	Calibration Standard, 25 ng/mL	6	08_071205E071205E	071205E-206
7	SS7273	Calibration Standard, 50 ng/mL	7	08_071205E071205E	071205E-207
8	Methanol Wash	Methanol Wash	92	08_071205E071205E	071205E-208
9	Reagent Control	C0054391 Control (1)	41	08_071205E071205E	071205E-209
10	Reagent Spk A	C0054391 Spike A, 2.5 ng/g (1)	42	08_071205E071205E	071205E-210
11	Reagent Spk B	C0054391 Spike B, 10 ng/g (1)	43	08_071205E071205E	071205E-211
12	SS7279	Calibration Standard, 0.5 ng/mL	1	08_071205E071205E	071205E-212
13	C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g	44	08_071205E071205E	071205E-213
14	C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	45	08_071205E071205E	071205E-214
15	C0049881	DF06V02RCP0010041007	46	08_071205E071205E	071205E-215
16	C0049881 Rep	DF06V02RCP0010041007	47	08_071205E071205E	071205E-216
17	SS7278	Calibration Standard, 1.0 ng/mL	2	08_071205E071205E	071205E-217
18	C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g	48	08_071205E071205E	071205E-218
19	C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	49	08_071205E071205E	071205E-219
20	C0049882	DF09V01AVP0010041007	50	08_071205E071205E	071205E-220
21	C0049882 Rep	DF09V01AVP0010041007	51	08_071205E071205E	071205E-221
22	SS7277	Calibration Standard, 2.5 ng/mL	3	08_071205E071205E	071205E-222
23	C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g	52	08_071205E071205E	071205E-223
24	C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	53	08_071205E071205E	071205E-224
25	C0049883	DF09V01PAP0010041007	54	08_071205E071205E	071205E-225
26	C0049883 Rep	DF09V01PAP0010041007	55	08_071205E071205E	071205E-226
27	SS7276	Calibration Standard, 5.0 ng/mL	4	08_071205E071205E	071205E-227
28	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g	56	08_071205E071205E	071205E-228
29	C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	57	08_071205E071205E	071205E-229
30	C0049884	DF09V01RCP0010041007	58	08_071205E071205E	071205E-230
31	C0049884 Rep	DF09V01RCP0010041007	59	08_071205E071205E	071205E-231
32	SS7275	Calibration Standard, 10 ng/mL	5	08_071205E071205E	071205E-232
33	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g	60	08_071205E071205E	071205E-233
34	C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	61	08_071205E071205E	071205E-234
35	C0049885	DF09V02RCP0010041007	62	08_071205E071205E	071205E-235
36	C0049885 Rep	DF09V02RCP0010041007	63	08_071205E071205E	071205E-236
37	SS7274	Calibration Standard, 25 ng/mL	6	08_071205E071205E	071205E-237
38	SS7273	Calibration Standard, 50 ng/mL	7	08_071205E071205E	071205E-238

(1) (R) 73483 BAK 09/20/05

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Study No: NA

Exygen Study No: P760/P1131

Instrument: API 4000 LC/MS/MS System, (LC/MS/MS #8)
Turbo Ion Spray Liquid Introduction Interface

Computer: DELL OptiPlex GX400

Software: Windows NT, Analyst 1.4

HPLC Equipment: 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 (Exygen ID MA0018925)
Column Temperature: 35°C

Mobile Phase (A) : 2 mM ammonium acetate in water (SL0014984)
Mobile Phase (B) : Methanol (RE0020196)

<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

Injected Volume: 15 µL

Ions monitored:

<u>Analyte</u>	<u>Mode</u>	<u>Transition Monitored</u>
PFBS	negative	299 → 99
PFHS	negative	399 → 80
PFOS	negative	499 → 80
PFOA	negative	413 → 369
PFOA Confirm Ion	negative	413 → 219
¹³ C PFOA	negative	415 → 370

Analyst: Amy Sheehan *AMB 07/14/05*
Exygen Research
3058 Research Drive, State College, PA 16801
Phone : (814) 272-1039 FAX : (814) 231-1580

NOTE: ALL HANDWRITTEN PEAK IDENTIFICATIONS BY *AMB / 07/21/05*

CEE 07/28/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
1 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	170926	0.500	0.378	071205E-201
2 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	299429	1.00	0.955	071205E-202
3 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	655566	2.50	2.55	071205E-203
4 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1308859	5.00	5.48	071205E-204
5 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2622018	10.0	11.4	071205E-205
6 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5770498	25.0	25.5	071205E-206
7 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10926992	50.0	48.6	071205E-207
8 Methanol Wash	Methanol Wash	Unknown	1	PFOA	22846	N/A	< 0	071205E-208
9 Reagent Control	C0073483 Control	Unknown	1	PFOA	47485	N/A	< 0	071205E-209
10 Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFOA	1619444	6.25	6.87	071205E-210
11 Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFOA	6104566	25.0	27.0	071205E-211
12 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	165897	0.500	0.356	071205E-212
13 C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g	Quality Control	1	PFOA	39819662	25.0	178.	071205E-213
14 C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	9453050	250.	420.	071205E-214
15 C0049881	DF06V02RCP0010041007	Unknown	1	PFOA	38546242	N/A	172.	071205E-215
16 C0049881 Rep	DF06V02RCP0010041007	Unknown	1	PFOA	39916149	N/A	179.	071205E-216
17 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	288145	1.00	0.904	071205E-217
18 C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g	Quality Control	1	PFOA	8899726	25.0	39.5	071205E-218
19 C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	4052837	250.	178.	071205E-219
20 C0049882	DF09V01AVP0010041007	Unknown	1	PFOA	5673154	N/A	25.0	071205E-220
21 C0049882 Rep	DF09V01AVP0010041007	Unknown	1	PFOA	5409398	N/A	23.9	071205E-221
22 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	785817	2.50	3.14	071205E-222
23 C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g	Quality Control	1	PFOA	25888168	25.0	116.	071205E-223
24 C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	6533125	250.	289.	071205E-224
25 C0049883	DF09V01PAP0010041007	Unknown	1	PFOA	21086657	N/A	94.2	071205E-225
26 C0049883 Rep	DF09V01PAP0010041007	Unknown	1	PFOA	22897488	N/A	102.	071205E-226
27 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1380382	5.00	5.80	071205E-227
28 C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g	Quality Control	1	PFOA	23339018	25.0	104.	071205E-228
29 C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	7497863	250.	332.	071205E-229
30 C0049884	DF09V01RCP0010041007	Unknown	1	PFOA	20135416	N/A	89.9	071205E-230
31 C0049884 Rep	DF09V01RCP0010041007	Unknown	1	PFOA	19710177	N/A	88.0	071205E-231
32 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2526179	10.0	10.9	071205E-232
33 C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g	Quality Control	1	PFOA	44450723	25.0	199.	071205E-233
34 C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	11013769	250.	490.	071205E-234
35 C0049885	DF09V02RCP0010041007	Unknown	1	PFOA	42795533	N/A	191.	071205E-235
36 C0049885 Rep	DF09V02RCP0010041007	Unknown	1	PFOA	42881886	N/A	192.	071205E-236
37 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5413773	25.0	23.9	071205E-237
38 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10827940	50.0	48.2	071205E-238

① Requires dilution, see data set 071205E.R. BAK 07/28/05

Fish Conversion

Compound: PFOA

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049881 Spk D	DF06-V02-RCP001-0-041007	420	168
C0049882	DF09-V01-AVP001-0-041007	25.0	10.0
C0049882 Rep	DF09-V01-AVP001-0-041007	23.9	9.56
C0049882 Spk E	DF09-V01-AVP001-0-041007	39.5	15.8
C0049882 Spk F	DF09-V01-AVP001-0-041007	178	71.2
C0049883 Spk H	DF09-V01-PAP001-0-041007	289	116
C0049884 Spk J	DF09-V01-RCP001-0-041007	332	133

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = ~~Peak Area~~ ^{AT OR ABOVE 0.2 ng/g} ①

NQ = Not quantifiable = Measured concentration ^{below} Limit of Quantitation (LOQ) which is 0.8 ng/g.

^
BETWEEN 0.2 ng/g AND ①

4①

① (RE) MLC 2/20/05

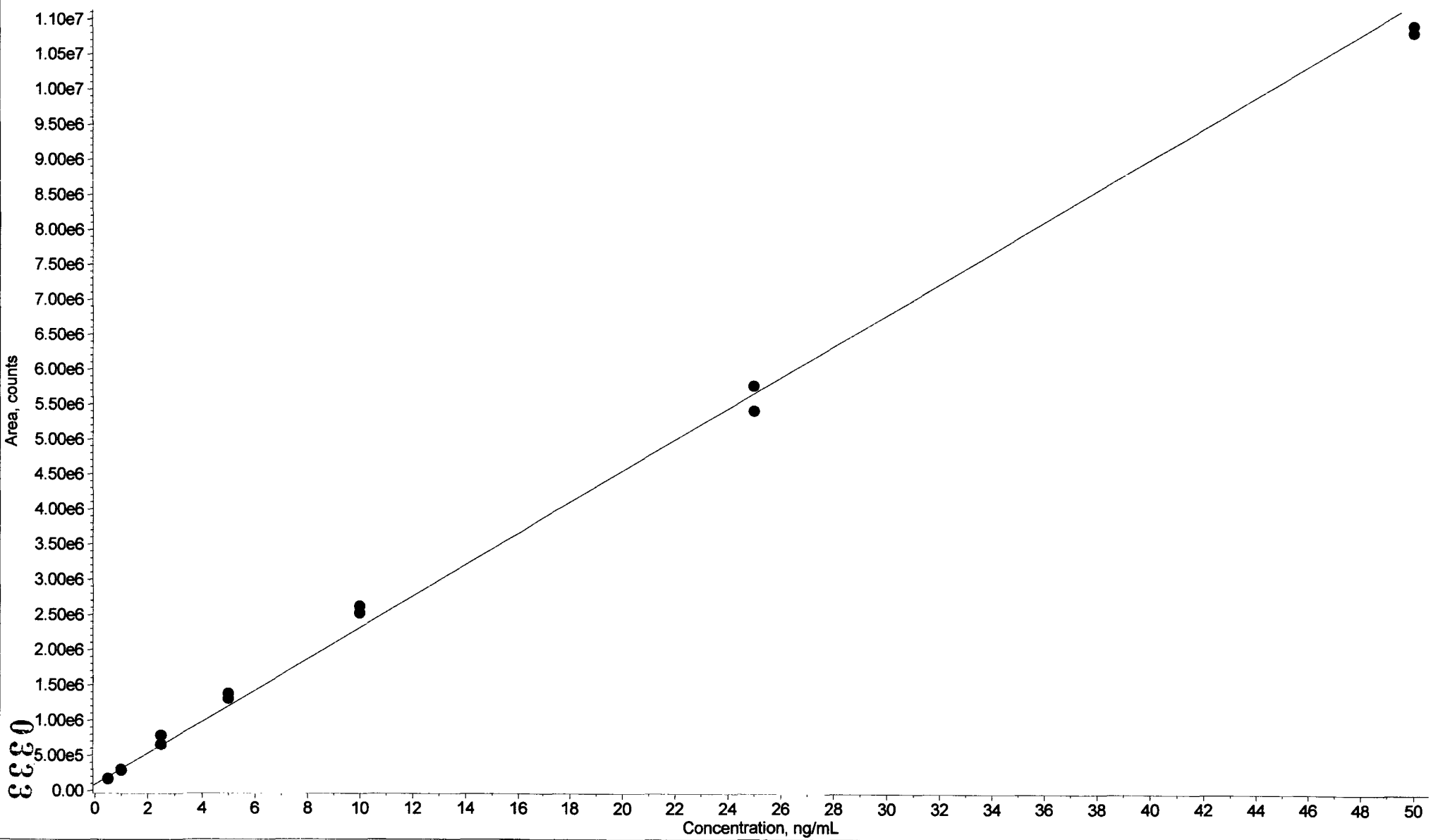
0331

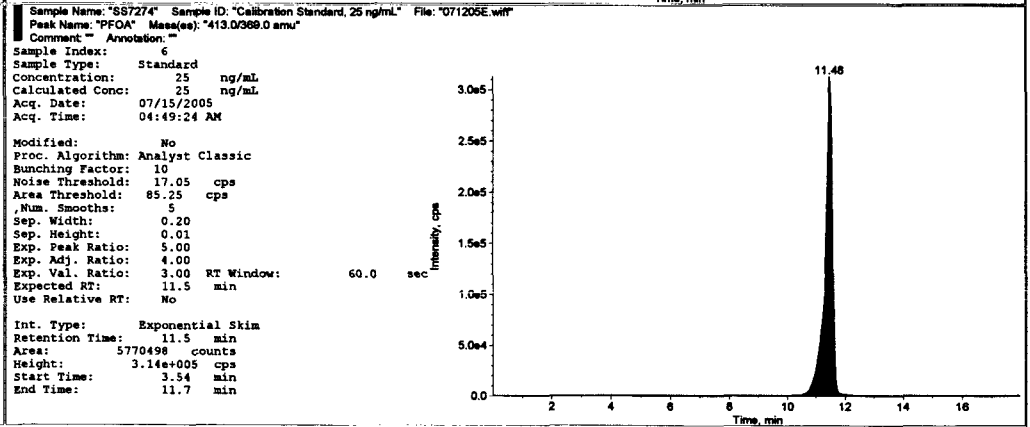
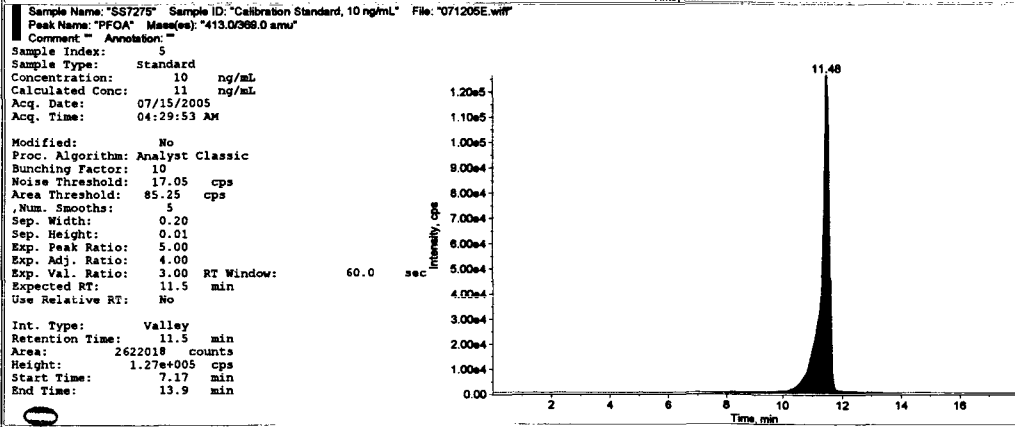
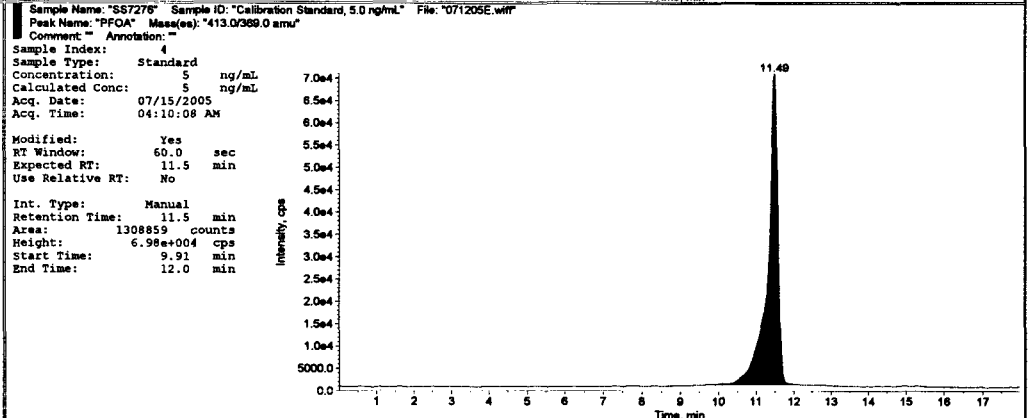
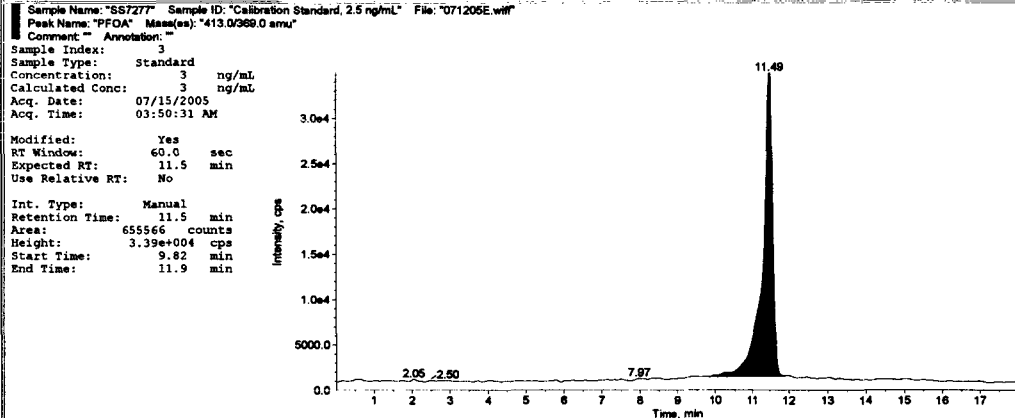
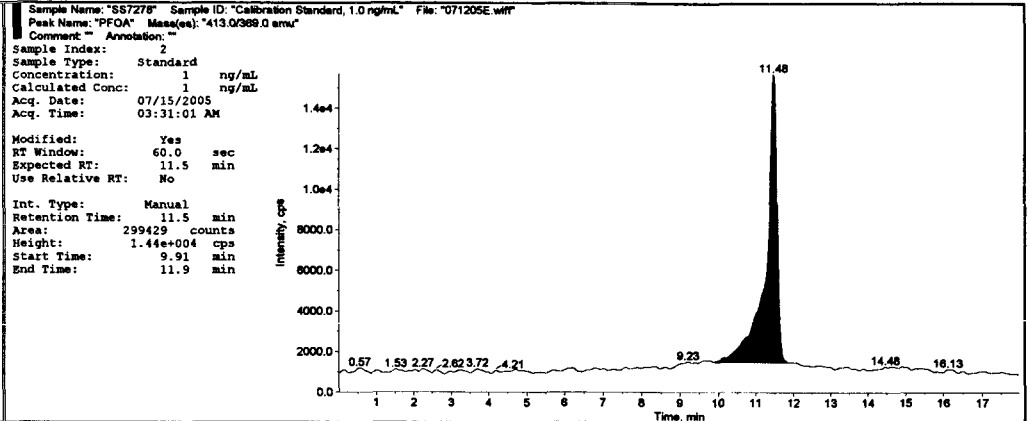
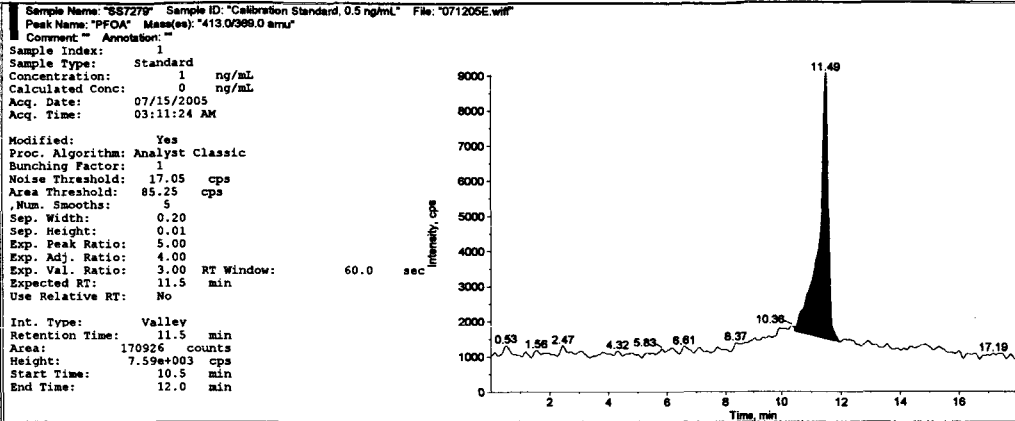
BK 02/20/05

	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound: PFOA		Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049881 Spk D	DF06-V02-RCP001-0-041007	=Raw Data!H142	=(C9*2)/5
10				
11	C0049882	DF09-V01-AVP001-0-041007	=Raw Data!H148	=(C11*2)/5
12	C0049882 Rep	DF09-V01-AVP001-0-041007	=Raw Data!H149	=(C12*2)/5
13	C0049882 Spk E	DF09-V01-AVP001-0-041007	=Raw Data!H146	=(C13*2)/5
14	C0049882 Spk F	DF09-V01-AVP001-0-041007	=Raw Data!H147	=(C14*2)/5
15				
16	C0049883 Spk H	DF09-V01-PAP001-0-041007	=Raw Data!H152	=(C16*2)/5
17				
18	C0049884 Spk J	DF09-V01-RCP001-0-041007	=Raw Data!H157	=(C18*2)/5
19				

AMB 07/21/05

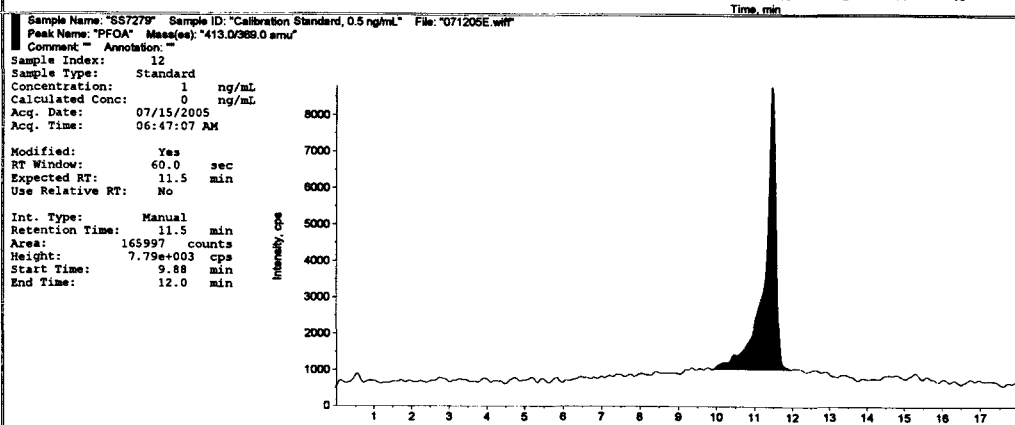
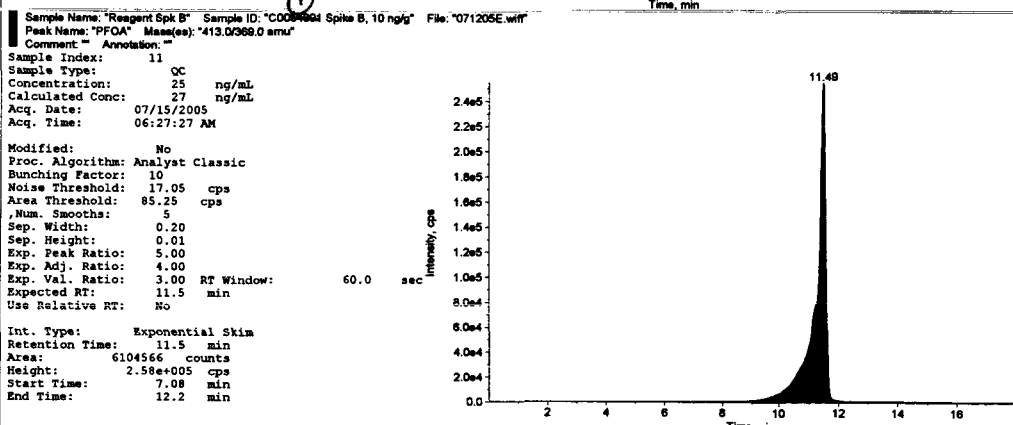
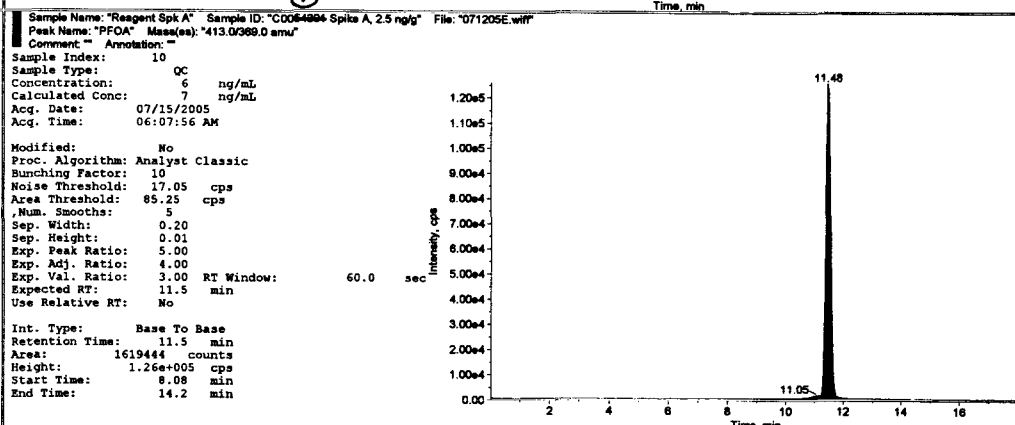
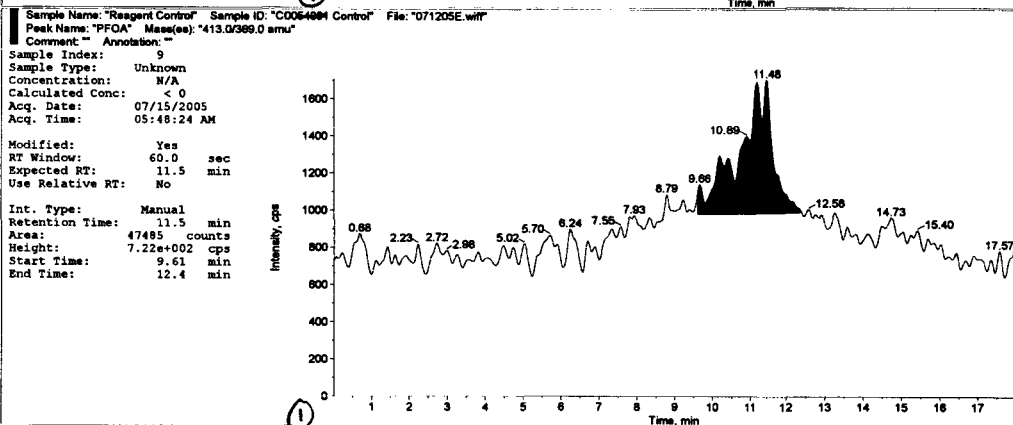
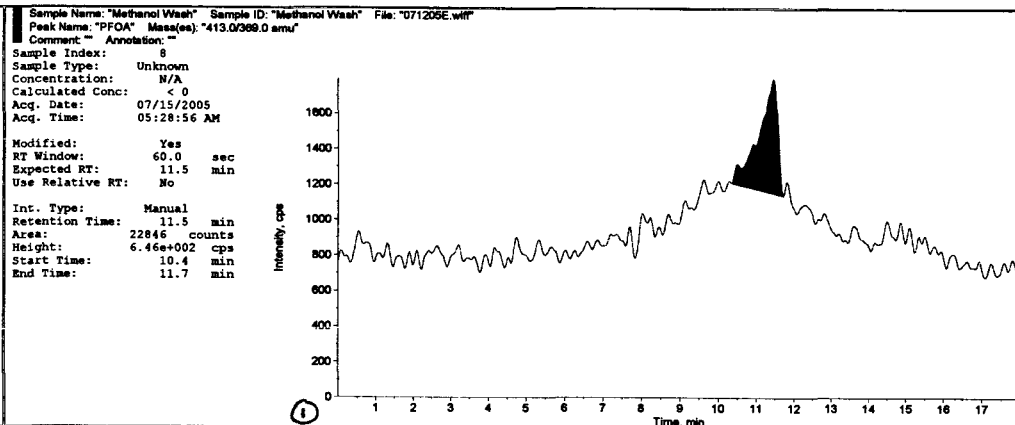
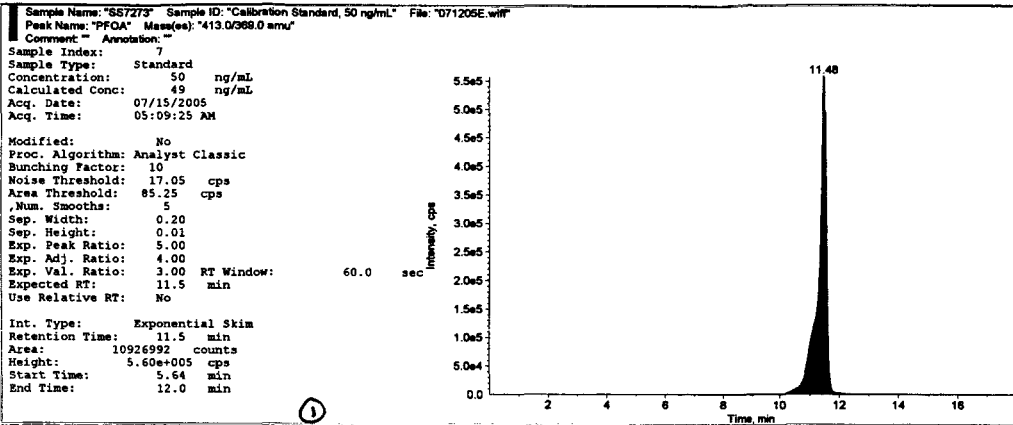
■ Untitled 6 (PFOA): "Linear" Regression ("1 / x" weighting): $y = 2.23e+005 x + 8.65e+004$ ($r = 0.9974$)





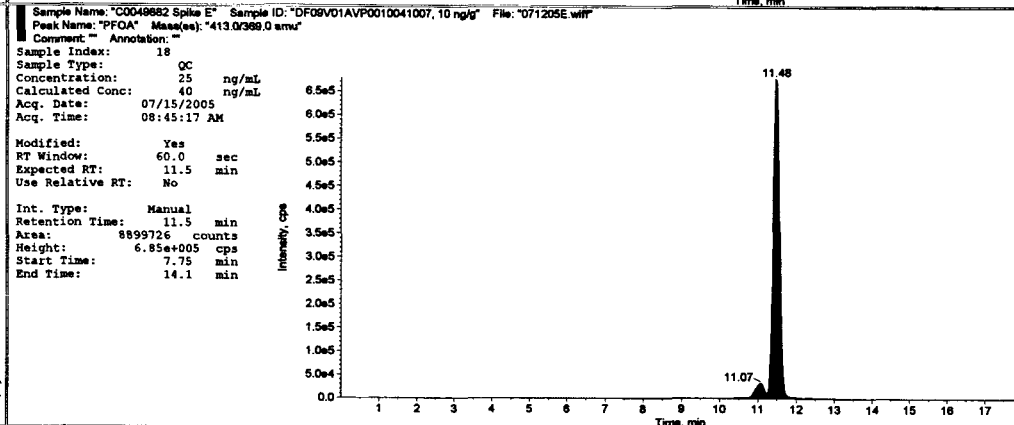
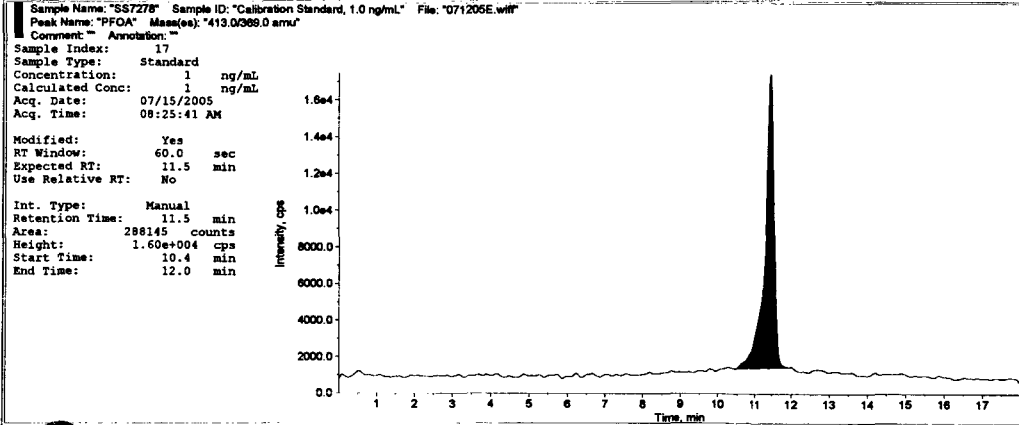
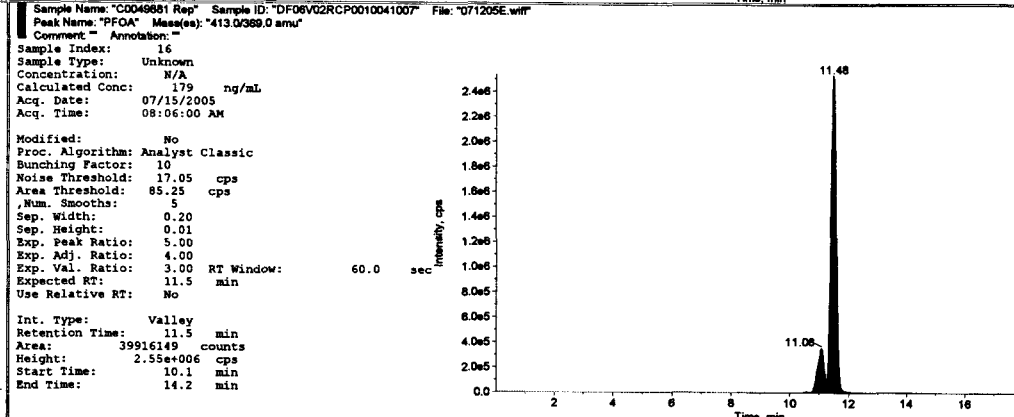
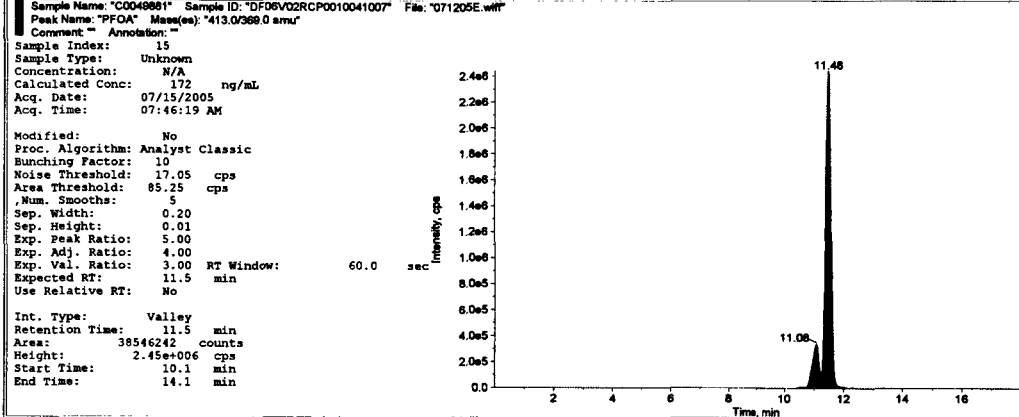
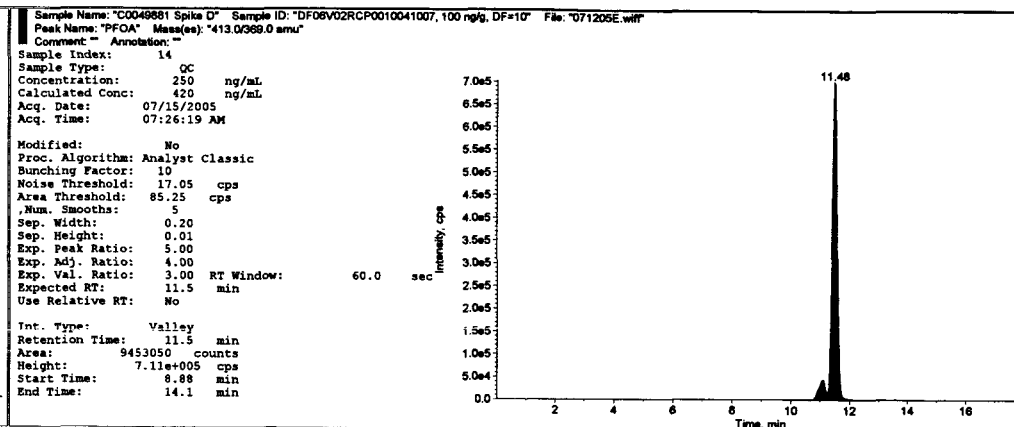
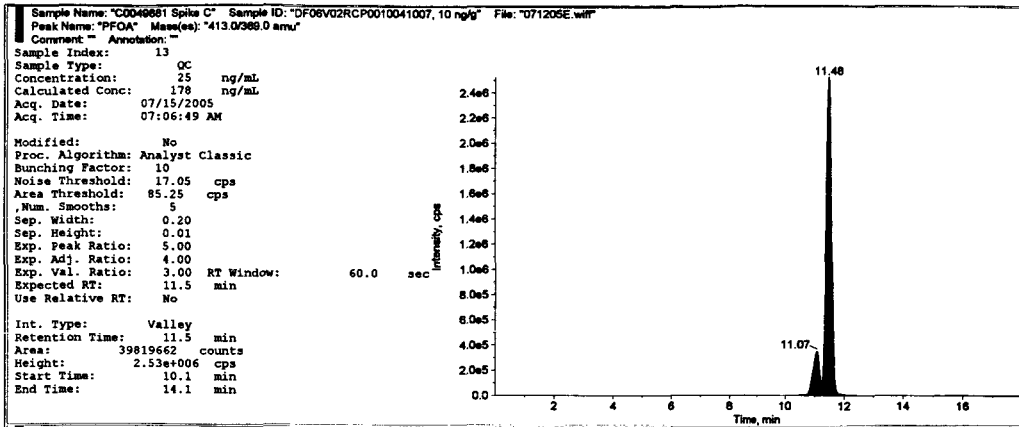
Initials AMS
 Date 07/21/05
 Run# 1 To 38
 Sample Index AMS
07/21/05

Verified by: DR
 Initials: DR Date 09/22/05
 Sample Index 1 To: 38

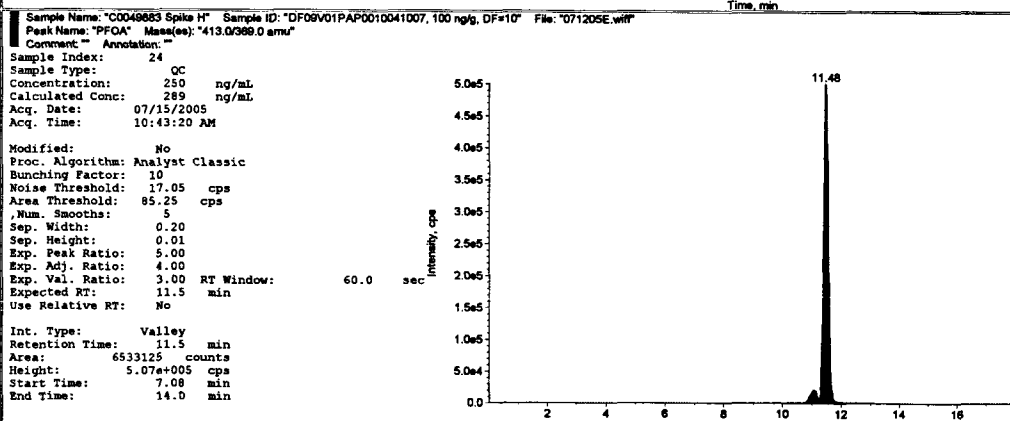
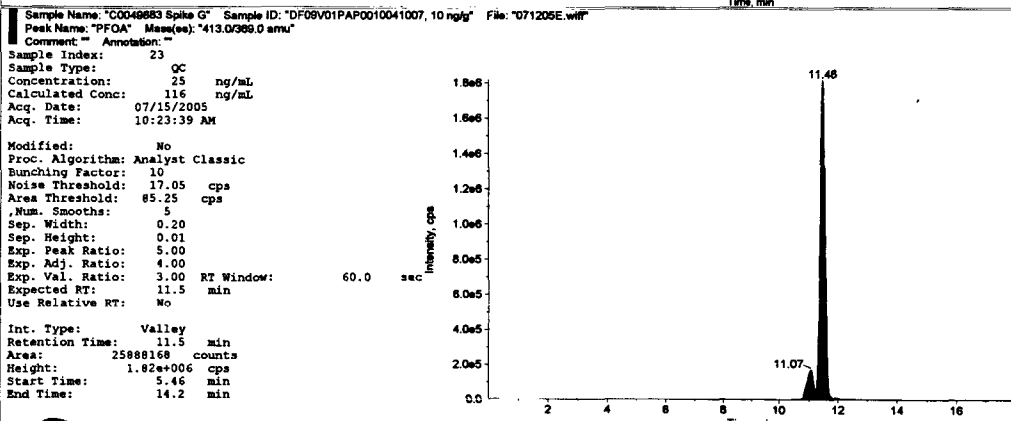
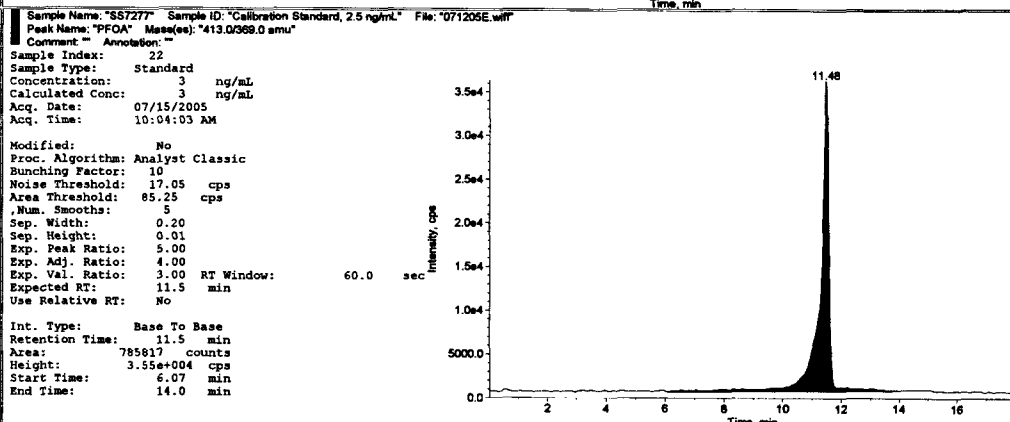
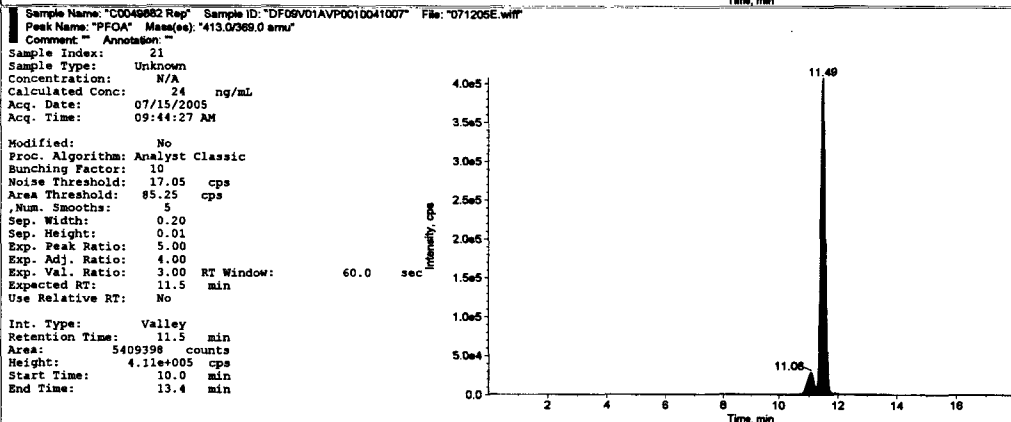
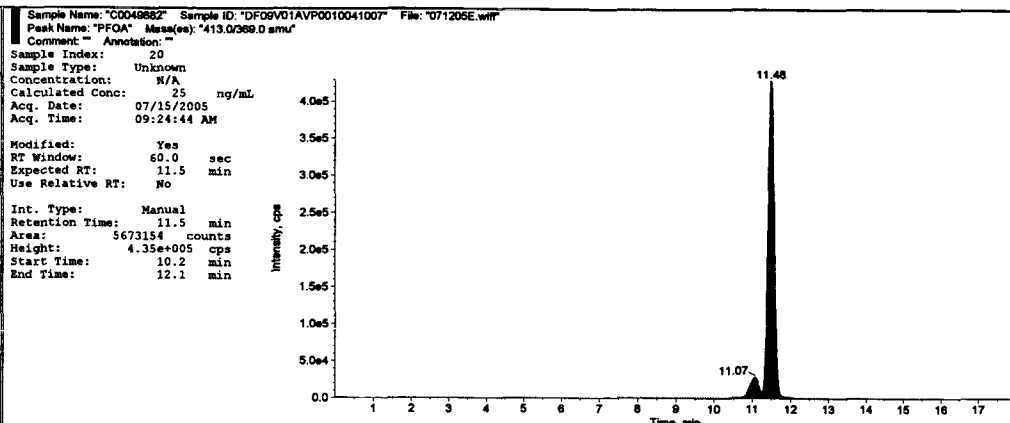
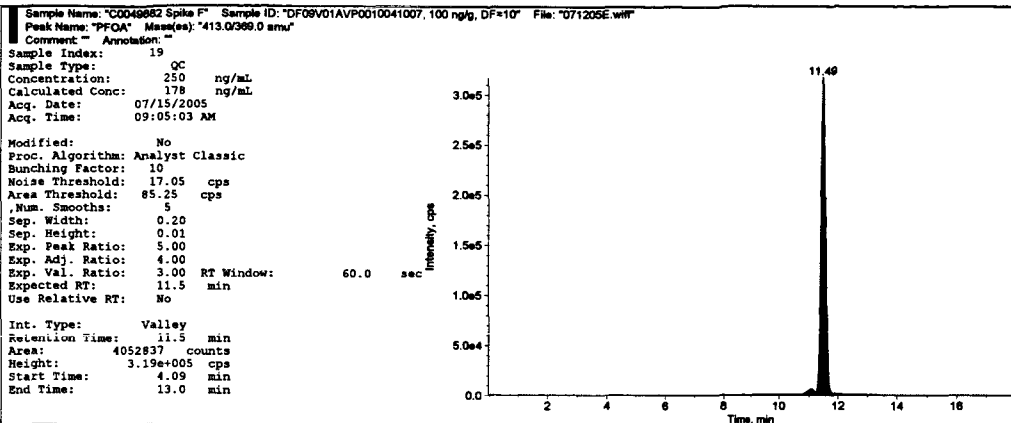


① (RE) 73483 BAK 01/20/05

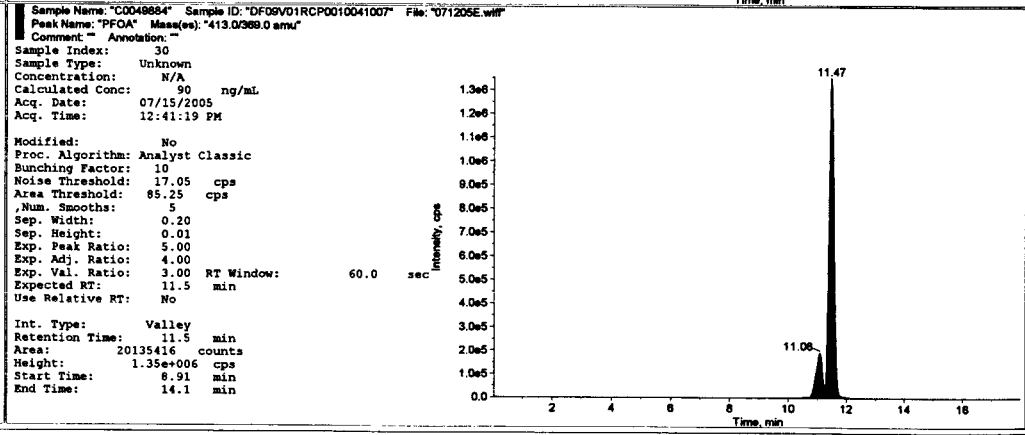
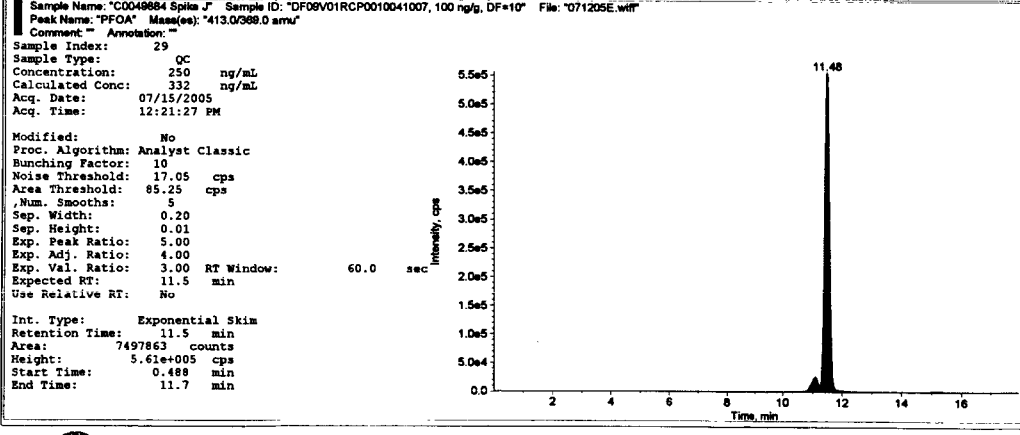
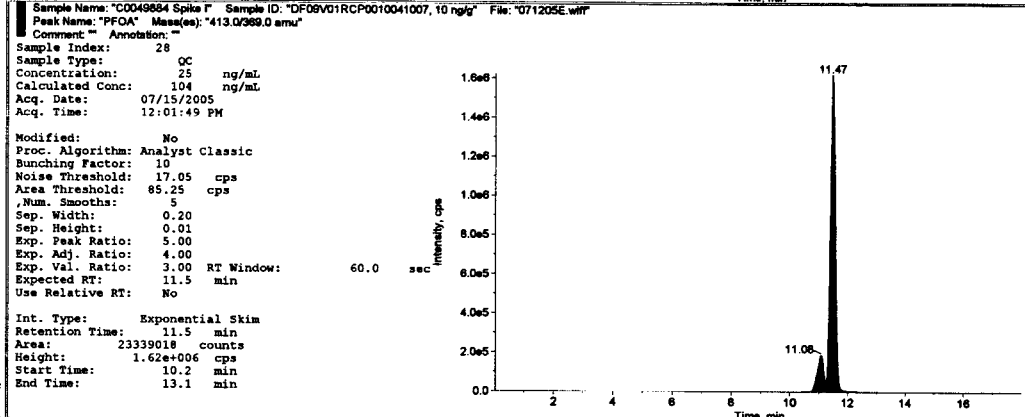
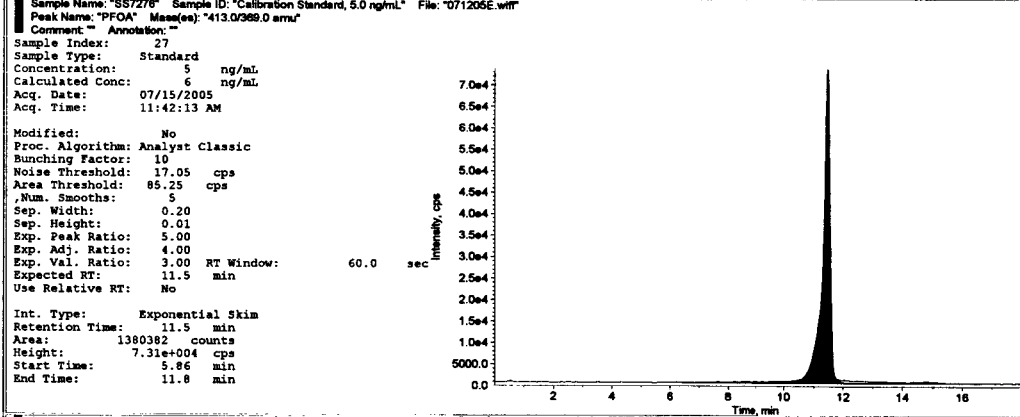
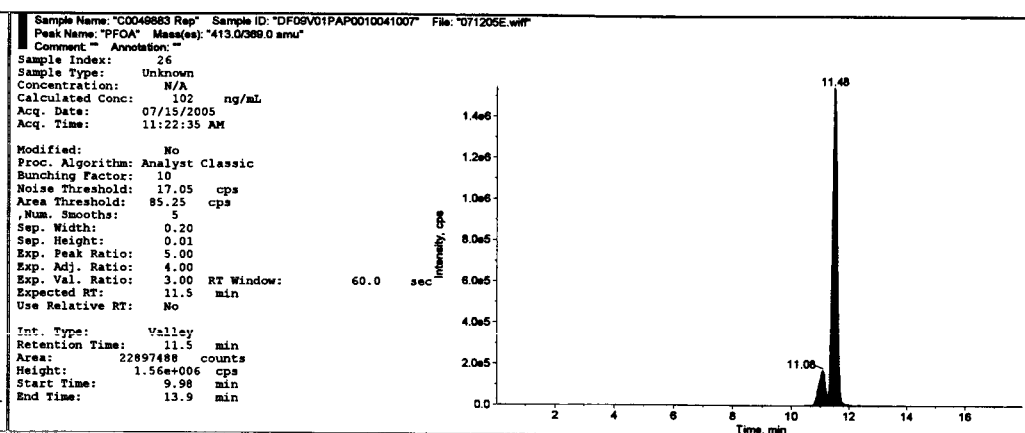
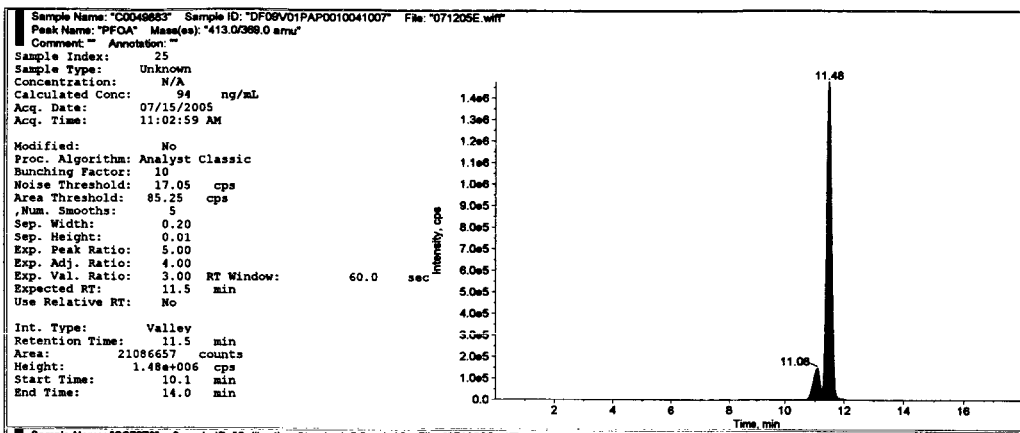
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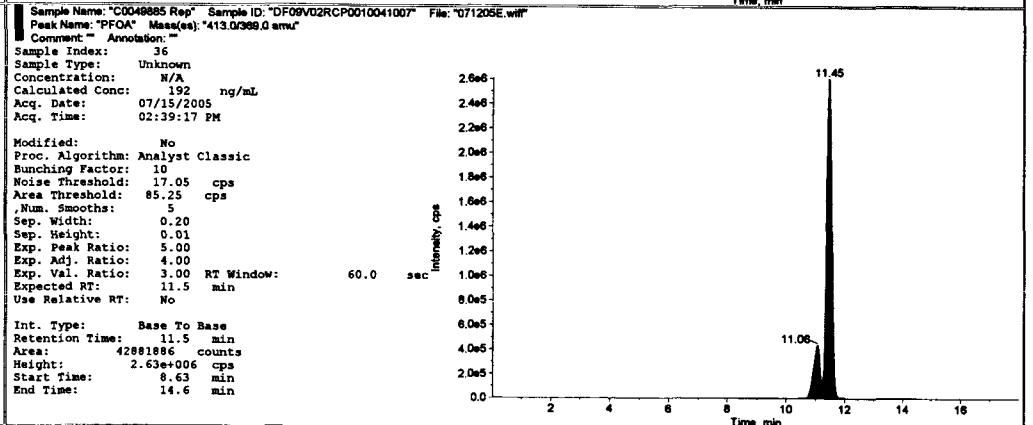
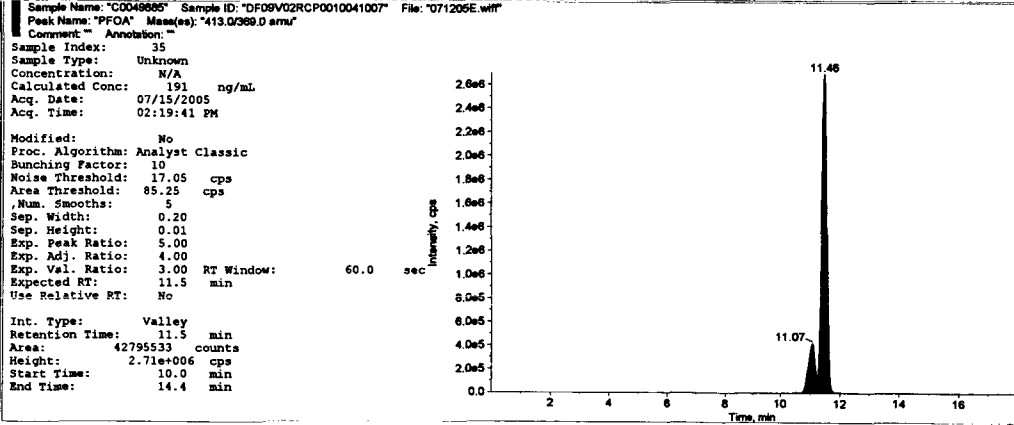
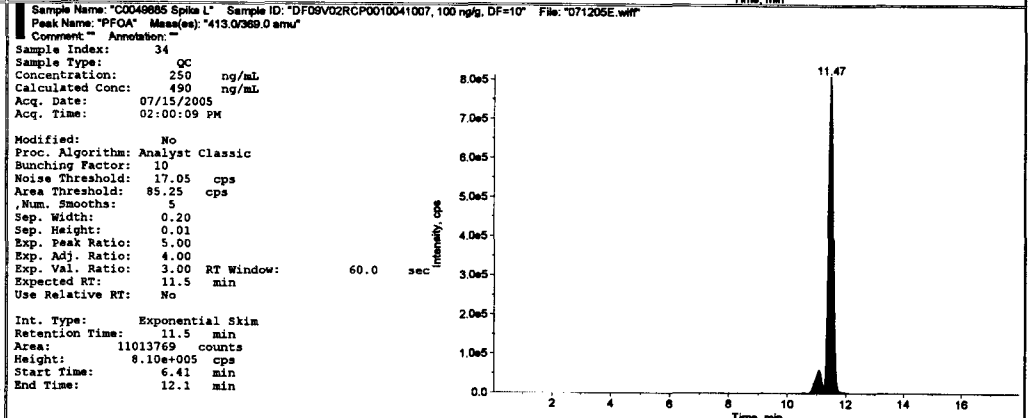
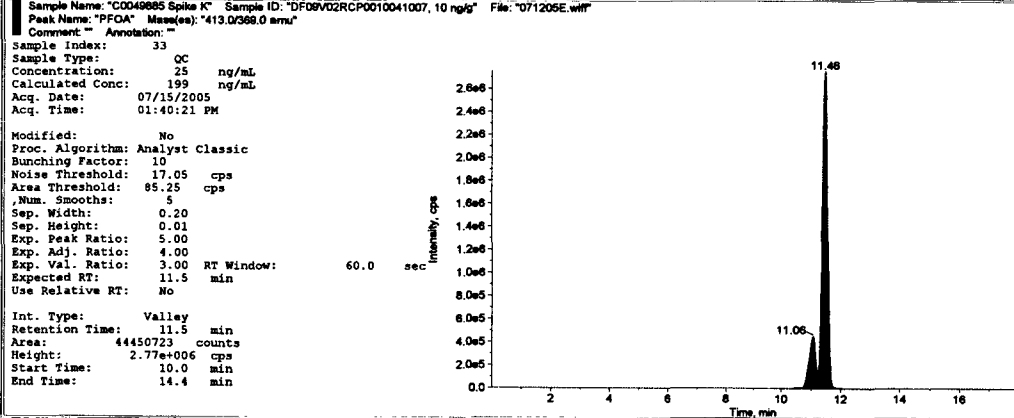
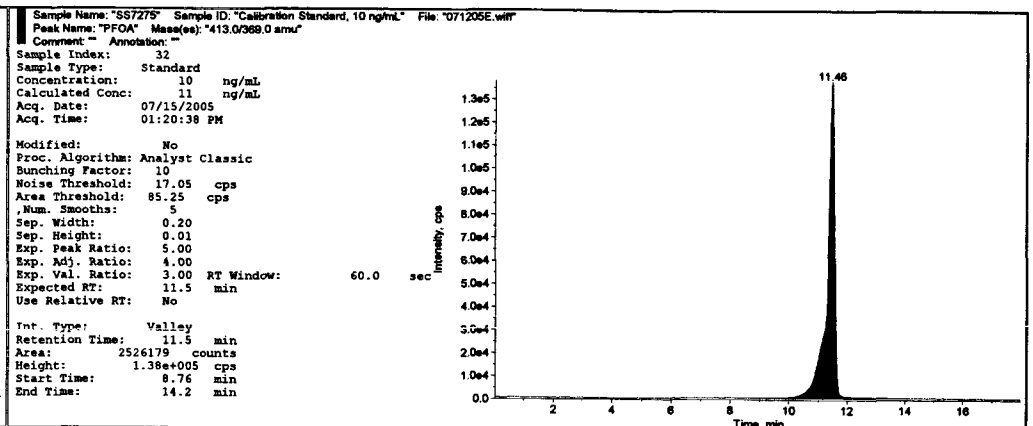
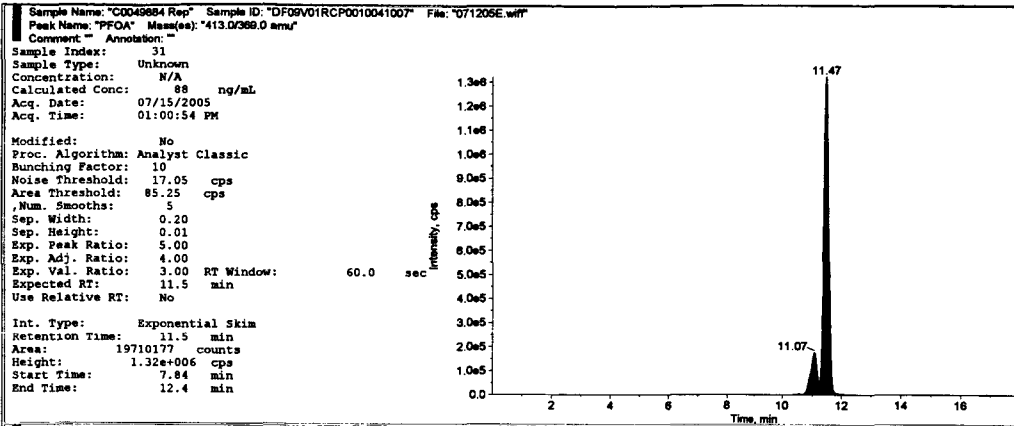
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0337



0338



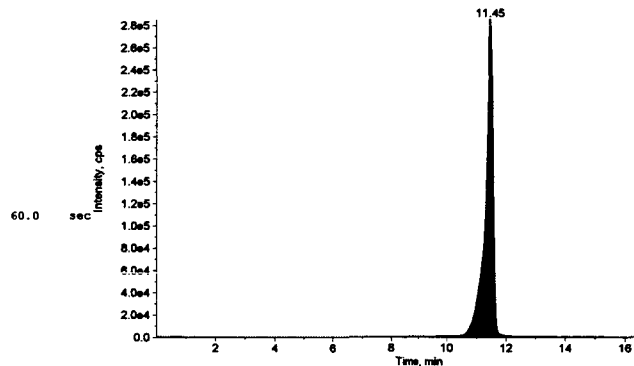
0339

Sample Name: "SS7274" Sample ID: "Calibration Standard, 25 ng/mL" File: "071205E.wiff"
Peak Name: "PFOA" Mass(es): "413.0/399.0 amu"

Comment: " Annotation: "
Sample Index: 37
Sample Type: Standard
Concentration: 25 ng/mL
Calculated Conc: 24 ng/mL
Acq. Date: 07/15/2005
Acq. Time: 02:58:54 PM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No

Int. Type: Exponential Skim
Retention Time: 11.5 min
Area: 5413773 counts
Height: 2.87e+005 cps
Start Time: 7.29 min
End Time: 12.1 min

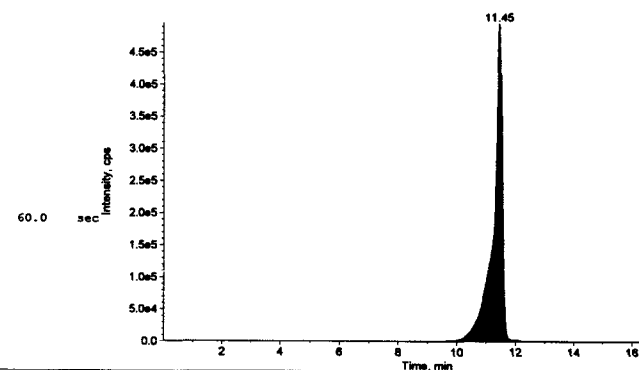


Sample Name: "SS7273" Sample ID: "Calibration Standard, 50 ng/mL" File: "071205E.wiff"
Peak Name: "PFOA" Mass(es): "413.0/399.0 amu"

Comment: " Annotation: "
Sample Index: 38
Sample Type: Standard
Concentration: 50 ng/mL
Calculated Conc: 48 ng/mL
Acq. Date: 07/15/2005
Acq. Time: 03:18:32 PM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 11.5 min
Area: 10827940 counts
Height: 4.99e+005 cps
Start Time: 6.44 min
End Time: 13.8 min



CEE 07/28/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5719	0.500	0.397	071205E-201
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	7564	1.00	0.686	071205E-202
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	17604	2.50	2.26	071205E-203
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	37689	5.00	5.41	071205E-204
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	68791	10.0	10.3	071205E-205
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	162804	25.0	25.0	071205E-206
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	315995	50.0	49.0	071205E-207
Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	071205E-208
Reagent Control	C0073483 Control	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	071205E-209
Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFOA Confirm Ion	42814	6.25	6.21	071205E-210
Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFOA Confirm Ion	177233	25.0	27.3	071205E-211
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	8151	0.500	0.778	071205E-212
C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	3135610	① 25.0	491.	071205E-213
C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	480450	250.	748.	071205E-214
C0049881	DF06V02RCP0010041007	Unknown	1	PFOA Confirm Ion	3056844	① N/A	479.	071205E-215
C0049881 Rep	DF06V02RCP0010041007	Unknown	1	PFOA Confirm Ion	3135037	① N/A	491.	071205E-216
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	8682	1.00	0.861	071205E-217
C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	419448	25.0	65.3	071205E-218
C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	131309	250.	201.	071205E-219
C0049882	DF09V01AVP0010041007	Unknown	1	PFOA Confirm Ion	315718	N/A	49.0	071205E-220
C0049882 Rep	DF09V01AVP0010041007	Unknown	1	PFOA Confirm Ion	321538	N/A	49.9	071205E-221
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	18456	2.50	2.39	071205E-222
C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	1598291	① 25.0	250.	071205E-223
C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	279934	250.	434.	071205E-224
C0049883	DF09V01PAP0010041007	Unknown	1	PFOA Confirm Ion	1346710	① N/A	211.	071205E-225
C0049883 Rep	DF09V01PAP0010041007	Unknown	1	PFOA Confirm Ion	1569130	① N/A	246.	071205E-226
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	36050	5.00	5.15	071205E-227
C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	1527361	① 25.0	239.	071205E-228
C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	308840	250.	479.	071205E-229
C0049884	DF09V01RCP0010041007	Unknown	1	PFOA Confirm Ion	1437143	① N/A	225.	071205E-230
C0049884 Rep	DF09V01RCP0010041007	Unknown	1	PFOA Confirm Ion	1388450	① N/A	217.	071205E-231
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	74614	10.0	11.2	071205E-232
C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	3477573	① 25.0	545.	071205E-233
C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	555392	① 250.	866.	071205E-234
C0049885	DF09V02RCP0010041007	Unknown	1	PFOA Confirm Ion	3335002	① N/A	522.	071205E-235
C0049885 Rep	DF09V02RCP0010041007	Unknown	1	PFOA Confirm Ion	3325777	① N/A	521.	071205E-236
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	165753	25.0	25.5	071205E-237
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	315862	50.0	49.0	071205E-238

① Requires dilution, see data set 071205ER. BAK 07/28/05

Fish Conversion

Compound: PFOA Confirm. Ion

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049881 Spk D	DF06-V02-RCP001-0-041007	748	299
C0049882	DF09-V01-AVP001-0-041007	49.0	19.6
C0049882 Rep	DF09-V01-AVP001-0-041007	49.9	20.0
C0049882 Spk E	DF09-V01-AVP001-0-041007	65.3	26.1
C0049882 Spk F	DF09-V01-AVP001-0-041007	201	80.4
C0049883 Spk H	DF09-V01-PAP001-0-041007	434	174
C0049884 Spk J	DF09-V01-RCP001-0-041007	479	192

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = ~~Peak Area 0~~ AT OR ABOVE 0.2 ng/g ①

NQ = Not quantifiable = Measured concentration below Limit of Quantitation (LOQ) which is 0.7 ng/g.

↑
BETWEEN 0.2 ng/g AND ①

40

① RE MLC 9/20/05

0342

MLC 07/20/05

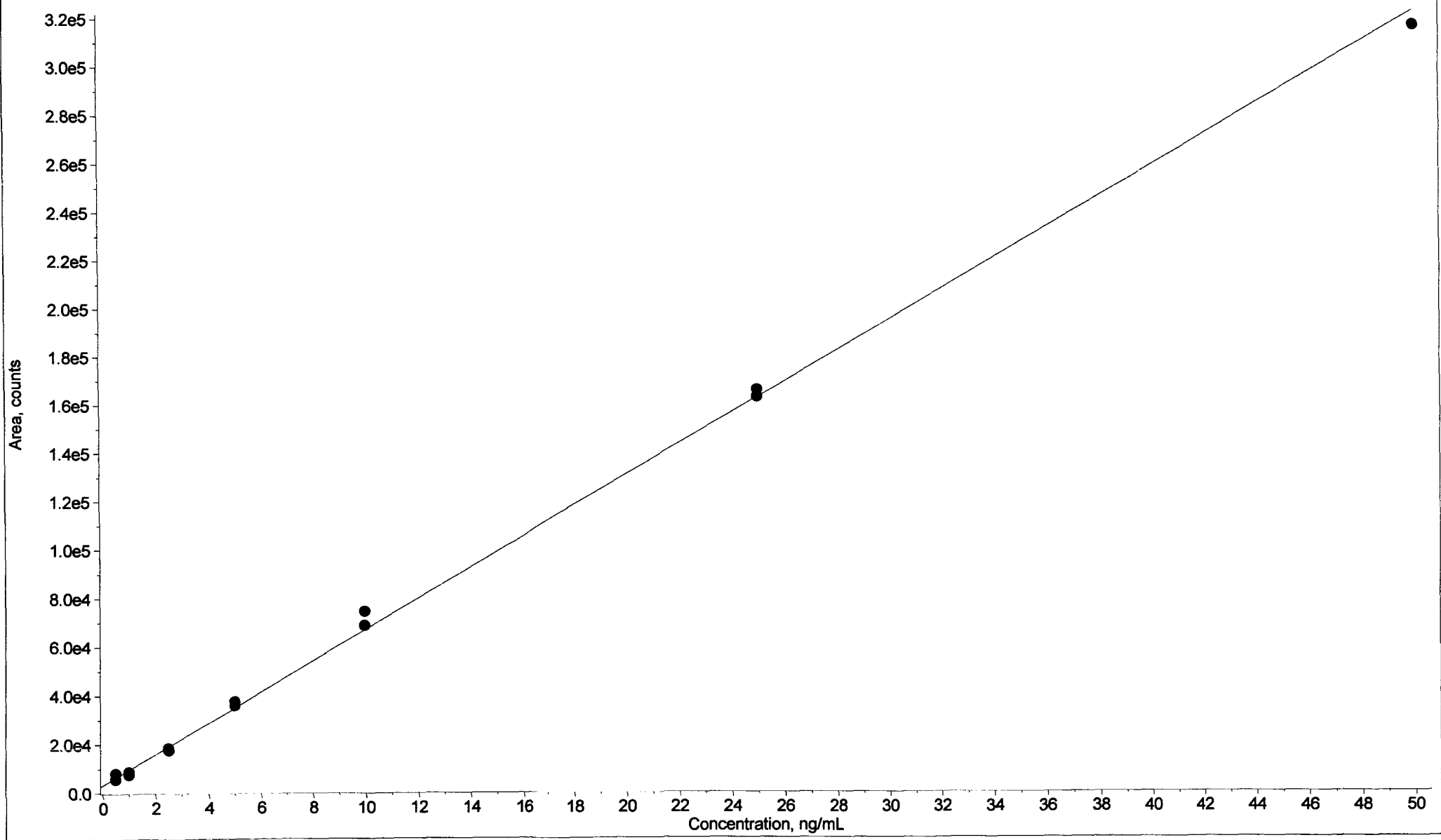
	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound:	PFOA Confirm. Ion	Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049881 Spk D	DF06-V02-RCP001-0-041007	=Raw Data!H184	=(C9*2)/5
10				
11	C0049882	DF09-V01-AVP001-0-041007	=Raw Data!H190	=(C11*2)/5
12	C0049882 Rep	DF09-V01-AVP001-0-041007	=Raw Data!H191	=(C12*2)/5
13	C0049882 Spk E	DF09-V01-AVP001-0-041007	=Raw Data!H188	=(C13*2)/5
14	C0049882 Spk F	DF09-V01-AVP001-0-041007	=Raw Data!H189	=(C14*2)/5
15				
16	C0049883 Spk H	DF09-V01-PAP001-0-041007	=Raw Data!H194	=(C16*2)/5
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18	C0049884 Spk J	DF09-V01-RCP001-0-041007	=Raw Data!H199	=(C18*2)/5
19				

0343

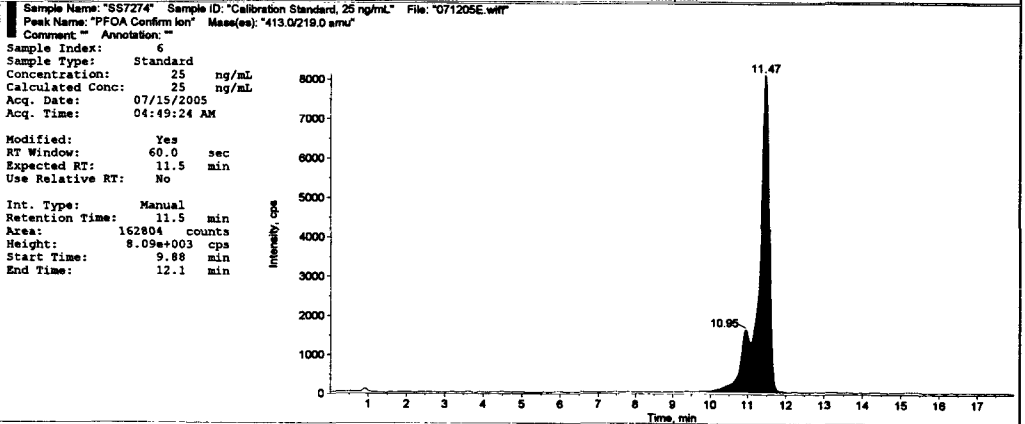
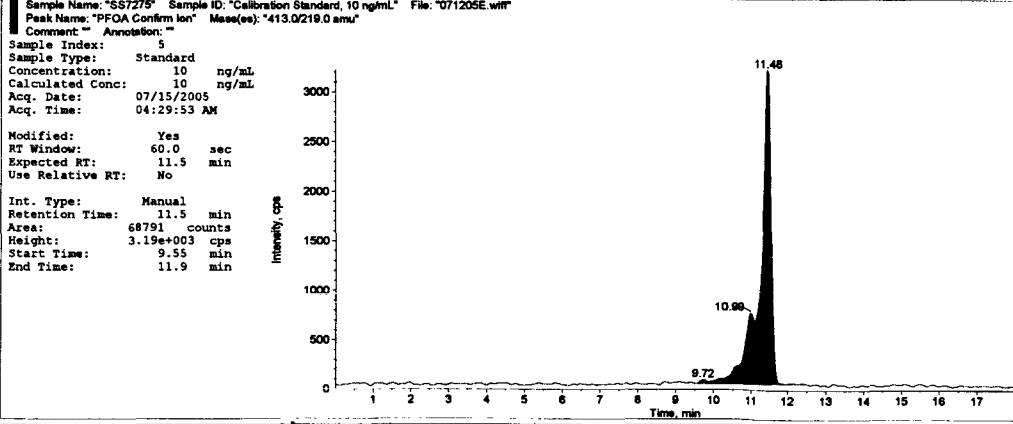
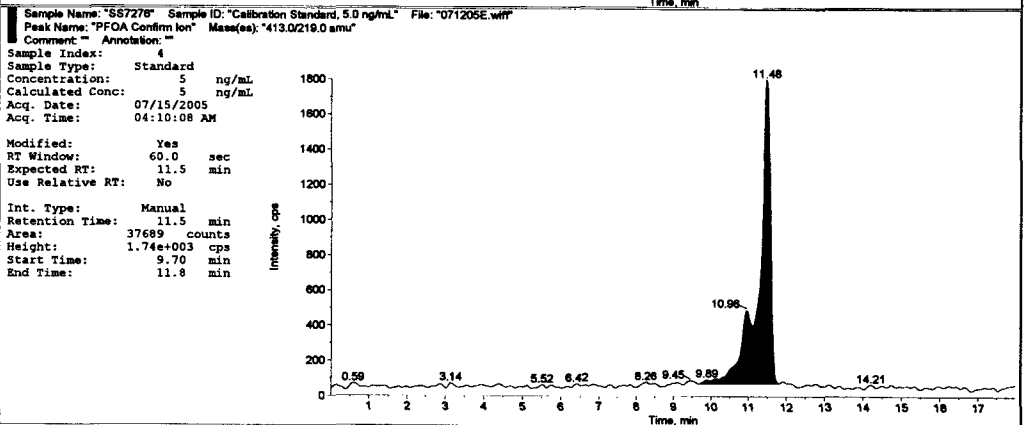
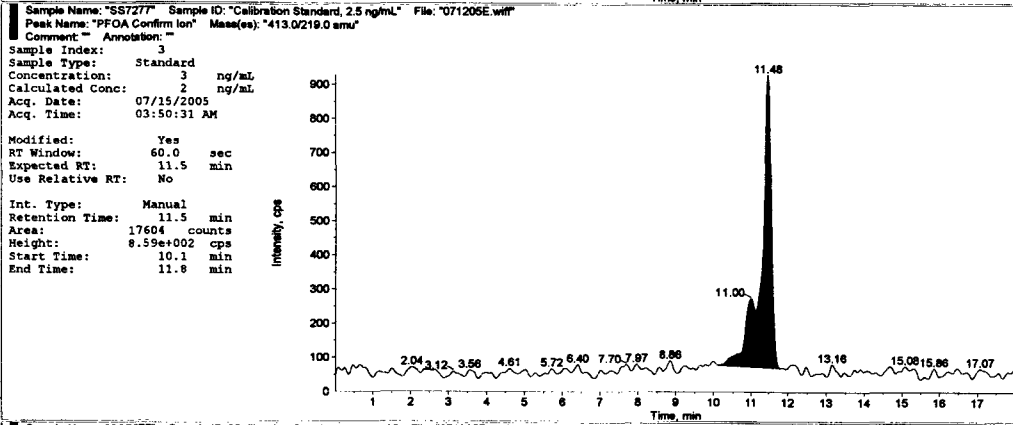
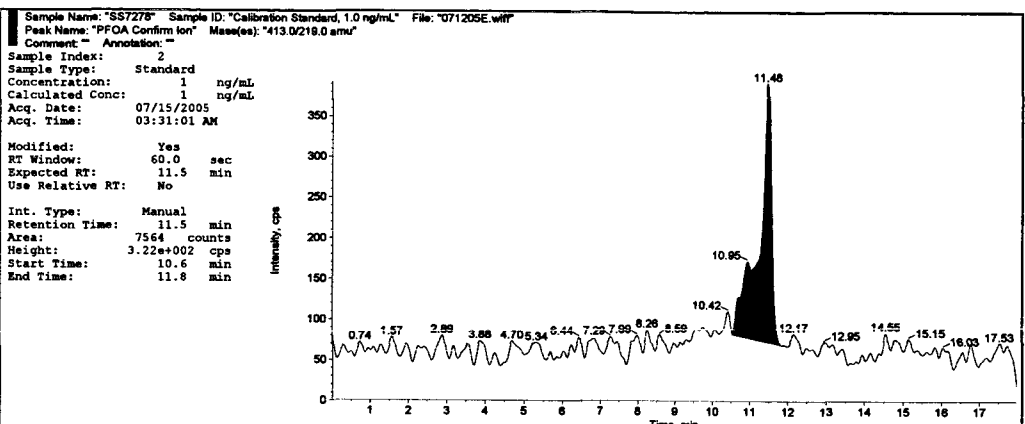
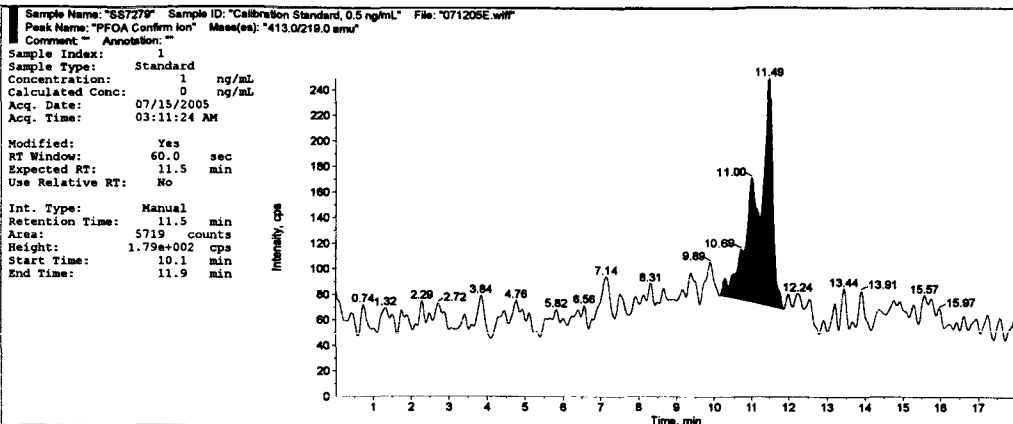
BAK 05/28/05

AMB 07/21/05

■ Untitled 6 (PFOA Confirm Ion): "Linear" Regression ("1 / x" weighting): $y = 6.38e+003 x + 3.19e+003$ ($r = 0.9983$)

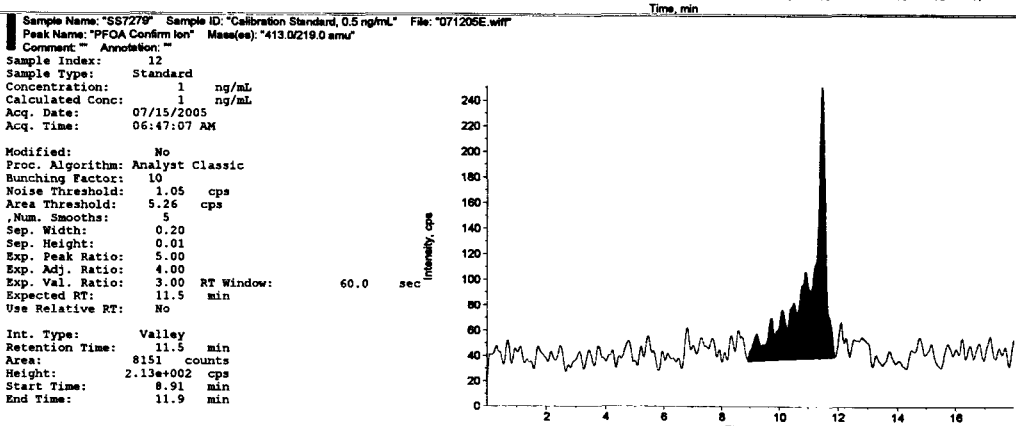
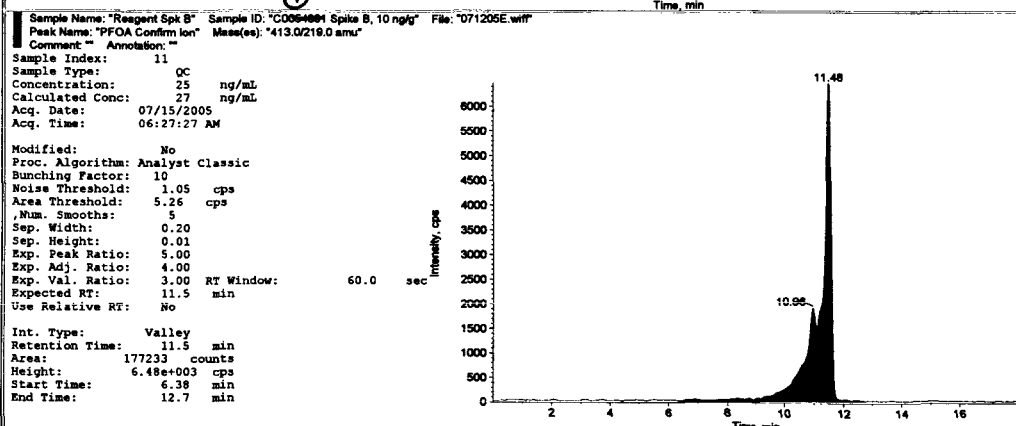
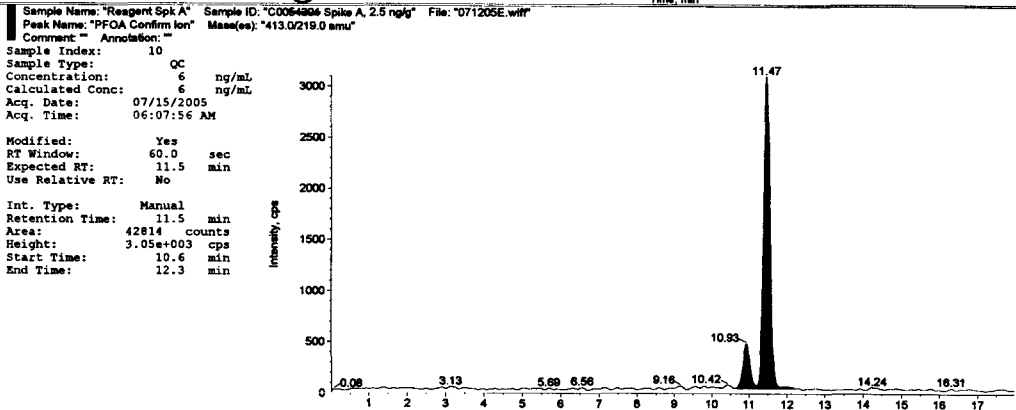
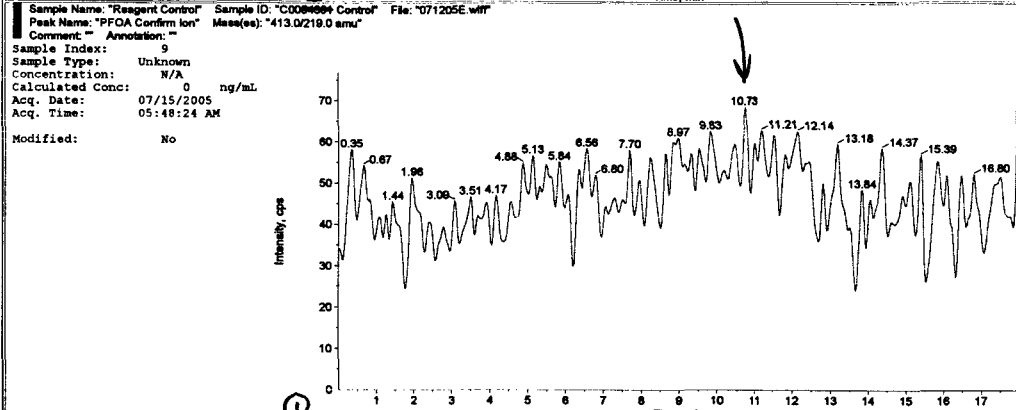
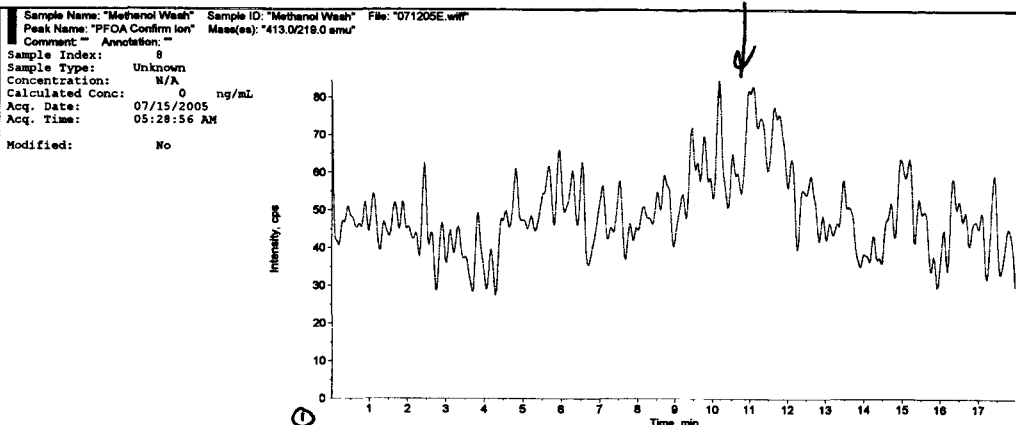
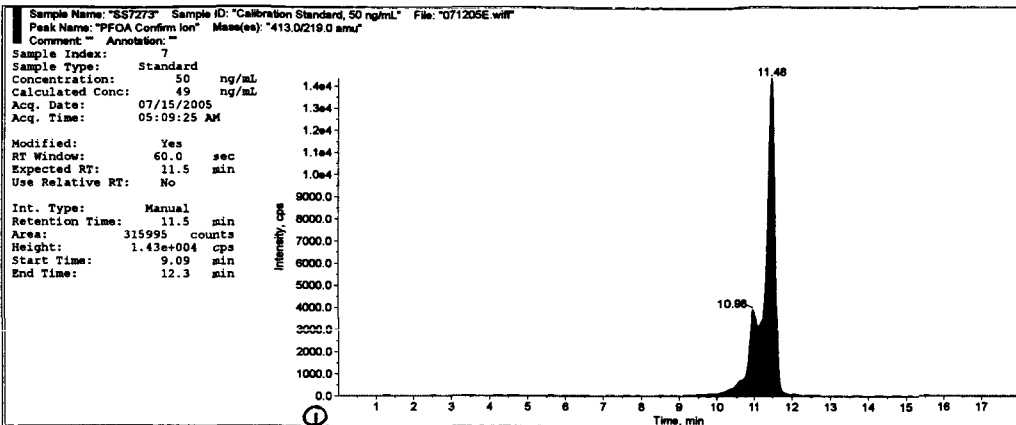


0344

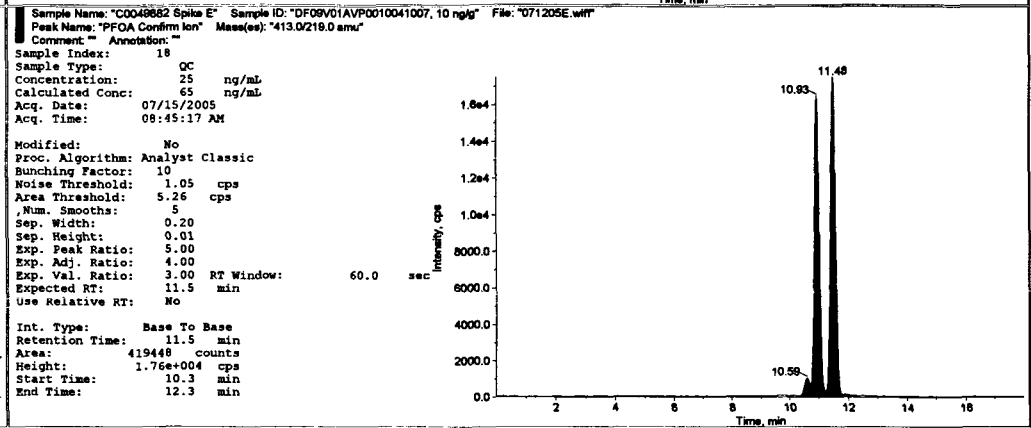
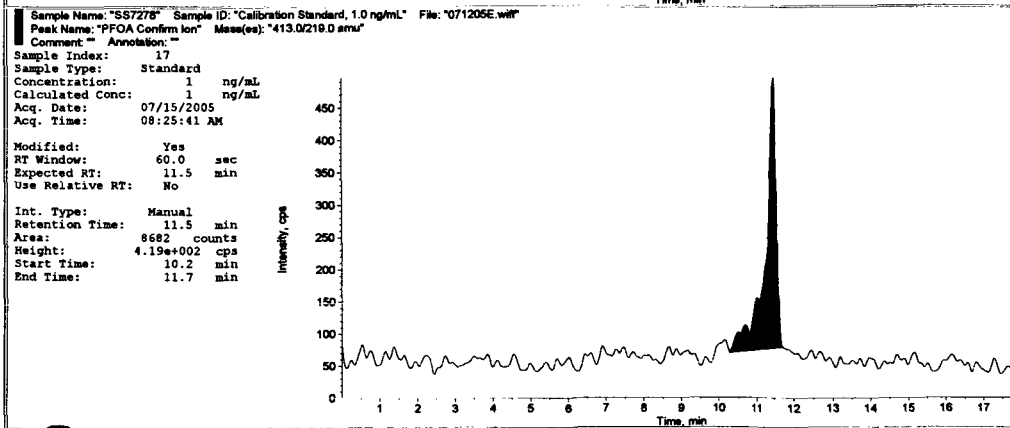
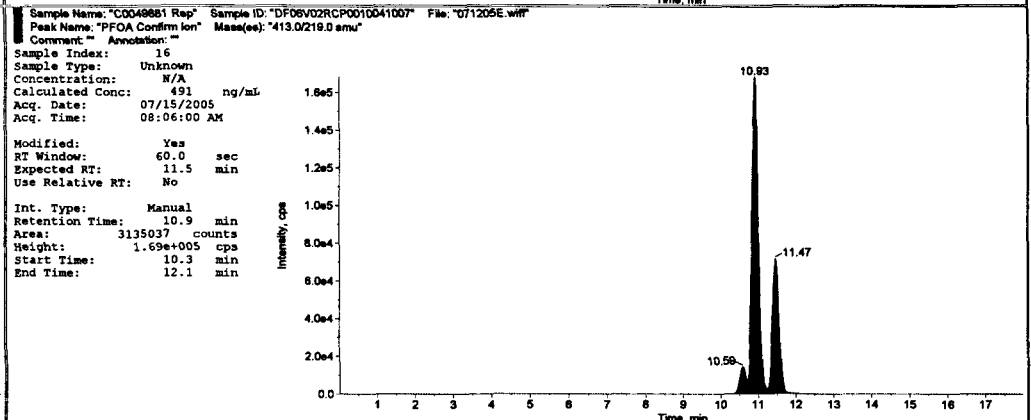
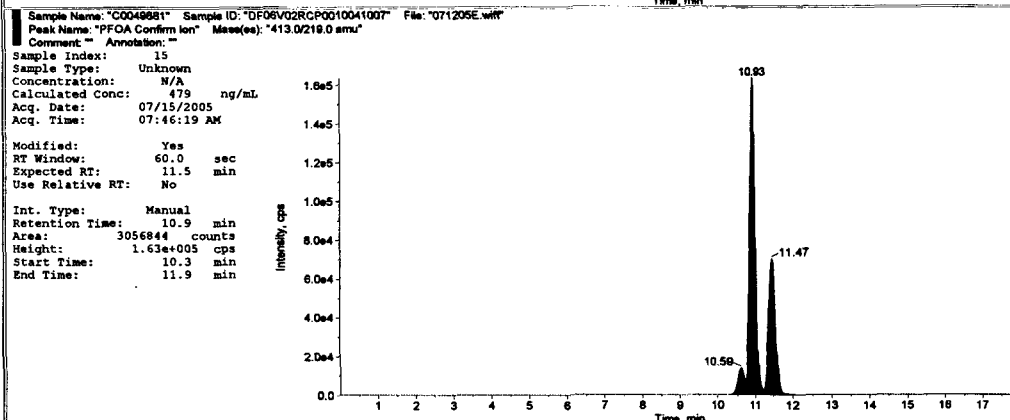
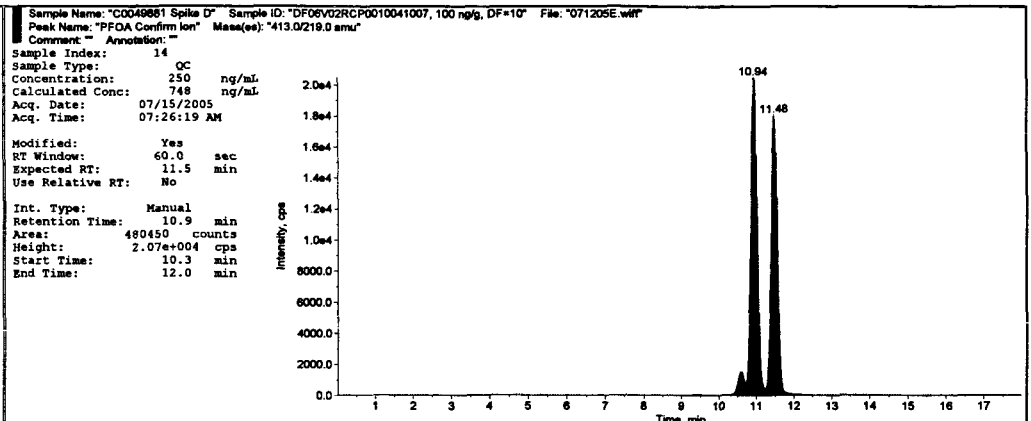
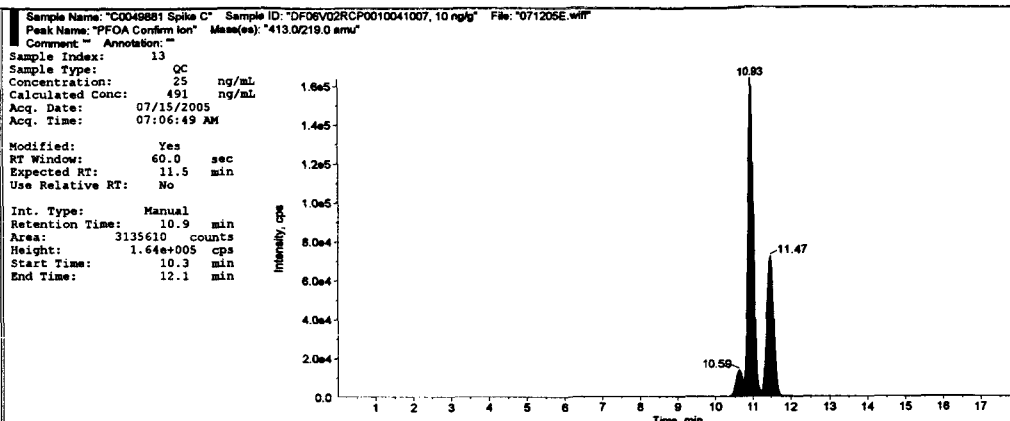


0345
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 Date 07/21/05
 Run# 1 To 38
 Sample Index @AMB 07/21/05

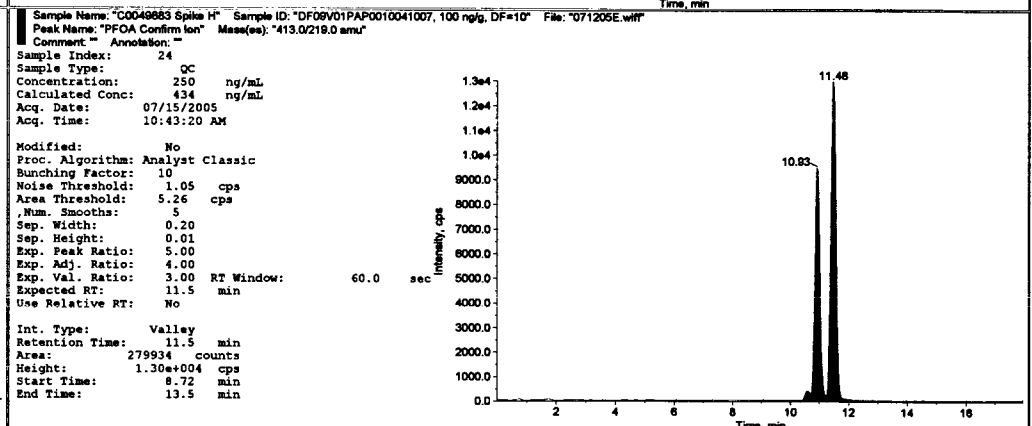
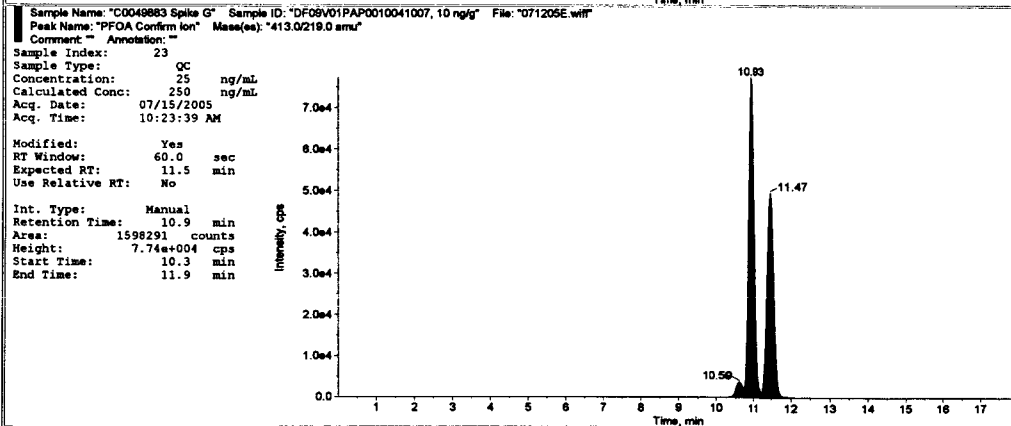
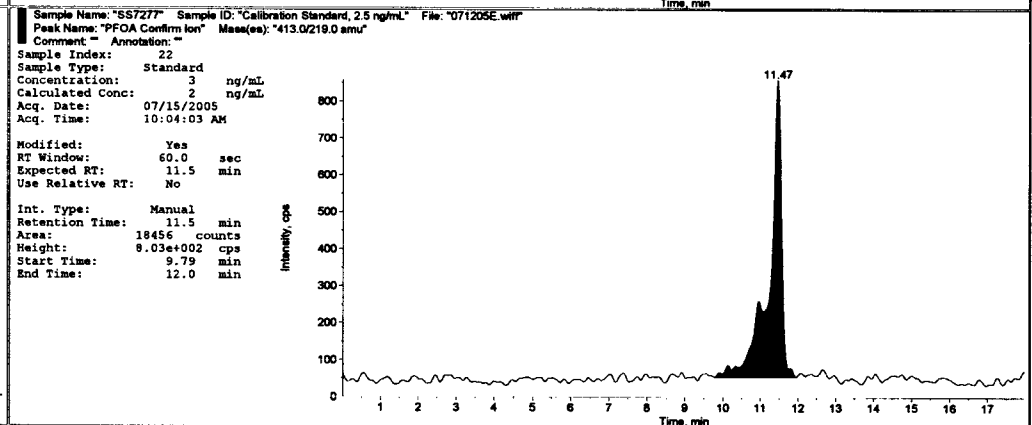
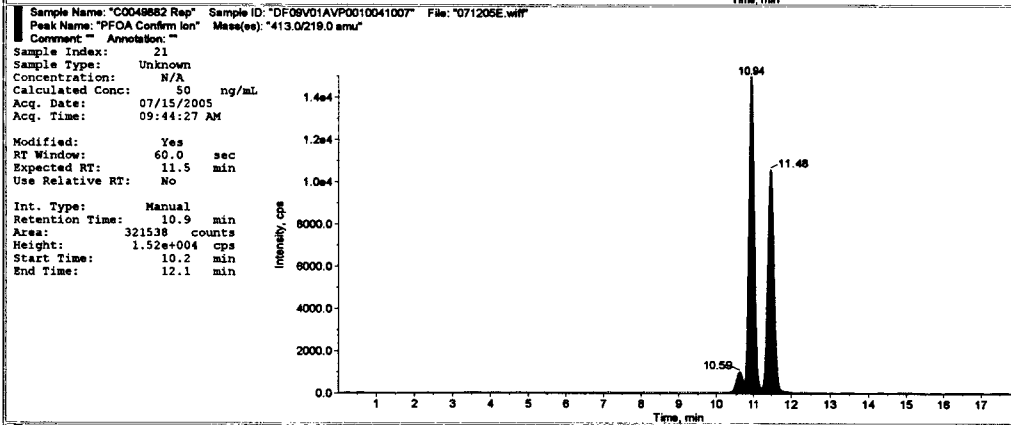
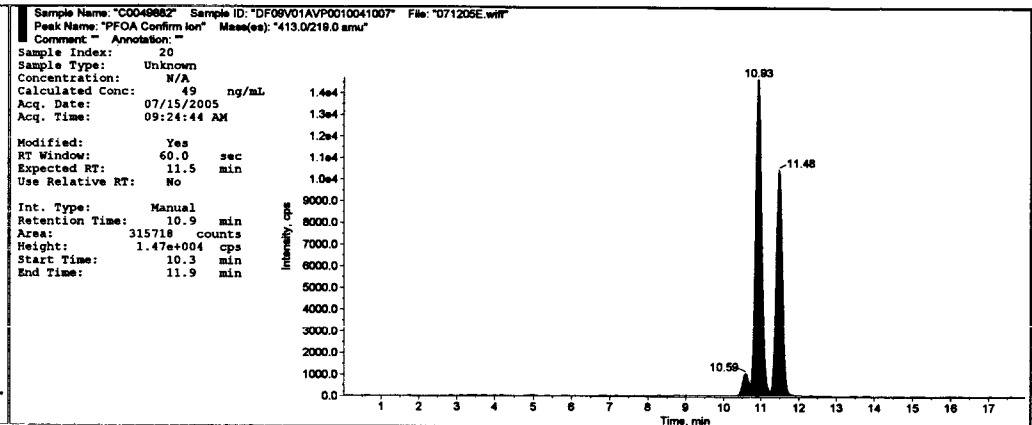
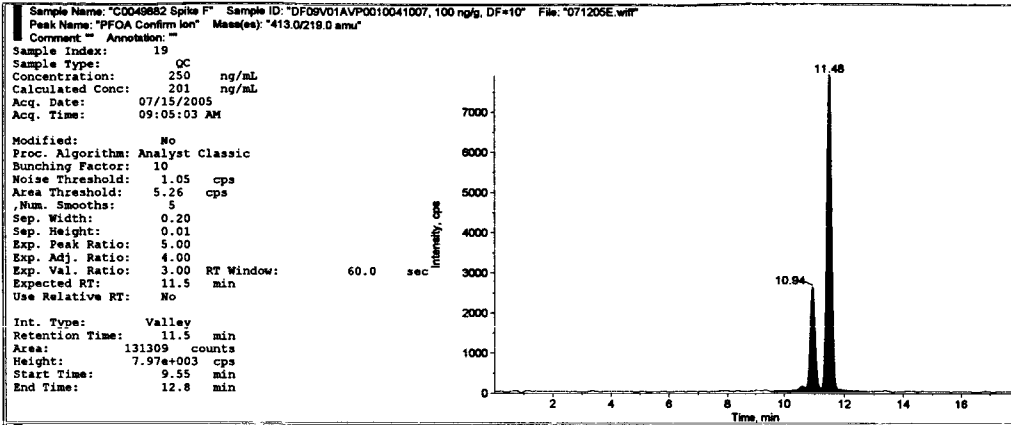
Verified by:
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 Sample Index: 1 To: 38

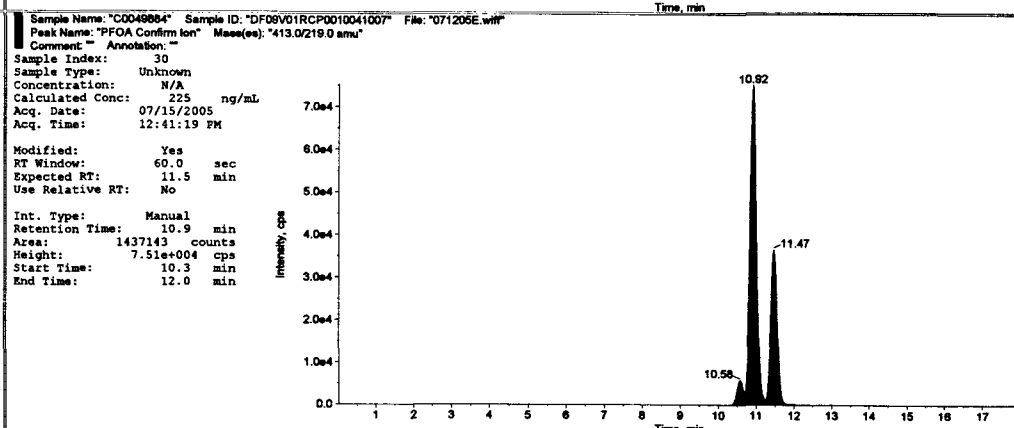
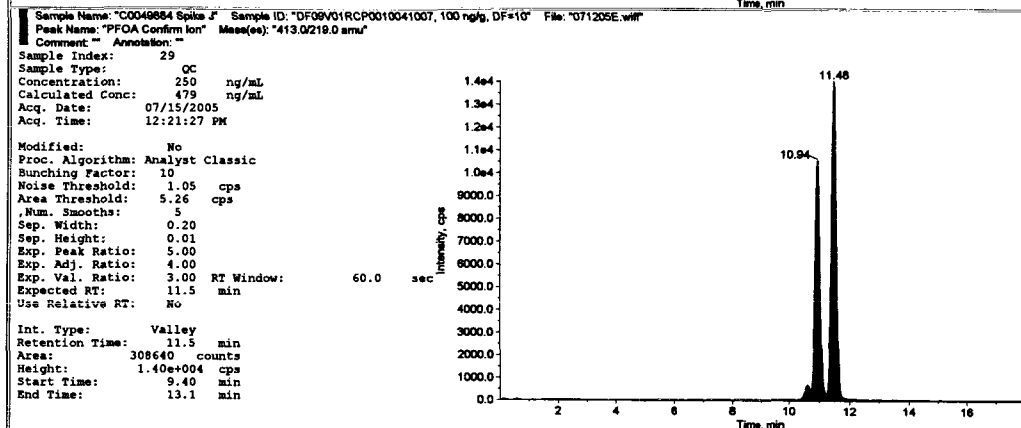
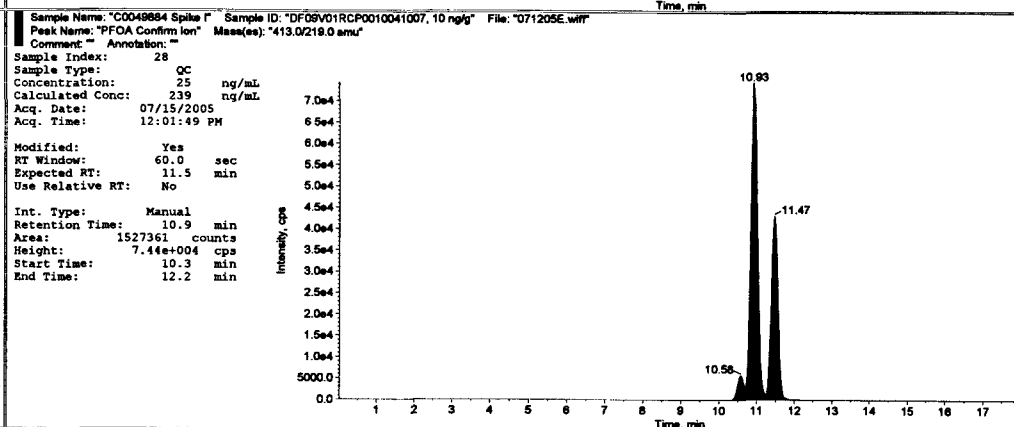
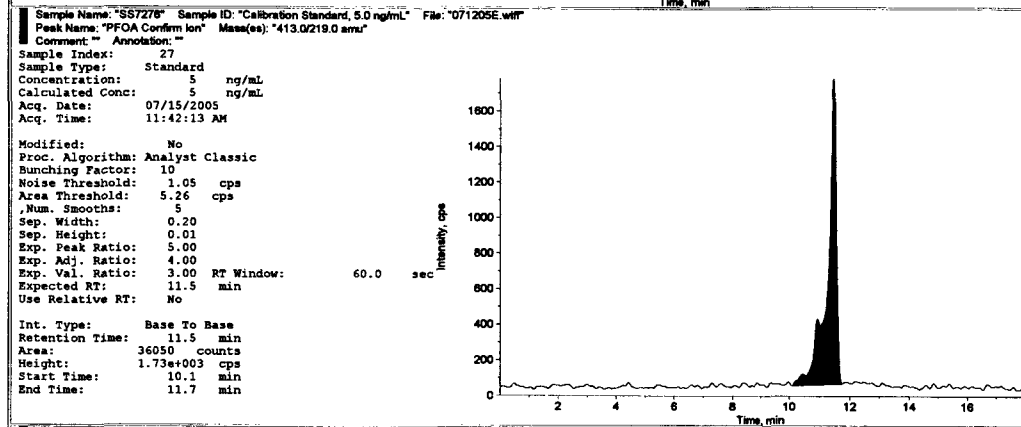
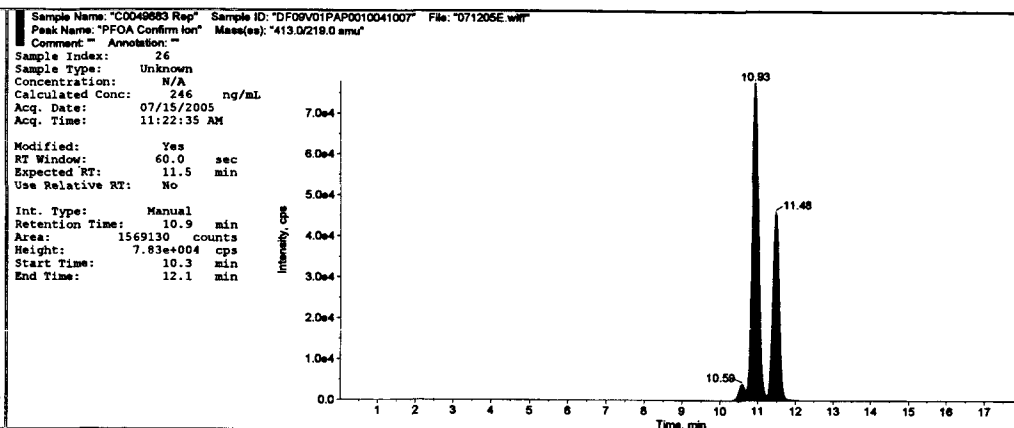
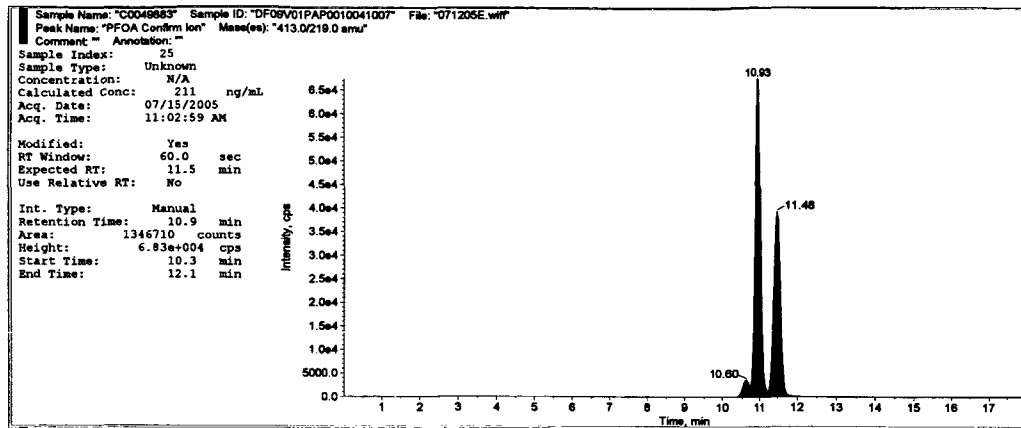


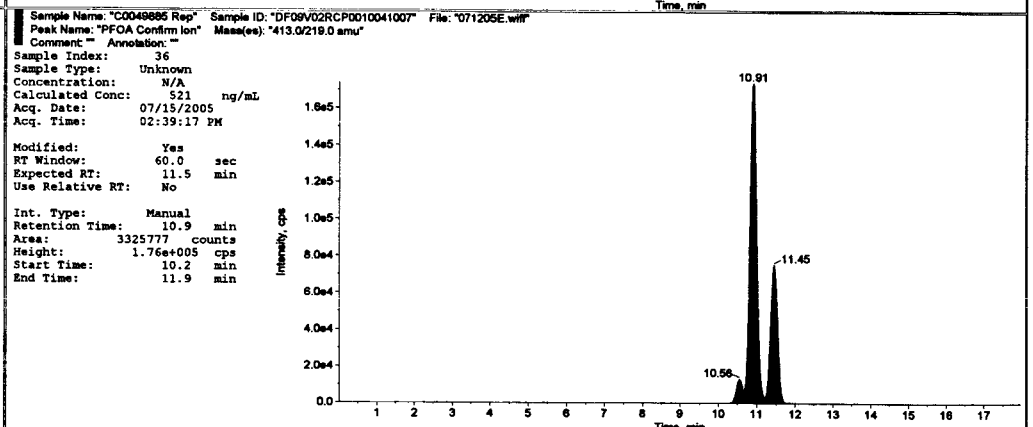
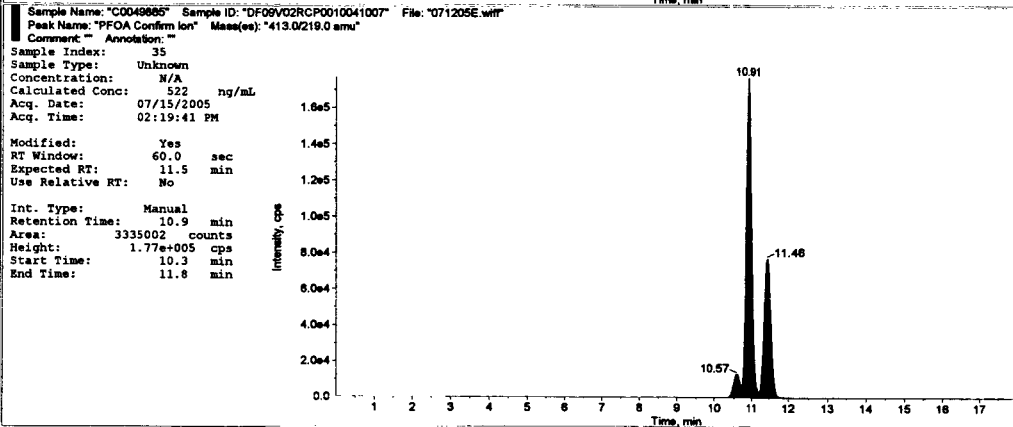
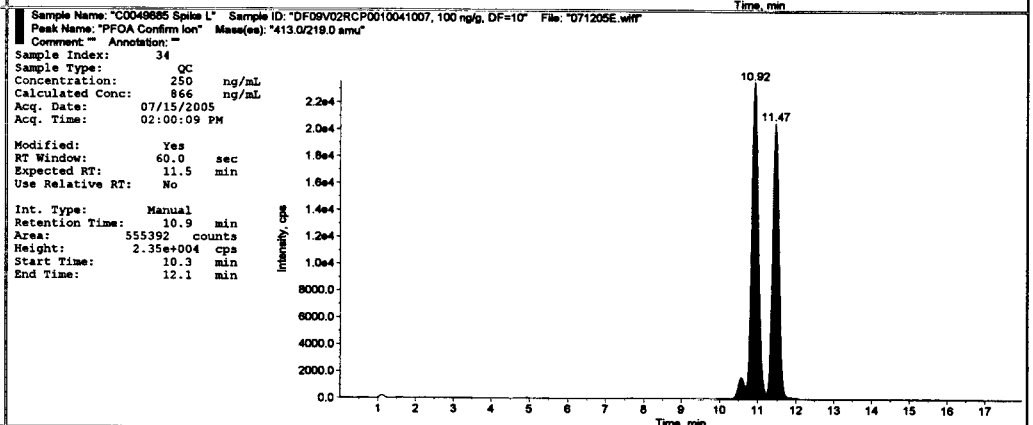
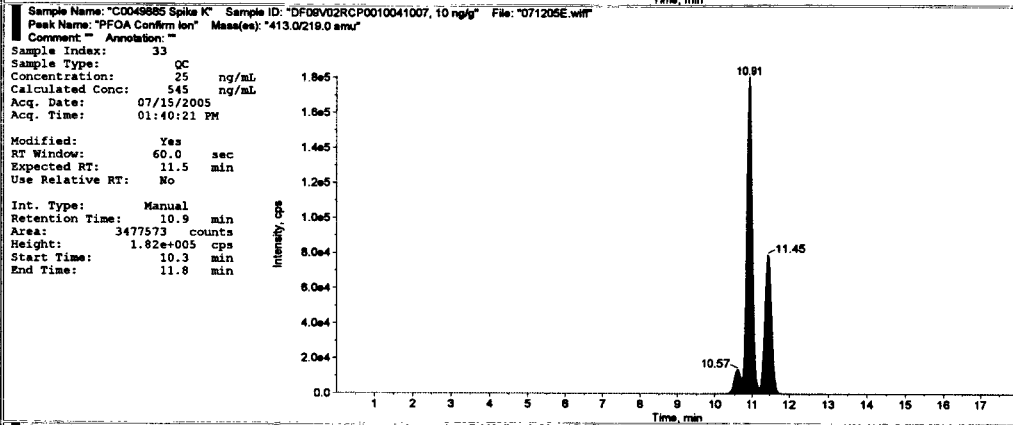
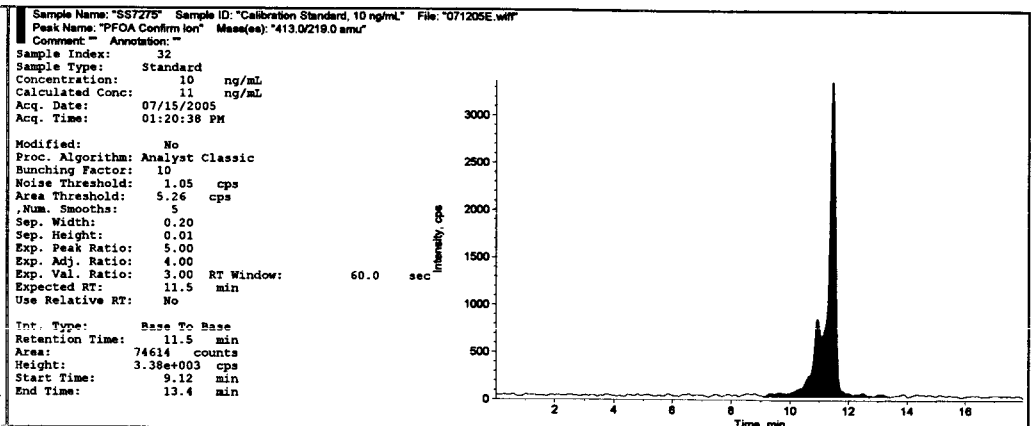
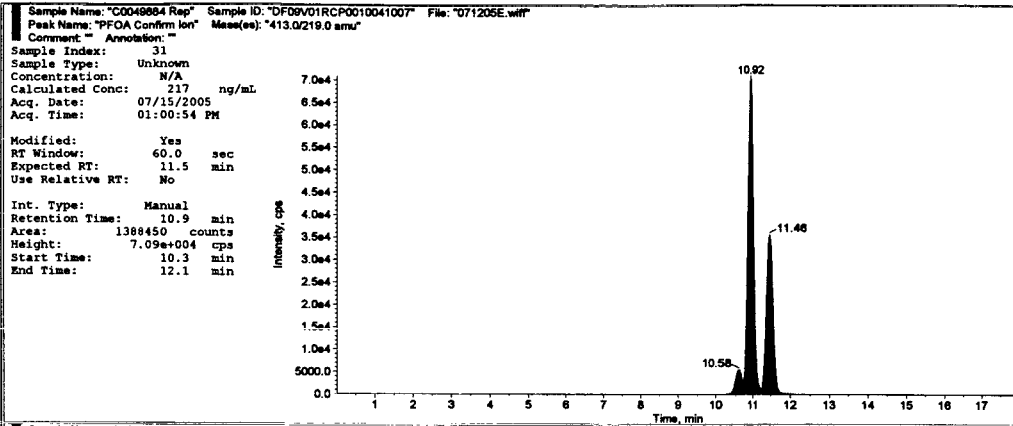
RE 73483 BAK 09/20/05
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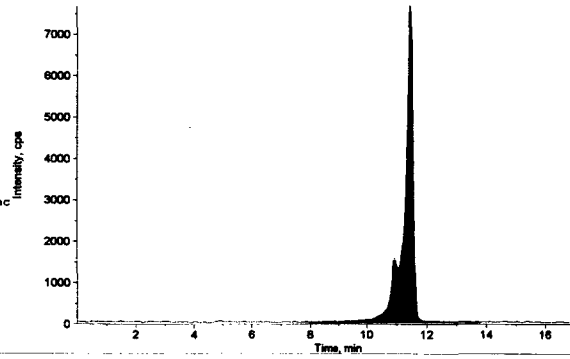


Sample Name: "S87274" Sample ID: "Calibration Standard, 25 ng/mL" File: "071205E.wif"
Peak Name: "PFOA Confirm Ion" Mass(es): "413.0/219.0 amu"

Comment: "Annotation:"
Sample Index: 37
Sample Type: Standard
Concentration: 25 ng/mL
Calculated Conc: 25 ng/mL
Acq. Date: 07/15/2005
Acq. Time: 02:58:54 PM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 1.05 cps
Area Threshold: 5.26 cps
, Num. Smooths: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window: 60.0 sec
Expected RT: 11.5 min
Use Relative RT: No

Int. Type: Valley
Retention Time: 11.4 min
Area: 165753 counts
Height: 7.65e+003 cps
Start Time: 7.60 min
End Time: 13.8 min

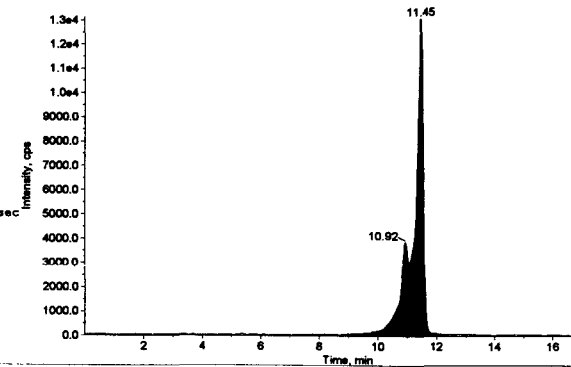


Sample Name: "S87273" Sample ID: "Calibration Standard, 50 ng/mL" File: "071205E.wif"
Peak Name: "PFOA Confirm Ion" Mass(es): "413.0/219.0 amu"

Comment: "Annotation:"
Sample Index: 38
Sample Type: Standard
Concentration: 50 ng/mL
Calculated Conc: 49 ng/mL
Acq. Date: 07/15/2005
Acq. Time: 03:18:32 PM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 1.05 cps
Area Threshold: 5.26 cps
, Num. Smooths: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window: 60.0 sec
Expected RT: 11.5 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 11.4 min
Area: 315862 counts
Height: 1.31e+004 cps
Start Time: 7.08 min
End Time: 12.4 min



CEE 07/28/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
1 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	178460	0.500	0.421	071205E-201
2 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	285950	1.00	0.993	071205E-202
3 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	615884	2.50	2.75	071205E-203
4 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1170985	5.00	5.70	071205E-204
5 SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2180974	10.0	11.1	071205E-205
6 SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	4902573	25.0	25.5	071205E-206
7 SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9251667	50.0	48.7	071205E-207
8 Methanol Wash	Methanol Wash	Unknown	1	13C PFOA	102138	N/A	0.0152	071205E-208
9 Reagent Control	C0073483 Control	Unknown	1	13C PFOA	83235	N/A	< 0	071205E-209
10 Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	13C PFOA	1408992	6.25	6.97	071205E-210
11 Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	13C PFOA	5192026	25.0	27.1	071205E-211
12 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	204486	0.500	0.560	071205E-212
13 C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2538404	25.0	13.0	071205E-213
14 C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3531422	250.	183.	071205E-214
15 C0049881	DF06V02RCP0010041007	Unknown	1	13C PFOA	2330899	N/A	11.9	071205E-215
16 C0049881 Rep	DF06V02RCP0010041007	Unknown	1	13C PFOA	2309360	N/A	11.8	071205E-216
17 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	231399	1.00	0.703	071205E-217
18 C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g	Quality Control	1	13C PFOA	3226572	25.0	16.6	071205E-218
19 C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2970587	250.	153.	071205E-219
20 C0049882	DF09V01AVP0010041007	Unknown	1	13C PFOA	2413891	N/A	12.3	071205E-220
21 C0049882 Rep	DF09V01AVP0010041007	Unknown	1	13C PFOA	2742993	N/A	14.1	071205E-221
22 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	548943	2.50	2.39	071205E-222
23 C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2971667	25.0	15.3	071205E-223
24 C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3231166	250.	167.	071205E-224
25 C0049883	DF09V01PAP0010041007	Unknown	1	13C PFOA	2285633	N/A	11.6	071205E-225
26 C0049883 Rep	DF09V01PAP0010041007	Unknown	1	13C PFOA	2555998	N/A	13.1	071205E-226
27 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1068620	5.00	5.16	071205E-227
28 C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	3191821	25.0	16.4	071205E-228
29 C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3854439	250.	200.	071205E-229
30 C0049884	DF09V01RCP0010041007	Unknown	1	13C PFOA	2870184	N/A	14.7	071205E-230
31 C0049884 Rep	DF09V01RCP0010041007	Unknown	1	13C PFOA	2775824	N/A	14.2	071205E-231
32 SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2093656	10.0	10.6	071205E-232
33 C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2265868	25.0	11.5	071205E-233
34 C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3555947	250.	184.	071205E-234
35 C0049885	DF09V02RCP0010041007	Unknown	1	13C PFOA	2298301	N/A	11.7	071205E-235
36 C0049885 Rep	DF09V02RCP0010041007	Unknown	1	13C PFOA	2065472	N/A	10.5	071205E-236
37 SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	4651683	25.0	24.2	071205E-237
38 SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9350444	50.0	49.2	071205E-238

Fish Conversion

Compound: 13C PFOA

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049881	DF06-V02-RCP001-0-041007	11.9	4.76
C0049881 Rep	DF06-V02-RCP001-0-041007	11.8	4.72
C0049881 Spk C	DF06-V02-RCP001-0-041007	13.0	5.20
C0049881 Spk D	DF06-V02-RCP001-0-041007	183	73.2
C0049882	DF09-V01-AVP001-0-041007	12.3	4.92
C0049882 Rep	DF09-V01-AVP001-0-041007	14.1	5.64
C0049882 Spk E	DF09-V01-AVP001-0-041007	16.6	6.64
C0049882 Spk F	DF09-V01-AVP001-0-041007	153	61.2
C0049883	DF09-V01-PAP001-0-041007	11.6	4.64
C0049883 Rep	DF09-V01-PAP001-0-041007	13.1	5.24
C0049883 Spk G	DF09-V01-PAP001-0-041007	15.3	6.12
C0049883 Spk H	DF09-V01-PAP001-0-041007	167	66.8
C0049884	DF09-V01-RCP001-0-041007	14.7	5.88
C0049884 Rep	DF09-V01-RCP001-0-041007	14.2	5.68
C0049884 Spk I	DF09-V01-RCP001-0-041007	16.4	6.56
C0049884 Spk J	DF09-V01-RCP001-0-041007	200	80.0
C0049885	DF09-V02-RCP001-0-041007	11.7	4.68
C0049885 Rep	DF09-V02-RCP001-0-041007	10.5	4.20
C0049885 Spk K	DF09-V02-RCP001-0-041007	11.5	4.60
C0049885 Spk L	DF09-V02-RCP001-0-041007	184	73.6

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = ~~Peak Area 0~~ ^{Peak Area 0} ~~Area Above 0.2 ng/g~~ ^{Area Above 0.2 ng/g} \odot

NQ = Not quantifiable = Measured concentration below Limit of Quantitation (LOQ) which is 0.5 ng/g.

^
BETWEEN 0.2 ng/g AND \odot

40

\odot RE MLC 9/20/05

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BAL 07/28/05

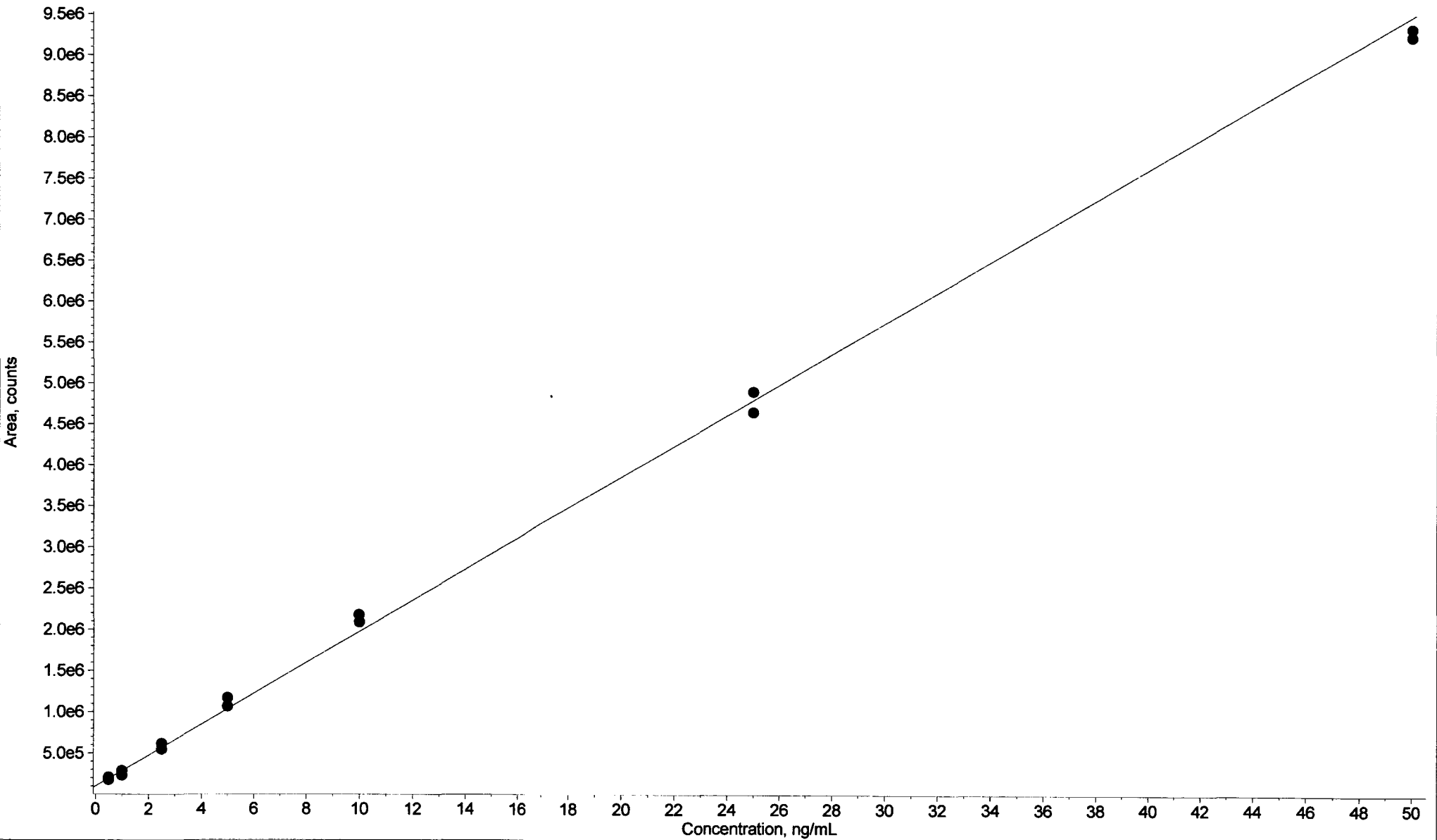
	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound:	13C PFOA	Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049881	DF06-V02-RCP001-0-041007	=Raw Data!H101	=(C9*2)/5
10	C0049881 Rep	DF06-V02-RCP001-0-041007	=Raw Data!H102	=(C10*2)/5
11	C0049881 Spk C	DF06-V02-RCP001-0-041007	=Raw Data!H99	=(C11*2)/5
12	C0049881 Spk D	DF06-V02-RCP001-0-041007	=Raw Data!H100	=(C12*2)/5
13				
14	C0049882	DF09-V01-AVP001-0-041007	=Raw Data!H106	=(C14*2)/5
15	C0049882 Rep	DF09-V01-AVP001-0-041007	=Raw Data!H107	=(C15*2)/5
16	C0049882 Spk E	DF09-V01-AVP001-0-041007	=Raw Data!H104	=(C16*2)/5
17	C0049882 Spk F	DF09-V01-AVP001-0-041007	=Raw Data!H105	=(C17*2)/5
18				
19	C0049883	DF09-V01-PAP001-0-041007	=Raw Data!H111	=(C19*2)/5
20	C0049883 Rep	DF09-V01-PAP001-0-041007	=Raw Data!H112	=(C20*2)/5
21	C0049883 Spk G	DF09-V01-PAP001-0-041007	=Raw Data!H109	=(C21*2)/5
22	C0049883 Spk H	DF09-V01-PAP001-0-041007	=Raw Data!H110	=(C22*2)/5
23				
24	C0049884	DF09-V01-RCP001-0-041007	=Raw Data!H116	=(C24*2)/5
25	C0049884 Rep	DF09-V01-RCP001-0-041007	=Raw Data!H117	=(C25*2)/5
26	C0049884 Spk I	DF09-V01-RCP001-0-041007	=Raw Data!H114	=(C26*2)/5
27	C0049884 Spk J	DF09-V01-RCP001-0-041007	=Raw Data!H115	=(C27*2)/5
28				
29	C0049885	DF09-V02-RCP001-0-041007	=Raw Data!H121	=(C29*2)/5
30	C0049885 Rep	DF09-V02-RCP001-0-041007	=Raw Data!H122	=(C30*2)/5
31	C0049885 Spk K	DF09-V02-RCP001-0-041007	=Raw Data!H119	=(C31*2)/5
32	C0049885 Spk L	DF09-V02-RCP001-0-041007	=Raw Data!H120	=(C32*2)/5
33				

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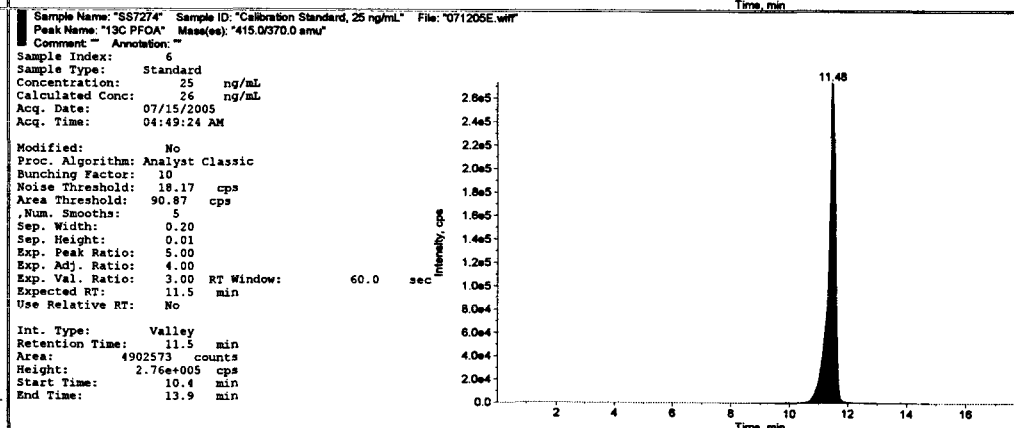
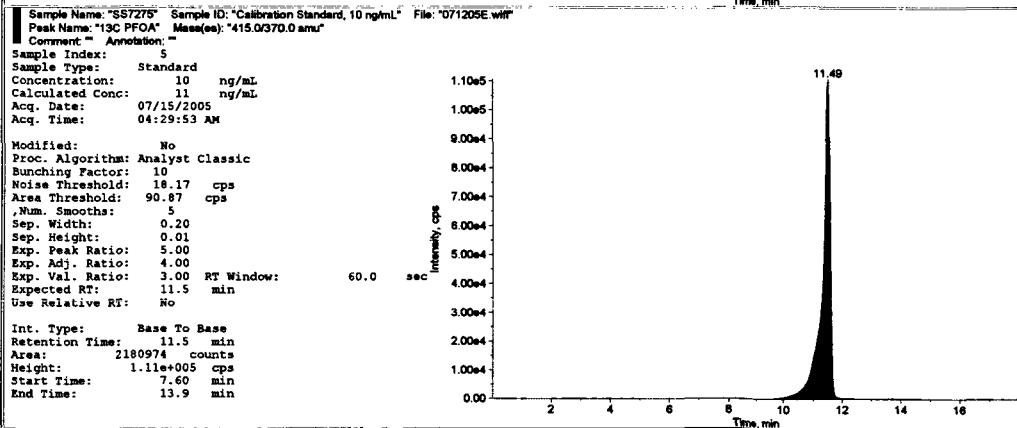
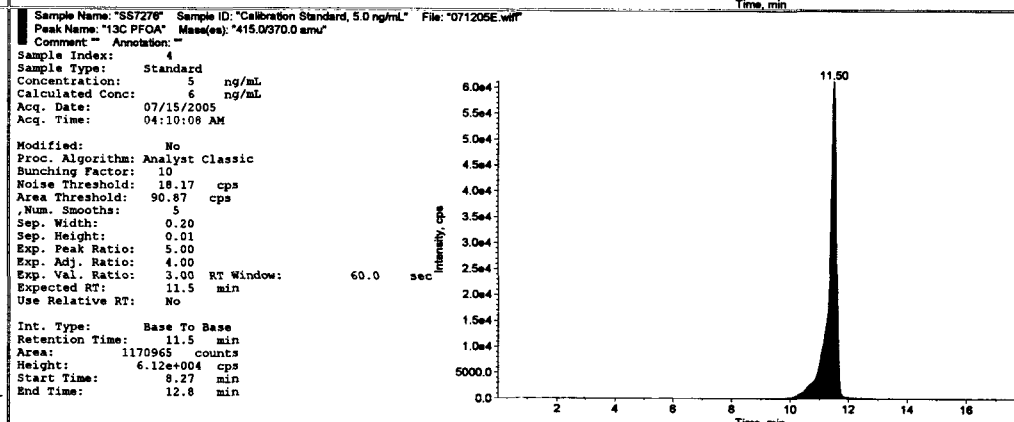
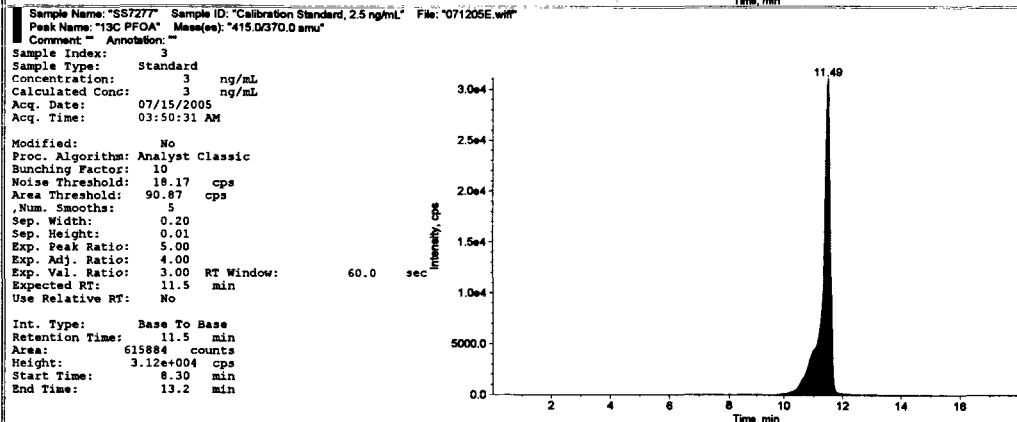
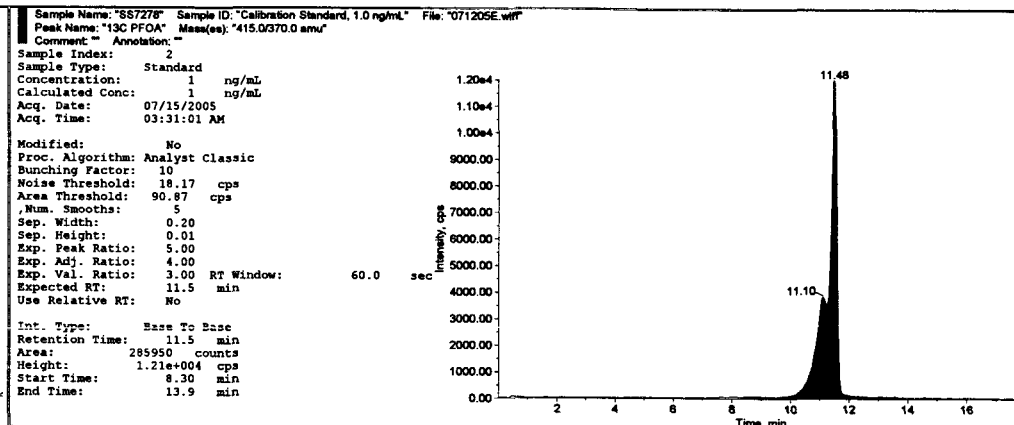
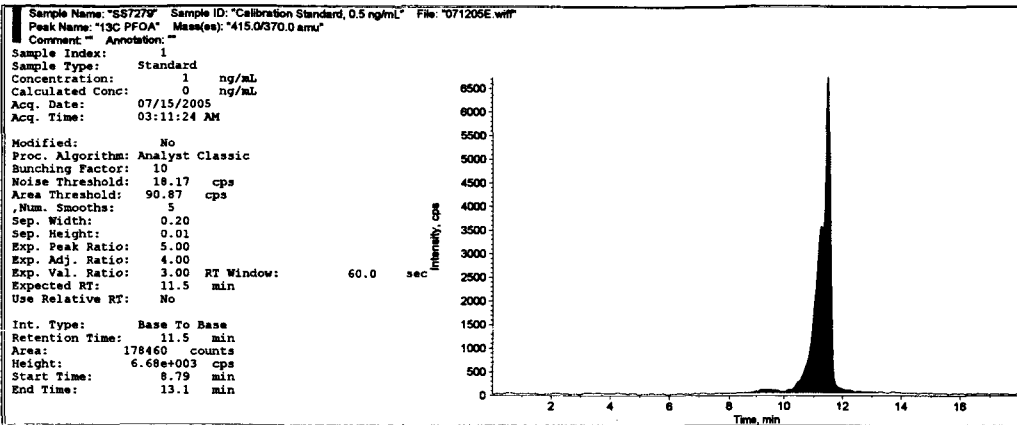
BAK 07/28/05

AMB 07/21/05

■ Untitled 6 (13C PFOA): "Linear" Regression ("1/x" weighting): $y = 1.88e+005 x + 9.93e+004$ ($r = 0.9985$)

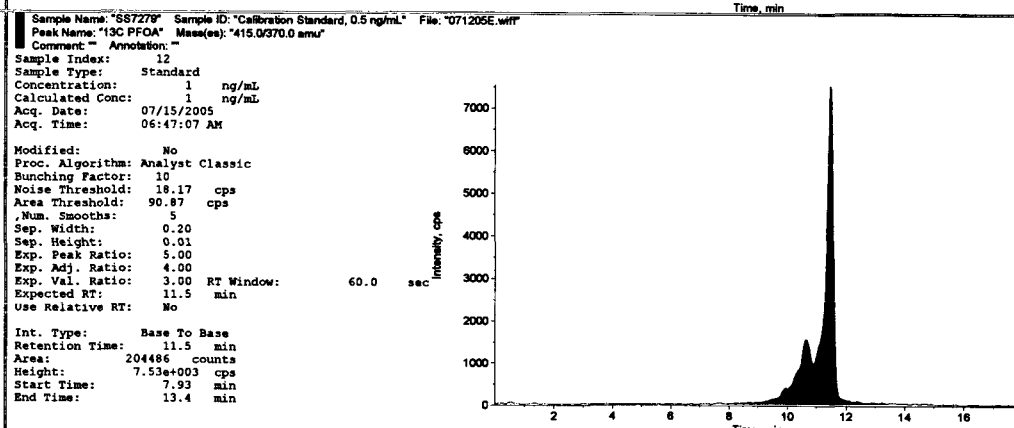
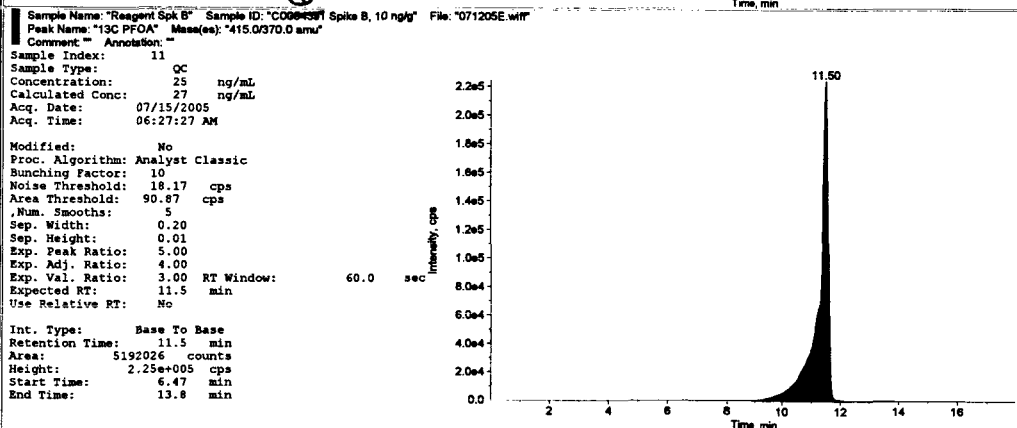
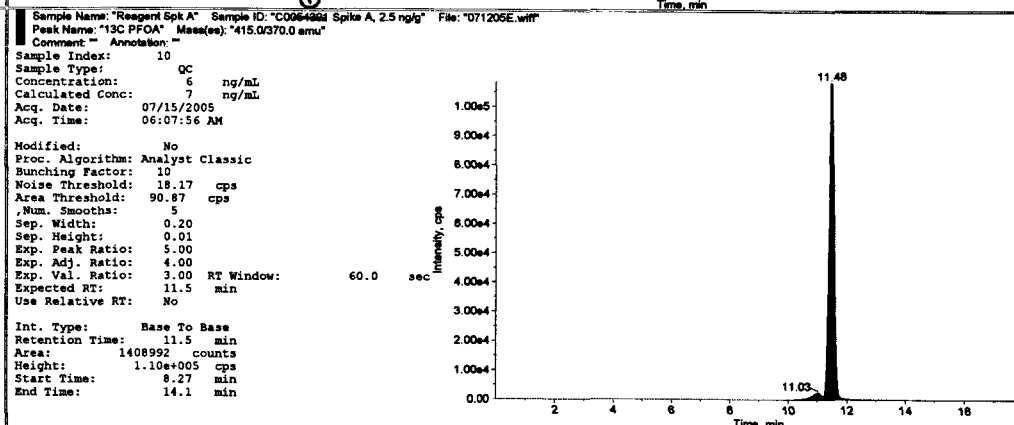
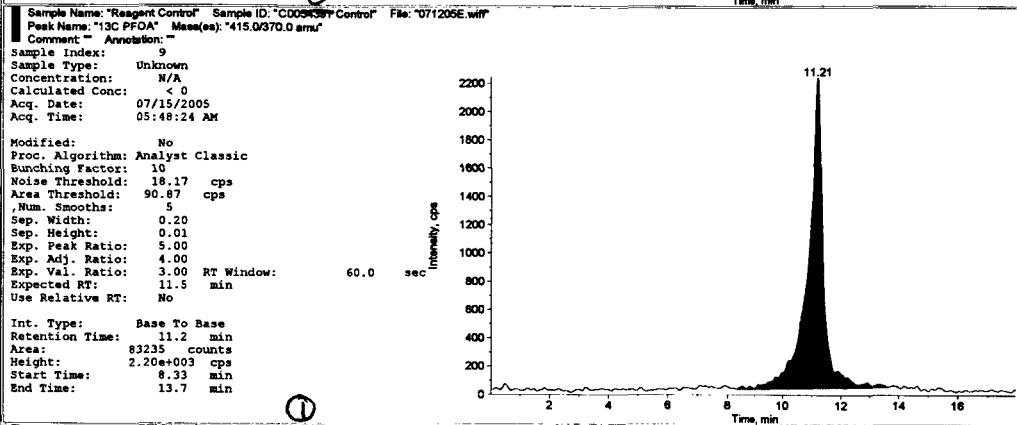
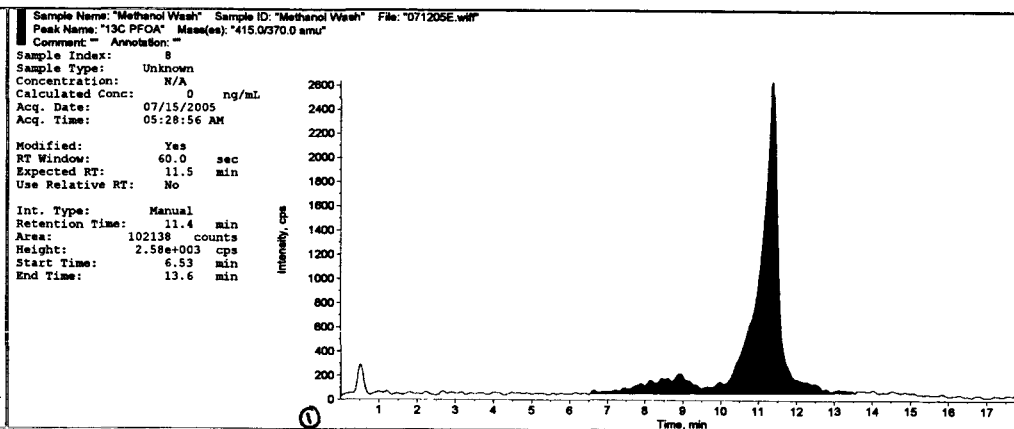
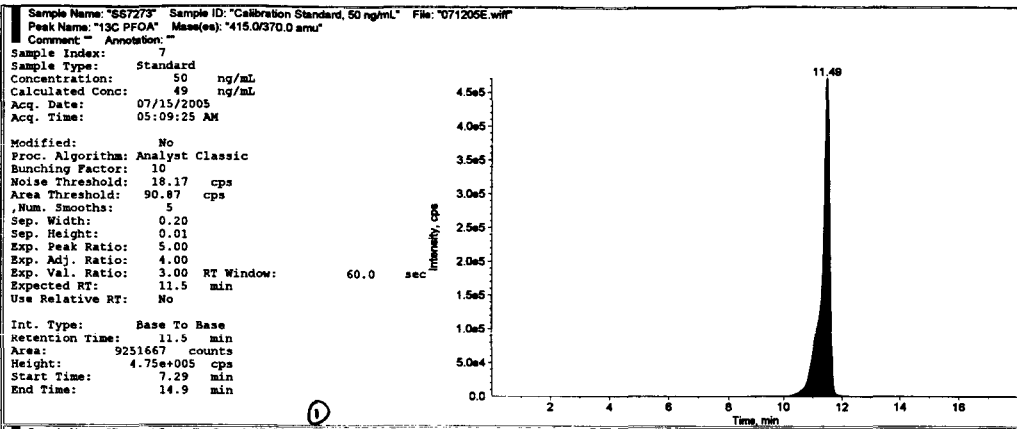


0355

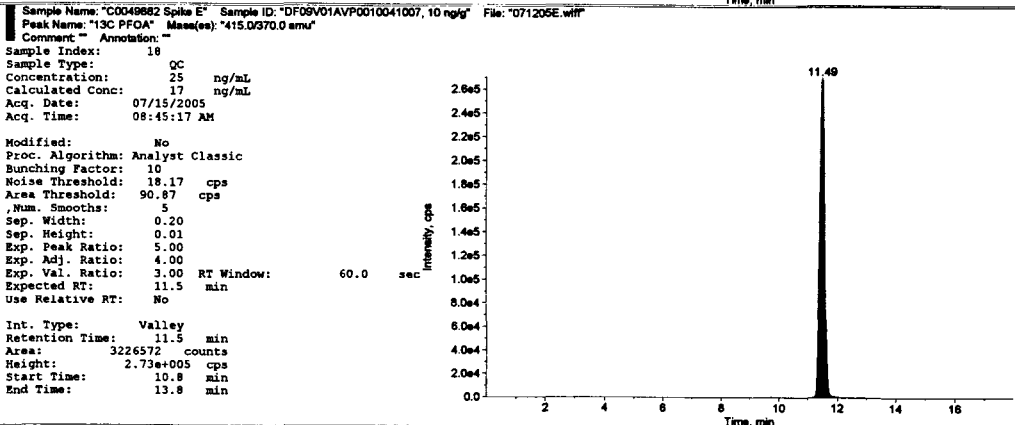
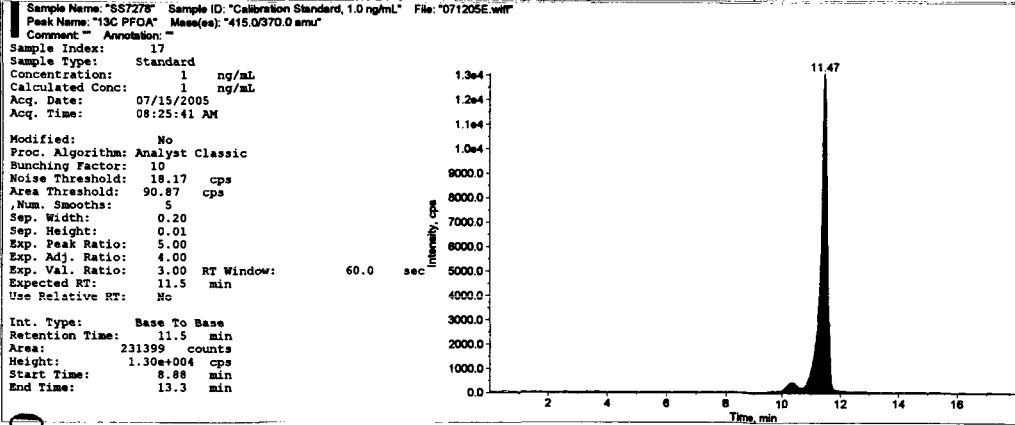
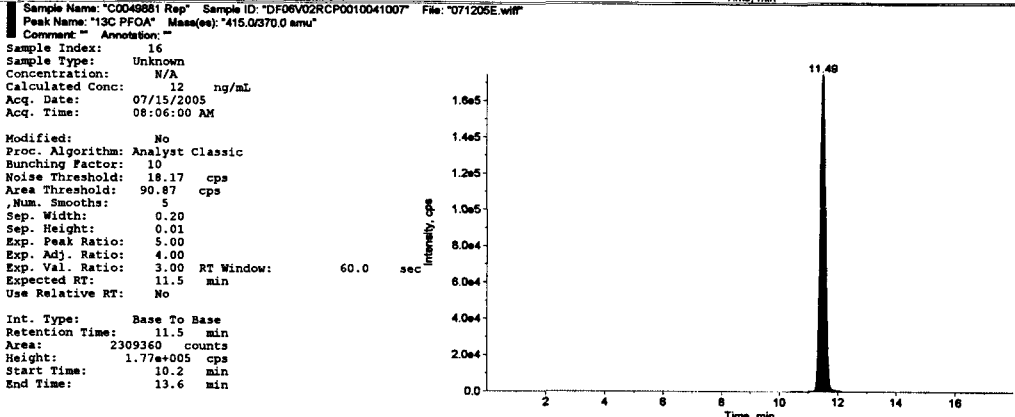
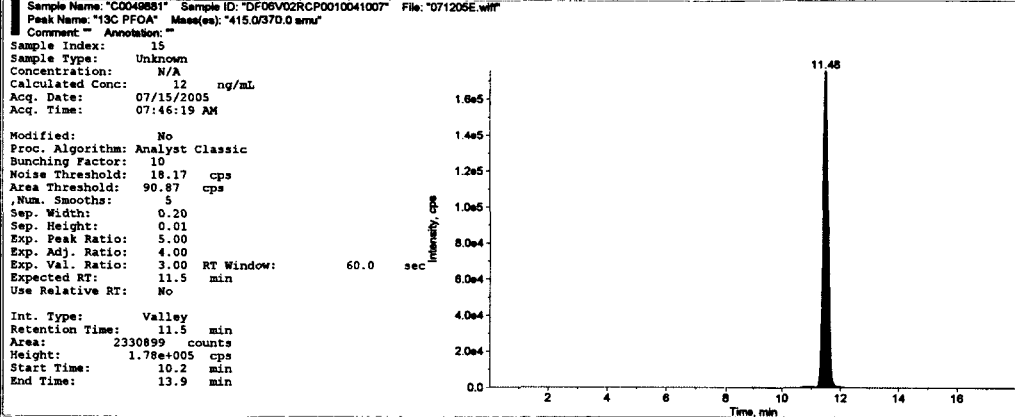
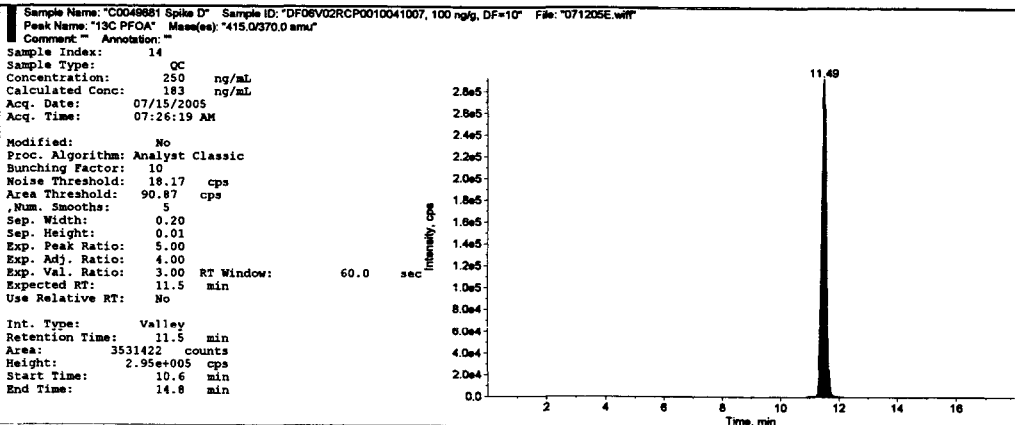
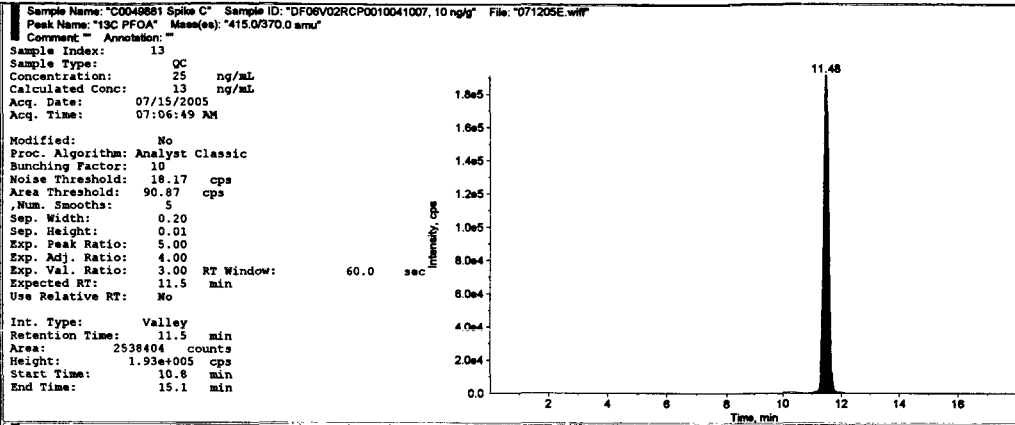


0 Initials AMB
 00 Date 07/21/05
 01 Run# 1 To 38
 02 Sample Index (C) AMB 07/21/05

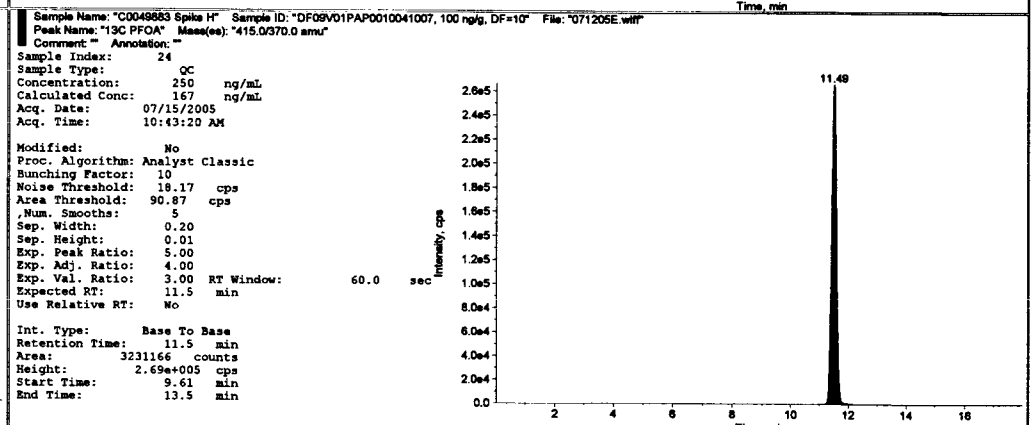
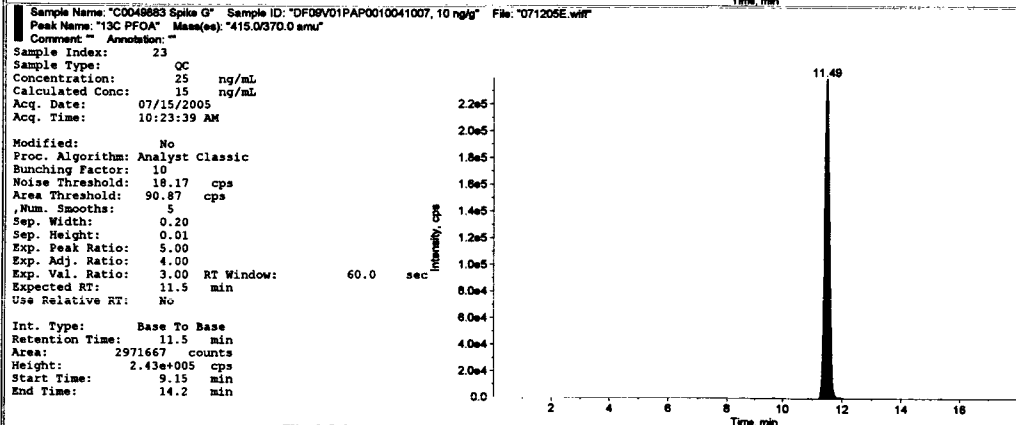
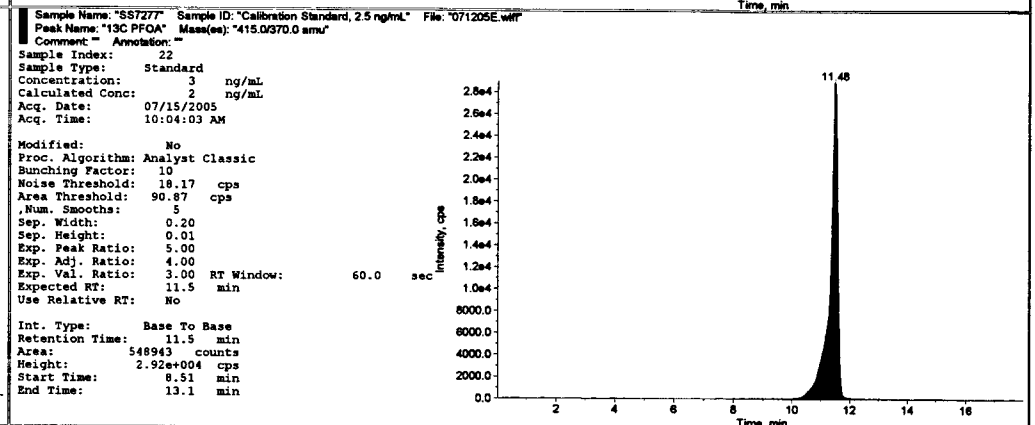
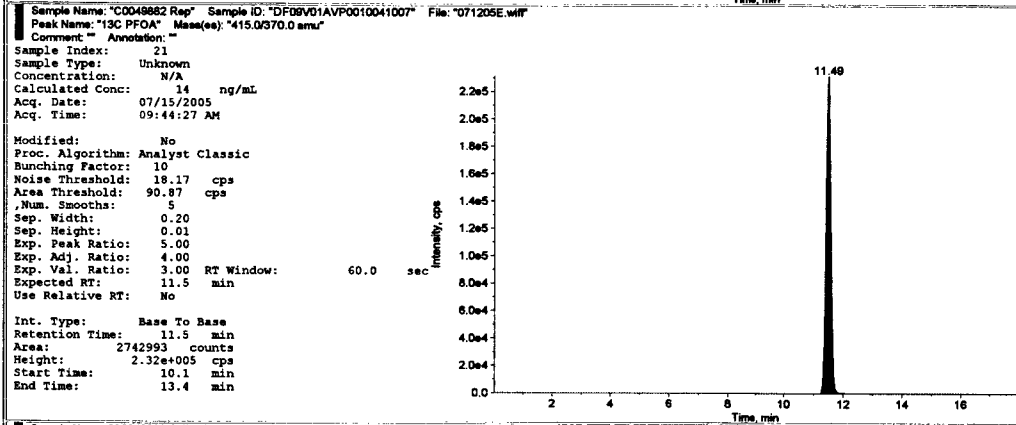
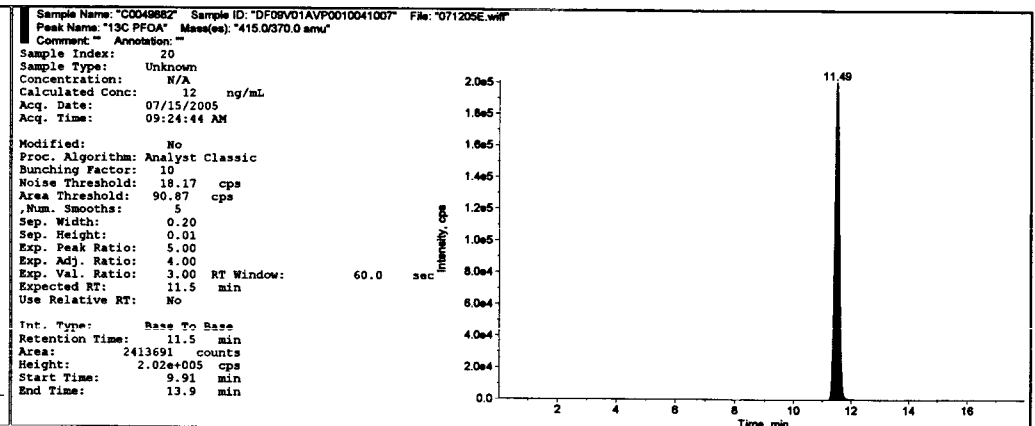
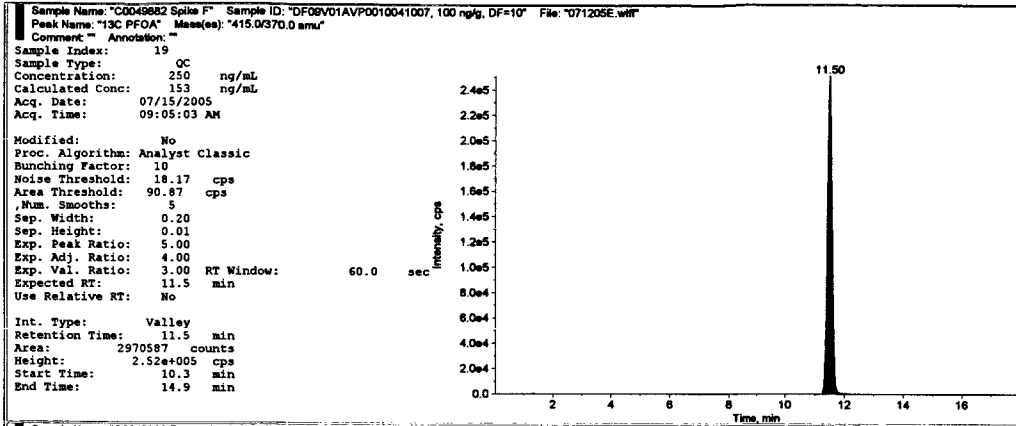
Verified by: DS
 Initials: DS Date 07/22/05
 Sample Index: 1 To: 38



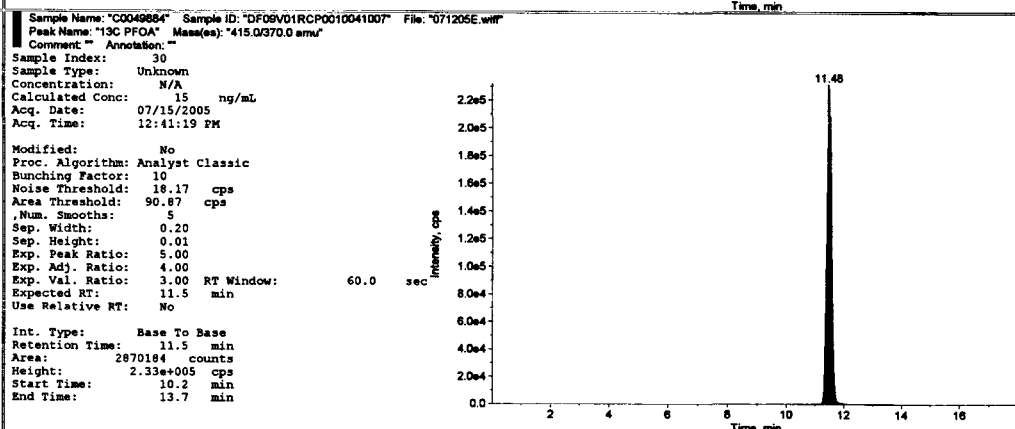
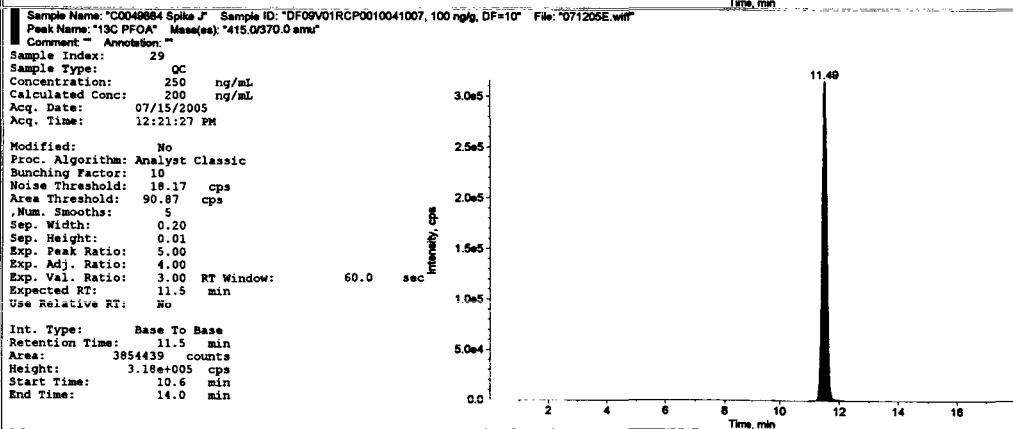
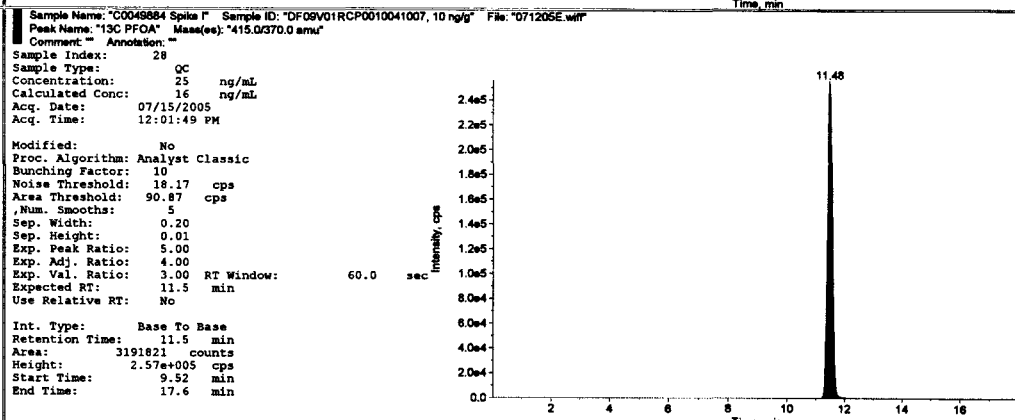
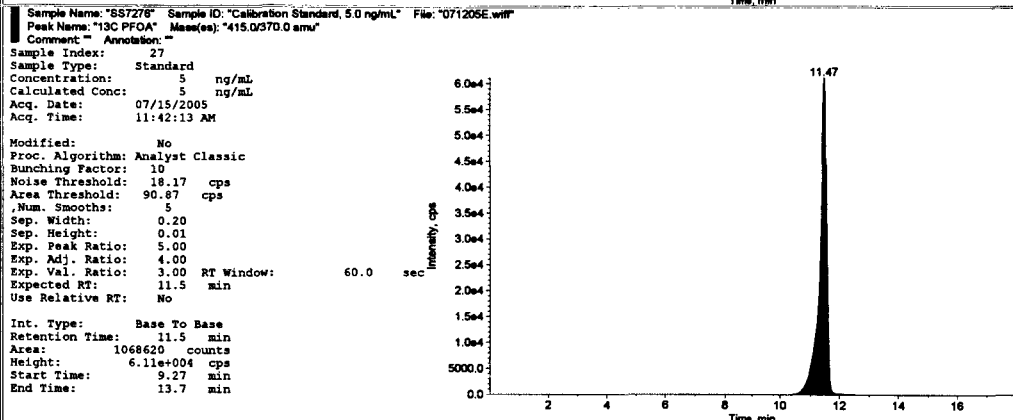
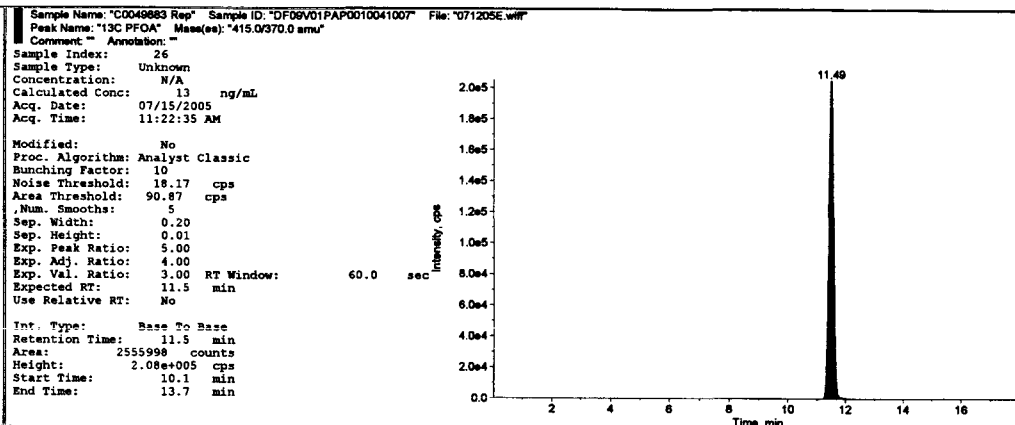
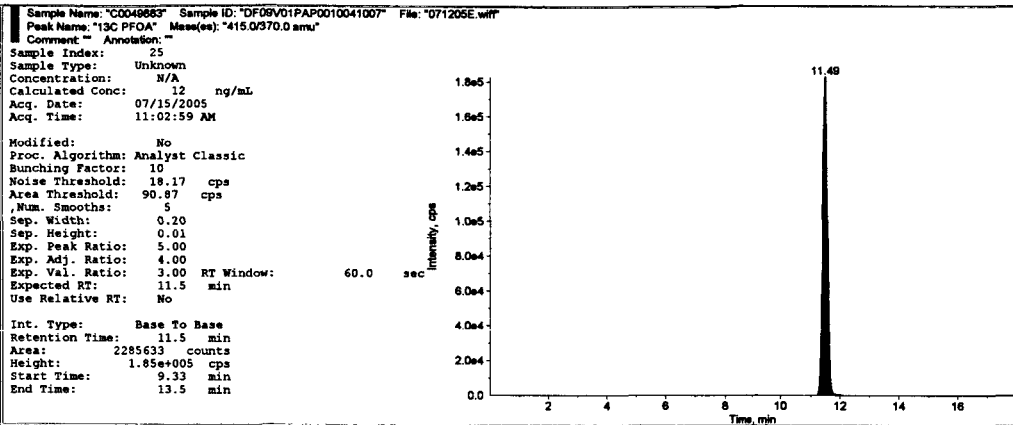
① RE BAK 09/20/05 0357



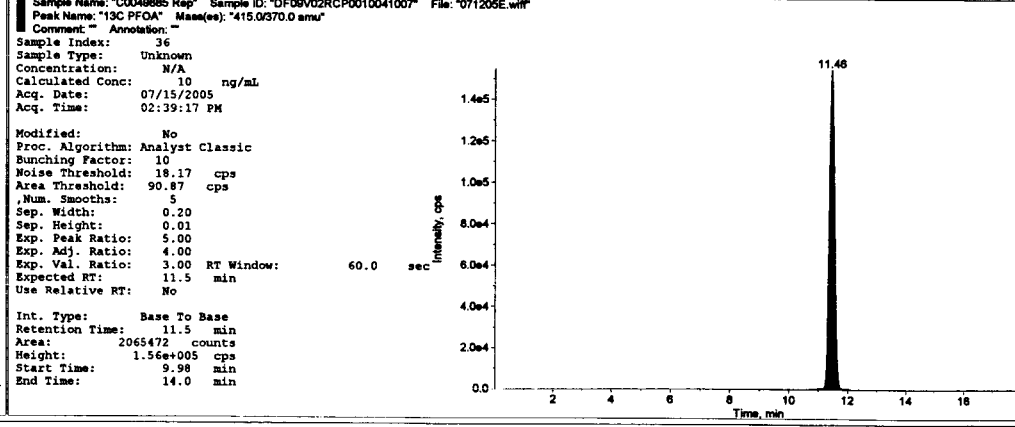
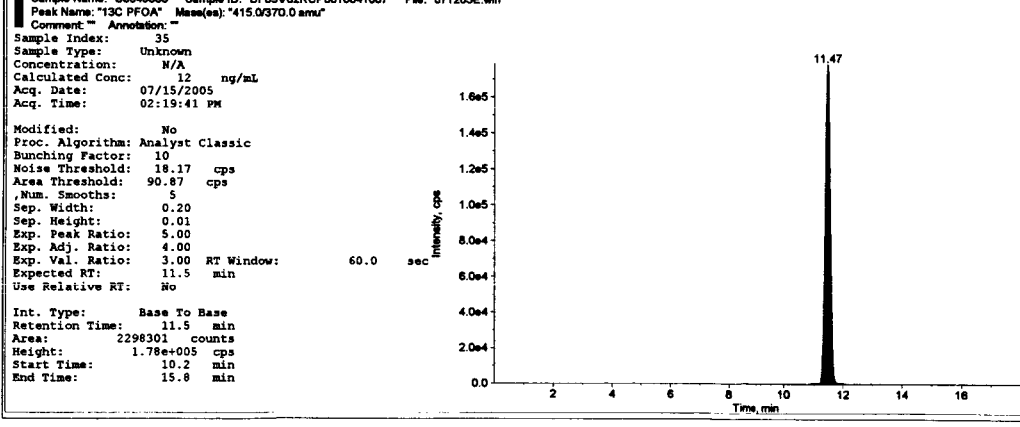
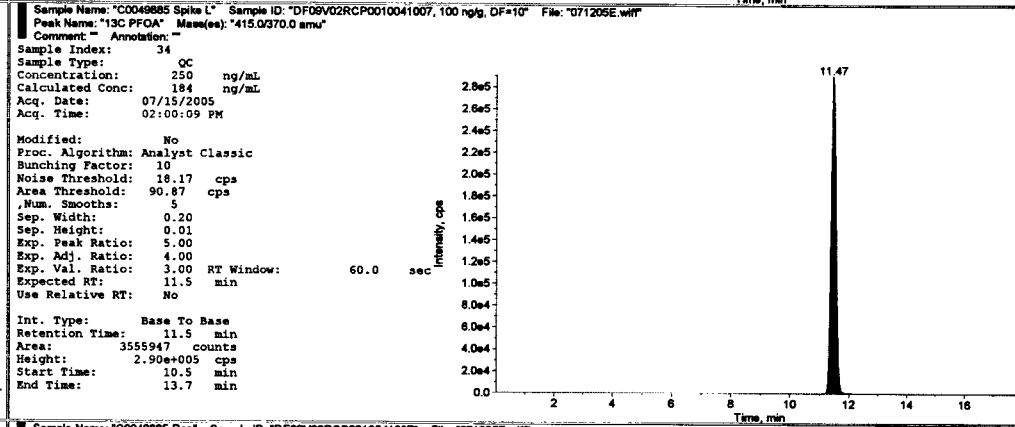
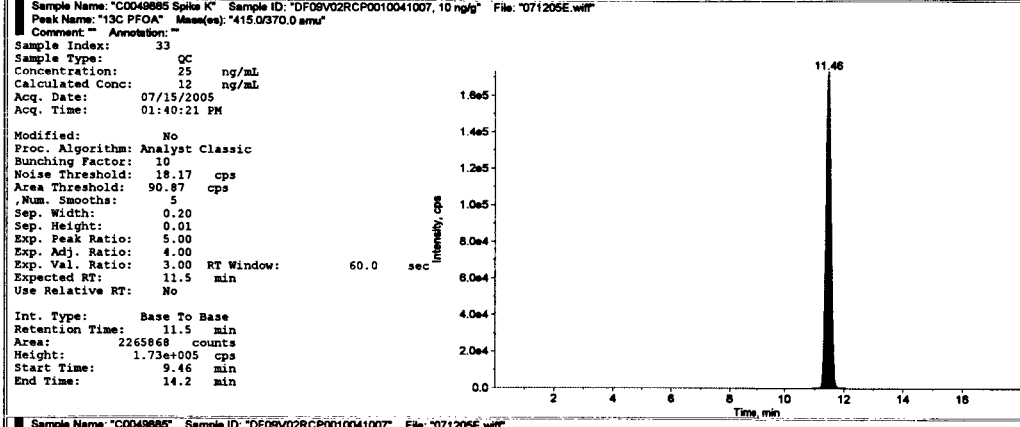
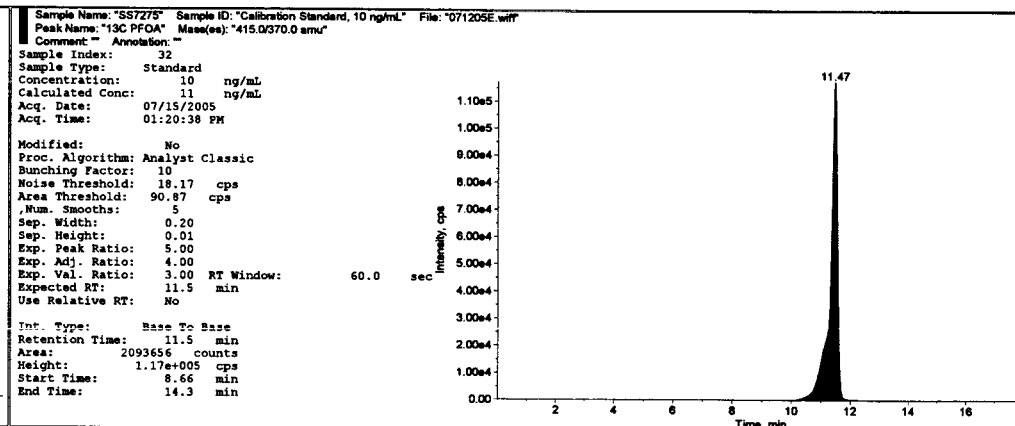
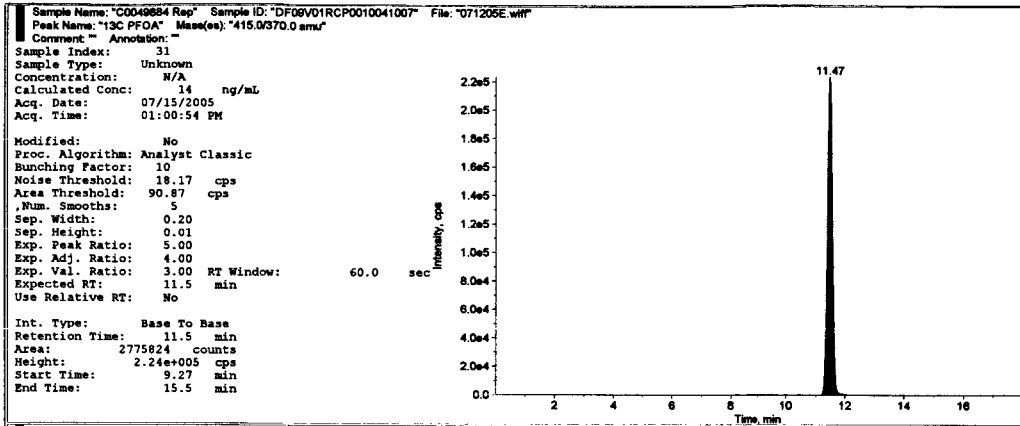
0328



0359

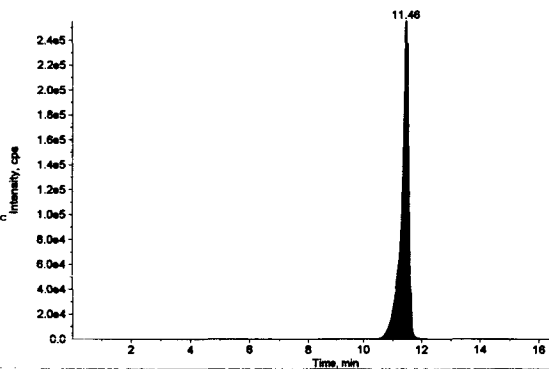


0360

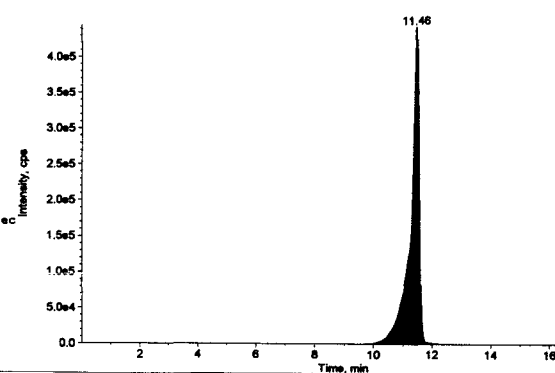


0361

Sample Name: "SS7274" Sample ID: "Calibration Standard, 25 ng/mL" File: "071205E.wiff"
Peak Name: "13C PFOA" Mass(es): "415.0370.0 amu"
Comment: "" Annotation: ""
Sample Index: 37
Sample Type: Standard
Concentration: 25 ng/mL
Calculated Conc: 24 ng/mL
Acq. Date: 07/15/2005
Acq. Time: 02:58:54 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 18.17 cps
Area Threshold: 90.87 cps
Num. Smooths: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 4651683 counts
Height: 2.58e+005 cps
Start Time: 8.69 min
End Time: 14.2 min



Sample Name: "SS7273" Sample ID: "Calibration Standard, 50 ng/mL" File: "071205E.wiff"
Peak Name: "13C PFOA" Mass(es): "415.0370.0 amu"
Comment: "" Annotation: ""
Sample Index: 38
Sample Type: Standard
Concentration: 50 ng/mL
Calculated Conc: 49 ng/mL
Acq. Date: 07/15/2005
Acq. Time: 03:18:32 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 18.17 cps
Area Threshold: 90.87 cps
Num. Smooths: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 9350444 counts
Height: 4.49e+005 cps
Start Time: 8.54 min
End Time: 14.0 min



PFBS								
Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng/ml)	Calculated Concentration (ng/ml)	Run #
SS7279	Calibration Standard, 0.5 ng/ml	Standard	1.0	PFBS	16651	0.5	0.44	7.12E-197
SS7278	Calibration Standard, 1.0 ng/ml	Standard	1.0	PFBS	33432	1	1.05	7.12E-198
SS7277	Calibration Standard, 2.5 ng/ml	Standard	1.0	PFBS	77743	2.5	2.66	7.12E-199
SS7276	Calibration Standard, 5.0 ng/ml	Standard	1.0	PFBS	150141	5	5.29	7.12E-200
SS7275	Calibration Standard, 10 ng/ml	Standard	1.0	PFBS	290990	10	10.4	7.12E-201
SS7274	Calibration Standard, 25 ng/ml	Standard	1.0	PFBS	716568	25	25.9	7.12E-202
SS7273	Calibration Standard, 50 ng/ml	Standard	1.0	PFBS	1402012	50	50.8	7.12E-203
Methanol Wash	Methanol Wash	Unknown	1.0	PFBS	1497	N/A	<0	7.12E-204
Reagent Control	C0073483 Control	Unknown	1.0	PFBS	0	N/A	No Peak	7.12E-205
Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1.0	PFBS	175830	6.25	6.23	7.12E-206
Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1.0	PFBS	699121	25	25.3	7.12E-207
SS7279	Calibration Standard, 0.5 ng/ml	Standard	1.0	PFBS	16842	0.5	0.447	7.12E-208
C004981 Spike C	DF06V02RCP0010041007, 10 ng/g	Quality Control	1.0	PFBS	932842	25	33.8	7.12E-209
C004981 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	594812	250	215	7.12E-210
C004981	DF06V02RCP0010041007	Unknown	1.0	PFBS	410915	N/A	14.8	7.12E-211
C004981 Rep	DF06V02RCP0010041007	Unknown	1.0	PFBS	414154	N/A	14.9	7.12E-212
SS7278	Calibration Standard, 1.0 ng/ml	Standard	1.0	PFBS	31434	1	0.977	7.12E-213
C004982 Spike E	DF09V01AVP0010041007, 10 ng/g	Quality Control	1.0	PFBS	772003	25	27.9	7.12E-214
C004982 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	505947	250	182	7.12E-215
C004982	DF09V01AVP0010041007	Unknown	1.0	PFBS	244365	N/A	8.72	7.12E-216
C004982 Rep	DF09V01AVP0010041007	Unknown	1.0	PFBS	259330	N/A	9.27	7.12E-217
SS7277	Calibration Standard, 2.5 ng/ml	Standard	1.0	PFBS	74286	2.5	2.54	7.12E-218
C004983 Spike G	DF09V01PAP0010041007, 10 ng/g	Quality Control	1.0	PFBS	843450	25	30.5	7.12E-219
C004983 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	567975	250	205	7.12E-220
C004983	DF09V01PAP0010041007	Unknown	1.0	PFBS	350535	N/A	12.6	7.12E-221
C004983 Rep	DF09V01PAP0010041007	Unknown	1.0	PFBS	341458	N/A	12.3	7.12E-222
SS7276	Calibration Standard, 5.0 ng/ml	Standard	1.0	PFBS	147221	5	5.19	7.12E-223
C004984 Spike I	DF09V01RCP0010041007, 10 ng/g	Quality Control	1.0	PFBS	633044	25	23.6	7.12E-224
C004984 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	625244	250	226	7.12E-225
C004984	DF09V01RCP0010041007	Unknown	1.0	PFBS	108023	N/A	3.76	7.12E-226
C004984 Rep	DF09V01RCP0010041007	Unknown	1.0	PFBS	1.01E+05	N/A	3.5	7.12E-227
SS7275	Calibration Standard, 10 ng/ml	Standard	1	PFBS	2.79E+05	10.0	10	7.12E-228
C004985 Spike K	DF09V02RCP0010041007, 10 ng/g	Quality Control	1	PFBS	6.16E+05	25.0	22.3	7.12E-229
C004985 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFBS	5.54E+05	250	200	7.12E-230
C004985	DF09V02RCP0010041007	Unknown	1	PFBS	9.97E+04	N/A	3.46	7.12E-231
C004985 Rep	DF09V02RCP0010041007	Unknown	1	PFBS	9.85E+04	N/A	3.42	7.12E-232
SS7274	Calibration Standard, 25 ng/ml	Standard	1	PFBS	6.71E+05	25.0	24.2	7.12E-233
SS7273	Calibration Standard, 50 ng/ml	Standard	1	PFBS	1.32E+06	50.0	48	7.12E-234

PFHS								
Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng/ml)	Calculated Concentration (ng/ml)	Run #
SS7279	Calibration Standard, 0.5 ng/ml	Standard	1	PFHS	25742	0.500	0.46	7.12E-197
SS7278	Calibration Standard, 1.0 ng/ml	Standard	1	PFHS	50510	1.00	0.968	7.12E-198
SS7277	Calibration Standard, 2.5 ng/ml	Standard	1	PFHS	126201	2.50	2.52	7.12E-199
SS7276	Calibration Standard, 5.0 ng/ml	Standard	1	PFHS	254801	5.00	5.16	7.12E-200
SS7275	Calibration Standard, 10 ng/ml	Standard	1	PFHS	492643	10.0	10	7.12E-201
SS7274	Calibration Standard, 25 ng/ml	Standard	1	PFHS	1226028	25.0	25.1	7.12E-202
SS7273	Calibration Standard, 50 ng/ml	Standard	1	PFHS	2580382	50.0	52.9	7.12E-203
Methanol Wash	Methanol Wash	Unknown	1	PFHS	494	N/A	<0	7.12E-204
Reagent Control	C0073483 Control	Unknown	1	PFHS	178	N/A	<0	7.12E-205
Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFHS	347636	6.25	7.07	7.12E-206
Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFHS	1207864	25.0	24.7	7.12E-207
SS7279	Calibration Standard, 0.5 ng/ml	Standard	1	PFHS	28102	0.500	0.508	7.12E-208
C004981 Spike C	DF06V02RCP0010041007, 10 ng/g	Quality Control	1	PFHS	2234936	25.0	45.8	7.12E-209
C004981 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1237191	250	257	7.12E-210
C004981	DF06V02RCP0010041007	Unknown	1	PFHS	1115610	N/A	22.8	7.12E-211
C004981 Rep	DF06V02RCP0010041007	Unknown	1	PFHS	1130902	N/A	23.2	7.12E-212
SS7278	Calibration Standard, 1.0 ng/ml	Standard	1	PFHS	51123	1.00	0.981	7.12E-213
C004982 Spike E	DF09V01AVP0010041007, 10 ng/g	Quality Control	1	PFHS	3479011	25.0	71.4	7.12E-214
C004982 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1189562	250	244	7.12E-215
C004982	DF09V01AVP0010041007	Unknown	1	PFHS	2599420	N/A	53.3	7.12E-216
C004982 Rep	DF09V01AVP0010041007	Unknown	1	PFHS	2595612	N/A	53.2	7.12E-217
SS7277	Calibration Standard, 2.5 ng/ml	Standard	1	PFHS	127125	2.50	2.54	7.12E-218
C004983 Spike G	DF09V01PAP0010041007, 10 ng/g	Quality Control	1	PFHS	2834090	25.0	58.1	7.12E-219
C004983 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1284811	250	263	7.12E-220
C004983	DF09V01PAP0010041007	Unknown	1	PFHS	1582498	N/A	32.4	7.12E-221
C004983 Rep	DF09V01PAP0010041007	Unknown	1	PFHS	1790953	N/A	36.7	7.12E-222
SS7276	Calibration Standard, 5.0 ng/ml	Standard	1	PFHS	252344	5.00	5.11	7.12E-223
C004984 Spike I	DF09V01RCP0010041007, 10 ng/g	Quality Control	1	PFHS	2417397	25.0	49.6	7.12E-224
C004984 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1433139	250	294	7.12E-225
C004984	DF09V01RCP0010041007	Unknown	1	PFHS	1357558	N/A	27.8	7.12E-226
C004984 Rep	DF09V01RCP0010041007	Unknown	1	PFHS	1331694	N/A	27.3	7.12E-227
SS7275	Calibration Standard, 10 ng/ml	Standard	1	PFHS	517029	10.0	10.5	7.12E-228
C004985 Spike K	DF09V02RCP0010041007, 10 ng/g	Quality Control	1	PFHS	5239759	25.0	108	7.12E-229
C004985 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1606791	250	329	7.12E-230
C004985	DF09V02RCP0010041007	Unknown	1	PFHS	4036982	N/A	82.8	7.12E-231
C004985 Rep	DF09V02RCP0010041007	Unknown	1	PFHS	4128565	N/A	84.7	7.12E-232
SS7274	Calibration Standard, 25 ng/ml	Standard	1	PFHS	1191964	25.0	24.4	7.12E-233
SS7273	Calibration Standard, 50 ng/ml	Standard	1	PFHS	2277538	50.0	46.7	7.12E-234

13C PFOA								
Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng/ml)	Calculated Concentration (ng/ml)	Run #
SS7279	Calibration Standard, 0.5 ng/ml	Standard	1	13C PFOA	178460	0.5	0.421	7.12E-197
SS7278	Calibration Standard, 1.0 ng/ml	Standard	1	13C PFOA	285930	1	0.993	7.12E-198
SS7277	Calibration Standard, 2.5 ng/ml	Standard	1	13C PFOA	615884	2.5	2.75	7.12E-199
SS7276	Calibration Standard, 5.0 ng/ml	Standard	1	13C PFOA	1170965	5	5.7	7.12E-200
SS7275	Calibration Standard, 10 ng/ml	Standard	1	13C PFOA	2180974	10	11.1	7.12E-201
SS7274	Calibration Standard, 25 ng/ml	Standard	1	13C PFOA	4902573	25	25.5	7.12E-202
SS7273	Calibration Standard, 50 ng/ml	Standard	1	13C PFOA	9251667	50	48.7	7.12E-203
Methanol Wash	Methanol Wash	Unknown	1	13C PFOA	102138	N/A	0.0152	7.12E-204
Reagent Control	C0073483 Control	Unknown	1	13C PFOA	83235	N/A	<0	7.12E-205
Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	13C PFOA	1408992	6.25	6.97	7.12E-206
Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	13C PFOA	5192026	25	27.1	7.12E-207
SS7279	Calibration Standard, 0.5 ng/ml	Standard	1	13C PFOA	204486	0.5	0.56	7.12E-208
C004981 Spike C	DF06V02RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2538404	25	13	7.12E-209
C004981 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3531422	250	183	7.12E-210
C004981	DF06V02RCP0010041007	Unknown	1	13C PFOA	2330899	N/A	11.9	7.12E-211
C004981 Rep	DF06V02RCP0010041007	Unknown	1	13C PFOA	2309560	N/A	11.8	7.12E-212
SS7278	Calibration Standard, 1.0 ng/ml	Standard	1	13C PFOA	231399	1	0.703	7.12E-213

0363
BAC 07/28/05

Raw Data

A	B	C	D	E	F	G	H	I	
104	C004982 Spike E	DF09V01AVP0010041007, 10 ng/g	Quality Control	1	13C PFOA	3226572	25	16.6	7.12E-214
105	C004982 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2970587	250	153	7.12E-215
106	C004982	DF09V01AVP0010041007	Unknown	1	13C PFOA	2413691	N/A	12.3	7.12E-216
107	C004982 Rep	DF09V01AVP0010041007	Unknown	1	13C PFOA	2742993	N/A	14.1	7.12E-217
108	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	348943	2.5	2.39	7.12E-218
109	C004983 Spike G	DF09V01PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2971667	25	15.3	7.12E-219
110	C004983 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3233166	250	167	7.12E-220
111	C004983	DF09V01PAP0010041007	Unknown	1	13C PFOA	2285653	N/A	11.6	7.12E-221
112	C004983 Rep	DF09V01PAP0010041007	Unknown	1	13C PFOA	2555998	N/A	13.1	7.12E-222
113	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1068200	5	5.16	7.12E-223
114	C004984 Spike I	DF09V01RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	3191821	25	16.4	7.12E-224
115	C004984 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3854439	250	200	7.12E-225
116	C004984	DF09V01RCP0010041007	Unknown	1	13C PFOA	2870184	N/A	14.7	7.12E-226
117	C004984 Rep	DF09V01RCP0010041007	Unknown	1	13C PFOA	2775824	N/A	14.2	7.12E-227
118	SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2093656	10	10.6	7.12E-228
119	C004985 Spike K	DF09V02RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	2265868	25	11.5	7.12E-229
120	C004985 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3555947	250	184	7.12E-230
121	C004985	DF09V02RCP0010041007	Unknown	1	13C PFOA	2298301	N/A	11.7	7.12E-231
122	C004985 Rep	DF09V02RCP0010041007	Unknown	1	13C PFOA	2065472	N/A	10.5	7.12E-232
123	SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	4651683	25	24.2	7.12E-233
124	SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9350444	50	49.2	7.12E-234

PFOA

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng)	Calculated Concentration (ng)	Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	170926	0.5	0.378	7.12E-197
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	299429	1	0.955	7.12E-198
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	655566	2.5	2.55	7.12E-199
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1308859	5	5.48	7.12E-200
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2622018	10	11.4	7.12E-201
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5770498	25	25.5	7.12E-202
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10926992	50	48.6	7.12E-203
Methanol Wash	Methanol Wash	Unknown	1	PFOA	22846	N/A	< 0	7.12E-204
Reagent Control	C0073483 Control	Unknown	1	PFOA	47485	N/A	< 0	7.12E-205
Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFOA	1619444	6.25	6.87	7.12E-206
Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFOA	6104566	25	27	7.12E-207
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	165997	0.5	0.356	7.12E-208
C004981 Spike C	DF06V02RCP0010041007, 10 ng/g	Quality Control	1	PFOA	39819662	25	178	7.12E-209
C004981 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	9453050	250	420	7.12E-210
C004981	DF06V02RCP0010041007	Unknown	1	PFOA	38546242	N/A	172	7.12E-211
C004981 Rep	DF06V02RCP0010041007	Unknown	1	PFOA	39916149	N/A	179	7.12E-212
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	288145	1	0.904	7.12E-213
C004982 Spike E	DF09V01AVP0010041007, 10 ng/g	Quality Control	1	PFOA	899726	25	39.5	7.12E-214
C004982 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	4052837	250	178	7.12E-215
C004982	DF09V01AVP0010041007	Unknown	1	PFOA	5673154	N/A	25	7.12E-216
C004982 Rep	DF09V01AVP0010041007	Unknown	1	PFOA	5409398	N/A	23.9	7.12E-217
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	785817	2.5	3.14	7.12E-218
C004983 Spike G	DF09V01PAP0010041007, 10 ng/g	Quality Control	1	PFOA	25888168	25	116	7.12E-219
C004983 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	6533125	250	289	7.12E-220
C004983	DF09V01PAP0010041007	Unknown	1	PFOA	21086657	N/A	94.2	7.12E-221
C004983 Rep	DF09V01PAP0010041007	Unknown	1	PFOA	22897488	N/A	102	7.12E-222
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1380382	5	5.8	7.12E-223
C004984 Spike I	DF09V01RCP0010041007, 10 ng/g	Quality Control	1	PFOA	23339018	25	104	7.12E-224
C004984 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	7497863	250	332	7.12E-225
C004984	DF09V01RCP0010041007	Unknown	1	PFOA	20135416	N/A	89.9	7.12E-226
C004984 Rep	DF09V01RCP0010041007	Unknown	1	PFOA	19710177	N/A	88	7.12E-227
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2526179	10	10.9	7.12E-228
C004985 Spike K	DF09V02RCP0010041007, 10 ng/g	Quality Control	1	PFOA	44450723	25	199	7.12E-229
C004985 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	11013769	250	490	7.12E-230
C004985	DF09V02RCP0010041007	Unknown	1	PFOA	42795533	N/A	191	7.12E-231
C004985 Rep	DF09V02RCP0010041007	Unknown	1	PFOA	4281886	N/A	192	7.12E-232
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	541373	25	23.9	7.12E-233
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10827940	50	48.2	7.12E-234

PFOA Confirm Ion

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng)	Calculated Concentration (ng)	Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	3719	0.5	0.397	7.12E-197
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	7564	1	0.686	7.12E-198
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	17604	2.5	2.26	7.12E-199
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	37689	5	5.41	7.12E-200
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	68791	10	10.3	7.12E-201
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	162804	25	25	7.12E-202
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	315995	50	49	7.12E-203
Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	7.12E-204
Reagent Control	C0073483 Control	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	7.12E-205
Reagent Spk A	C0073483 Spk A, 2.5 ng/g	Quality Control	1	PFOA Confirm Ion	42814	6.25	6.21	7.12E-206
Reagent Spk B	C0073483 Spk B, 10 ng/g	Quality Control	1	PFOA Confirm Ion	177233	25	27.3	7.12E-207
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	8151	0.5	0.778	7.12E-208
C004981 Spike C	DF06V02RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	3135610	25	491	7.12E-209
C004981 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	480450	250	748	7.12E-210
C004981	DF06V02RCP0010041007	Unknown	1	PFOA Confirm Ion	3056844	N/A	479	7.12E-211
C004981 Rep	DF06V02RCP0010041007	Unknown	1	PFOA Confirm Ion	3135027	N/A	491	7.12E-212
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	8682	1	0.861	7.12E-213
C004982 Spike E	DF09V01AVP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	419448	25	65.3	7.12E-214
C004982 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	131309	250	201	7.12E-215
C004982	DF09V01AVP0010041007	Unknown	1	PFOA Confirm Ion	315718	N/A	49	7.12E-216
C004982 Rep	DF09V01AVP0010041007	Unknown	1	PFOA Confirm Ion	321538	N/A	49.9	7.12E-217
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	18456	2.5	2.39	7.12E-218
C004983 Spike G	DF09V01PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	1598291	25	250	7.12E-219
C004983 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	279934	250	434	7.12E-220
C004983	DF09V01PAP0010041007	Unknown	1	PFOA Confirm Ion	1346710	N/A	211	7.12E-221
C004983 Rep	DF09V01PAP0010041007	Unknown	1	PFOA Confirm Ion	1569130	N/A	246	7.12E-222
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	36609	5	5.15	7.12E-223
C004984 Spike I	DF09V01RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	1527261	25	239	7.12E-224
C004984 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	308640	250	479	7.12E-225
C004984	DF09V01RCP0010041007	Unknown	1	PFOA Confirm Ion	1437143	N/A	225	7.12E-226
C004984 Rep	DF09V01RCP0010041007	Unknown	1	PFOA Confirm Ion	1388450	N/A	217	7.12E-227
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	74614	10	11.2	7.12E-228
C004985 Spike K	DF09V02RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	3477573	25	545	7.12E-229
C004985 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	553392	250	866	7.12E-230
C004985	DF09V02RCP0010041007	Unknown	1	PFOA Confirm Ion	3335002	N/A	522	7.12E-231
C004985 Rep	DF09V02RCP0010041007	Unknown	1	PFOA Confirm Ion	3325777	N/A	521	7.12E-232
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	165753	25	25.5	7.12E-233
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	315862	50	49	7.12E-234

0364

BPK 07/28/05

Vegetation Set #2

Data Set 071205ER

Extracted on 07/12/05

(Analyzed on 07/26-27/05)

AMB 07/26/05

Project: D:\Analyst Data\Projects\P1131 on LCMSMS 9 Batch:071205ER VEG P760 Tab:Sample Set:SET1 AcqMethod:P760-1131 Isomer Separation 032

	Sample Name	Sample ID	Vial Position	Data File	Run #
1	SS7279	Calibration Standard, 0.5 ng/mL	1	071205ER\071205ER	071205ER-201
2	SS7278	Calibration Standard, 1.0 ng/mL	2	071205ER\071205ER	071205ER-202
3	SS7277	Calibration Standard, 2.5 ng/mL	3	071205ER\071205ER	071205ER-203
4	SS7276	Calibration Standard, 5.0 ng/mL	4	071205ER\071205ER	071205ER-204
5	SS7275	Calibration Standard, 10 ng/mL	5	071205ER\071205ER	071205ER-205
6	SS7274	Calibration Standard, 25 ng/mL	6	071205ER\071205ER	071205ER-206
7	SS7273	Calibration Standard, 50 ng/mL	7	071205ER\071205ER	071205ER-207
8	Methanol Wash	Methanol Wash	92	071205ER\071205ER	071205ER-208
9	SS7279	Calibration Standard, 0.5 ng/mL	1	071205ER\071205ER	071205ER-209
10	C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=100	11	071205ER\071205ER	071205ER-210
11	C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=10	12	071205ER\071205ER	071205ER-211
12	C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=100	13	071205ER\071205ER	071205ER-212
13	C0049881	DF06V02RCP0010041007, DF=100	14	071205ER\071205ER	071205ER-213
14	C0049881 Rep	DF06V02RCP0010041007, DF=100	15	071205ER\071205ER	071205ER-214
15	C0049881	DF06V02RCP0010041007, DF=10	16	071205ER\071205ER	071205ER-215
16	C0049881 Rep	DF06V02RCP0010041007, DF=10	17	071205ER\071205ER	071205ER-216
17	SS7278	Calibration Standard, 1.0 ng/mL	2	071205ER\071205ER	071205ER-217
18	C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=100	18	071205ER\071205ER	071205ER-218
19	C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=10	19	071205ER\071205ER	071205ER-219
20	C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=100	20	071205ER\071205ER	071205ER-220
21	C0049882	DF09V01AVP0010041007, DF=100	21	071205ER\071205ER	071205ER-221
22	C0049882 Rep	DF09V01AVP0010041007, DF=100	22	071205ER\071205ER	071205ER-222
23	C0049882	DF09V01AVP0010041007, DF=10	23	071205ER\071205ER	071205ER-223
24	C0049882 Rep	DF09V01AVP0010041007, DF=10	24	071205ER\071205ER	071205ER-224
25	SS7277	Calibration Standard, 2.5 ng/mL	3	071205ER\071205ER	071205ER-225
26	C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=100	25	071205ER\071205ER	071205ER-226
27	C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=10	26	071205ER\071205ER	071205ER-227
28	C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=100	27	071205ER\071205ER	071205ER-228
29	C0049883	DF09V01PAP0010041007, DF=100	28	071205ER\071205ER	071205ER-229
30	C0049883 Rep	DF09V01PAP0010041007, DF=100	29	071205ER\071205ER	071205ER-230
31	C0049883	DF09V01PAP0010041007, DF=10	30	071205ER\071205ER	071205ER-231
32	C0049883 Rep	DF09V01PAP0010041007, DF=10	31	071205ER\071205ER	071205ER-232
33	SS7276	Calibration Standard, 5.0 ng/mL	4	071205ER\071205ER	071205ER-233
34	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=100	32	071205ER\071205ER	071205ER-234
35	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=10	33	071205ER\071205ER	071205ER-235
36	C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=100	34	071205ER\071205ER	071205ER-236
37	C0049884	DF09V01RCP0010041007, DF=100	35	071205ER\071205ER	071205ER-237
38	C0049884 Rep	DF09V01RCP0010041007, DF=100	36	071205ER\071205ER	071205ER-238
39	C0049884	DF09V01RCP0010041007, DF=10	37	071205ER\071205ER	071205ER-239
40	C0049884 Rep	DF09V01RCP0010041007, DF=10	38	071205ER\071205ER	071205ER-240
41	SS7275	Calibration Standard, 10 ng/mL	5	071205ER\071205ER	071205ER-241
42	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=100	39	071205ER\071205ER	071205ER-242
43	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=10	40	071205ER\071205ER	071205ER-243
44	C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=100	41	071205ER\071205ER	071205ER-244
45	C0049885	DF09V02RCP0010041007, DF=100	42	071205ER\071205ER	071205ER-245
46	C0049885 Rep	DF09V02RCP0010041007, DF=100	43	071205ER\071205ER	071205ER-246
47	C0049885	DF09V02RCP0010041007, DF=10	44	071205ER\071205ER	071205ER-247

Analyst Version: 1.4.1
09:32:03 AM
Tuesday, July 26, 2005

Set No.: 071205ER
Exygen Study No.: P760/P1131

Page 2 of 2
Instrument No.: LC/MS/MS #9
Analyst: A. Sheehan

AMB 07/26/05

Project: D:\Analyst Data\Projects\P1131 on LCMSMS 9 Batch:071205ER VEG P760 Tab:Sample Set:SET1 AcqMethod:P760-1131 Isomer Separation 032
Sample

	Sample Name	Sample ID	Vial Position	Data File	Run #
48	C0049885 Rep	DF09V02RCP0010041007, DF=10	45	071205ER\071205ER	071205ER-248
49	SS7274	Calibration Standard, 25 ng/mL	6	071205ER\071205ER	071205ER-249
50	SS7273	Calibration Standard, 50 ng/mL	7	071205ER\071205ER	071205ER-250

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Study No: NA

Exygen Study No: P760/P1131

Instrument: API 4000 LC/MS/MS System, (LC/MS/MS #9)
Turbo Ion Spray Liquid Introduction Interface

Computer: DELL OptiPlex GX400

Software: Windows NT, Analyst 1.4.1

HPLC Equipment: 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 (Exygen ID MA0017077)

Column Temperature: 30°C

Mobile Phase (A) : 2 mM ammonium acetate in water (SL0014274)

Mobile Phase (B) : Methanol (RE0019344)

<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

Injected Volume: 15 µL

Ions monitored:

<u>Analyte</u>	<u>Mode</u>	<u>Transition Monitored</u>
PFBS	negative	299 → 99
PFHS	negative	399 → 80
PFOS	negative	499 → 80
PFOA	negative	413 → 369
PFOA Confirm Ion	negative	413 → 219
¹³ C PFOA	negative	415 → 370

Analyst: Amy Sheehan *AMB 07/26/05*
Exygen Research
3058 Research Drive, State College, PA 16801
Phone : (814) 272-1039 FAX : (814) 231-1580

NOTE: ALL HANDWRITTEN PEAK IDENTIFICATIONS BY *AMB / 07/28/05*

0368

CEE 07/28/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
1 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	125540	0.500	0.441	071205ER-201
2 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	231963	1.00	0.985	071205ER-202
3 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	559219	2.50	2.66	071205ER-203
4 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1075916	5.00	5.30	071205ER-204
5 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2152125	10.0	10.8	071205ER-205
6 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	4959601	25.0	25.2	071205ER-206
7 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	9469168	50.0	48.2	071205ER-207
8 Methanol Wash	Methanol Wash	Unknown	1	PFOA	0	N/A	No Peak	071205ER-208
9 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	113602	0.500	0.380	071205ER-209
10 C0049881 Spike C	DF09V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	634231	* 25.0	304.	071205ER-210
11 C0049881 Spike C	DF09V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	6318002	* 25.0	321.	071205ER-211
12 C0049881 Spike D	DF09V02RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	1030616	* 250.	607.	071205ER-212
13 C0049881	DF09V02RCP0010041007, DF=100	Unknown	100	PFOA	660379	* N/A	318.	071205ER-213
14 C0049881 Rep	DF09V02RCP0010041007, DF=100	Unknown	100	PFOA	738550	* N/A	358.	071205ER-214
15 C0049881	DF09V02RCP0010041007, DF=10	Unknown	10	PFOA	6041554	* N/A	307.	071205ER-215
16 C0049881 Rep	DF09V02RCP0010041007, DF=10	Unknown	10	PFOA	6807331	* N/A	346.	071205ER-216
17 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	240356	1.00	1.03	071205ER-217
18 C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	99967	* 25.0	31.0	071205ER-218
19 C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	921474	* 25.0	45.1	071205ER-219
20 C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	380526	* 250.	174.	071205ER-220
21 C0049882	DF09V01AVP0010041007, DF=100	Unknown	100	PFOA	53096	* N/A	7.05	071205ER-221
22 C0049882 Rep	DF09V01AVP0010041007, DF=100	Unknown	100	PFOA	34784	* N/A	< 0	071205ER-222
23 C0049882	DF09V01AVP0010041007, DF=10	Unknown	10	PFOA	565989	* N/A	26.9	071205ER-223
24 C0049882 Rep	DF09V01AVP0010041007, DF=10	Unknown	10	PFOA	556849	* N/A	26.5	071205ER-224
25 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	533753	2.50	2.53	071205ER-225
26 C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	355836	* 25.0	162.	071205ER-226
27 C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	3457978	* 25.0	175.	071205ER-227
28 C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	711564	* 250.	344.	071205ER-228
29 C0049883	DF09V01PAP0010041007, DF=100	Unknown	100	PFOA	312691	* N/A	140.	071205ER-229
30 C0049883 Rep	DF09V01PAP0010041007, DF=100	Unknown	100	PFOA	332124	* N/A	150.	071205ER-230
31 C0049883	DF09V01PAP0010041007, DF=10	Unknown	10	PFOA	3068314	* N/A	155.	071205ER-231
32 C0049883 Rep	DF09V01PAP0010041007, DF=10	Unknown	10	PFOA	3210366	* N/A	162.	071205ER-232
33 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1098166	5.00	5.41	071205ER-233
34 C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	280192	* 25.0	123.	071205ER-234
35 C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	2875699	* 25.0	145.	071205ER-235
36 C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	760917	* 250.	369.	071205ER-236
37 C0049884	DF09V01RCP0010041007, DF=100	Unknown	100	PFOA	240782	* N/A	103.	071205ER-237
38 C0049884 Rep	DF09V01RCP0010041007, DF=100	Unknown	100	PFOA	240655	* N/A	103.	071205ER-238
39 C0049884	DF09V01RCP0010041007, DF=10	Unknown	10	PFOA	2499185	* N/A	128.	071205ER-239
40 C0049884 Rep	DF09V01RCP0010041007, DF=10	Unknown	10	PFOA	2432541	* N/A	122.	071205ER-240
41 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2158798	10.0	10.8	071205ER-241
42 C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	846576	* 25.0	413.	071205ER-242
43 C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	8090144	* 25.0	412.	071205ER-243
44 C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	1243986	* 250.	616.	071205ER-244
45 C0049885	DF09V02RCP0010041007, DF=100	Unknown	100	PFOA	740390	* N/A	358.	071205ER-245
46 C0049885 Rep	DF09V02RCP0010041007, DF=100	Unknown	100	PFOA	835235	* N/A	407.	071205ER-246
47 C0049885	DF09V02RCP0010041007, DF=10	Unknown	10	PFOA	7352275	* N/A	374.	071205ER-247
48 C0049885 Rep	DF09V02RCP0010041007, DF=10	Unknown	10	PFOA	8117333	* N/A	413.	071205ER-248
49 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	6011716	25.0	25.4	071205ER-249
50 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	9590978	50.0	48.8	071205ER-250

* Analysis not required. *BTK 07/28/05*

Vegetation Conversion

Compound: PFOA

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049881	DF06-V02-RCP001-0-041007	307	123
C0049881 Rep	DF06-V02-RCP001-0-041007	346	138
C0049881 Spk C	DF06-V02-RCP001-0-041007	321	128
C0049883	DF09-V01-PAP001-0-041007	155	62.0
C0049883 Rep	DF09-V01-PAP001-0-041007	162	64.8
C0049883 Spk G	DF09-V01-PAP001-0-041007	175	70.0
C0049884	DF09-V01-RCP001-0-041007	126	50.4
C0049884 Rep	DF09-V01-RCP001-0-041007	122	48.8
C0049884 Spk I	DF09-V01-RCP001-0-041007	145	58.0
C0049885	DF09-V02-RCP001-0-041007	374	150
C0049885 Rep	DF09-V02-RCP001-0-041007	413	165
C0049885 Spk K	DF09-V02-RCP001-0-041007	412	165
C0049885 Spk L	DF09-V02-RCP001-0-041007	616	246

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = ~~Peak Area = 0~~ At or Above 0.2 ng/g @

NQ = Not quantifiable = Measured concentration below Limit of Quantitation (LOQ) which is 0.8 ng/g.

A
BETWEEN 0.2 ng/g AND @ 40

ORE MLC 9/20/05

0370

BAK 07/20/05

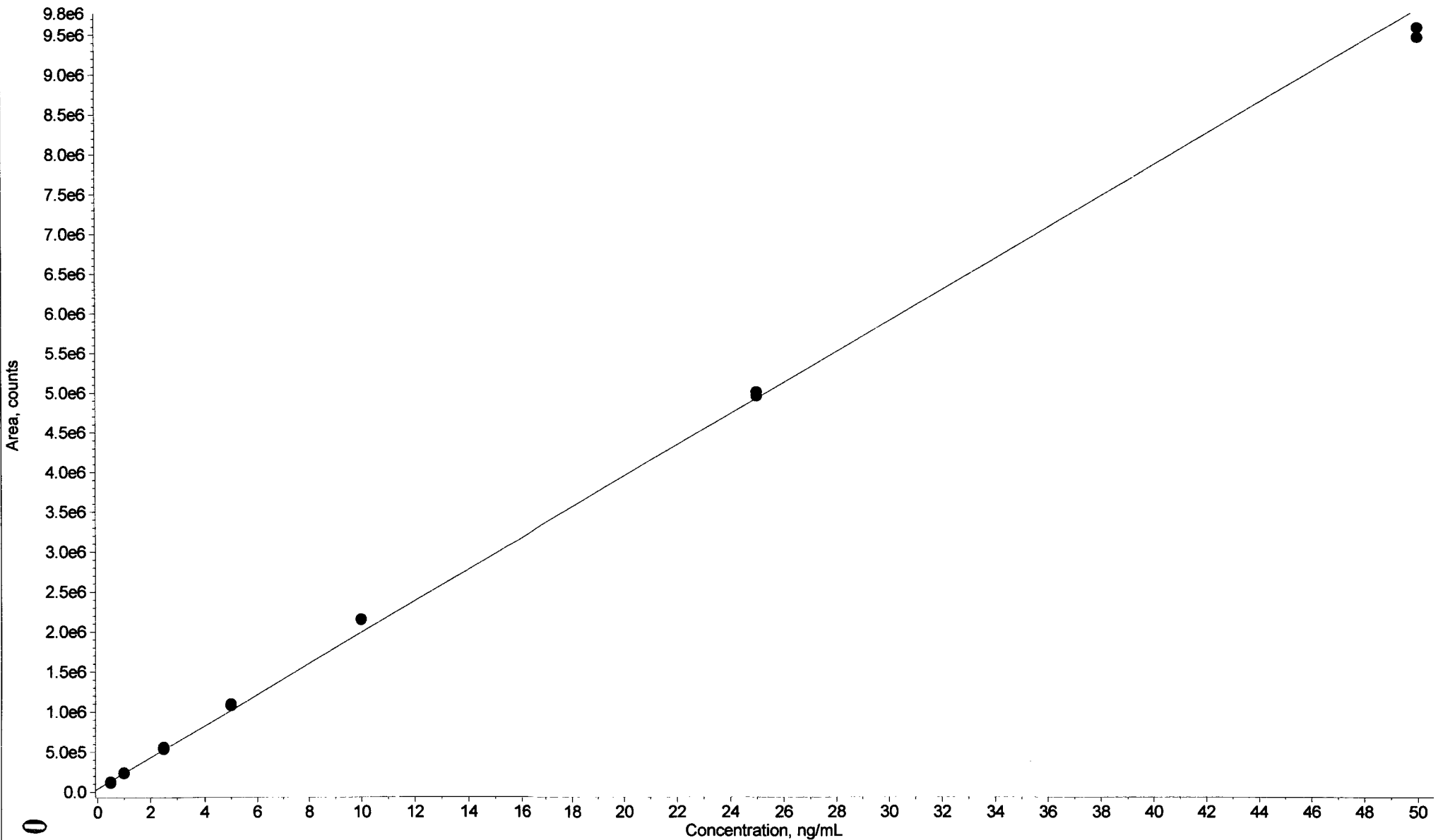
	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound: PFOA		Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049881	DF06-V02-RCP001-0-041007	=Raw Data!H124	=(C9*2)/5
10	C0049881 Rep	DF06-V02-RCP001-0-041007	=Raw Data!H125	=(C10*2)/5
11	C0049881 Spk C	DF06-V02-RCP001-0-041007	=Raw Data!H120	=(C11*2)/5
12				
13	C0049883	DF09-V01-PAP001-0-041007	=Raw Data!H140	=(C13*2)/5
14	C0049883 Rep	DF09-V01-PAP001-0-041007	=Raw Data!H141	=(C14*2)/5
15	C0049883 Spk G	DF09-V01-PAP001-0-041007	=Raw Data!H136	=(C15*2)/5
16				
17	C0049884	DF09-V01-RCP001-0-041007	=Raw Data!H148	=(C17*2)/5
18	C0049884 Rep	DF09-V01-RCP001-0-041007	=Raw Data!H149	=(C18*2)/5
19	C0049884 Spk I	DF09-V01-RCP001-0-041007	=Raw Data!H144	=(C19*2)/5
20				
21	C0049885	DF09-V02-RCP001-0-041007	=Raw Data!H156	=(C21*2)/5
22	C0049885 Rep	DF09-V02-RCP001-0-041007	=Raw Data!H157	=(C22*2)/5
23	C0049885 Spk K	DF09-V02-RCP001-0-041007	=Raw Data!H152	=(C23*2)/5
24	C0049885 Spk L	DF09-V02-RCP001-0-041007	=Raw Data!H153	=(C24*2)/5
25				

0371

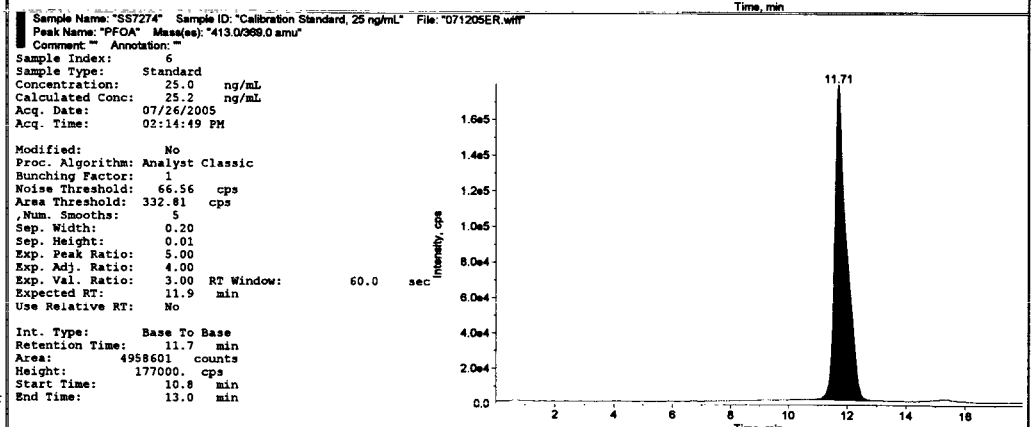
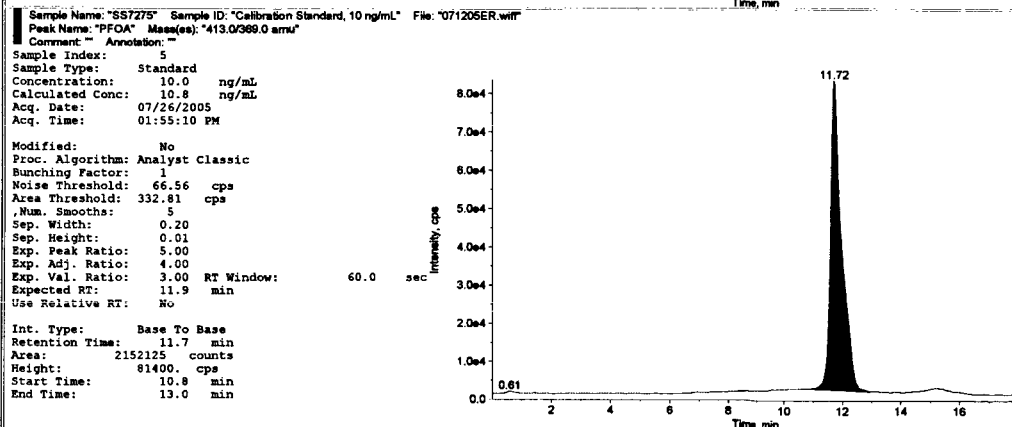
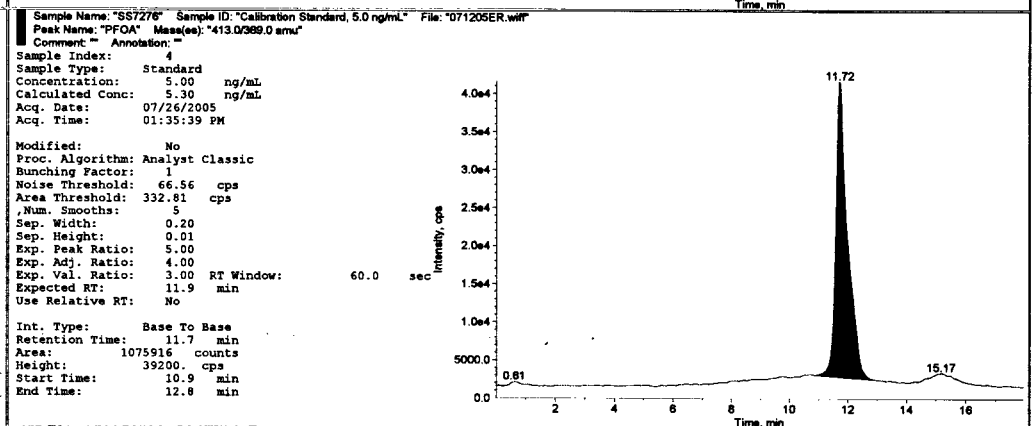
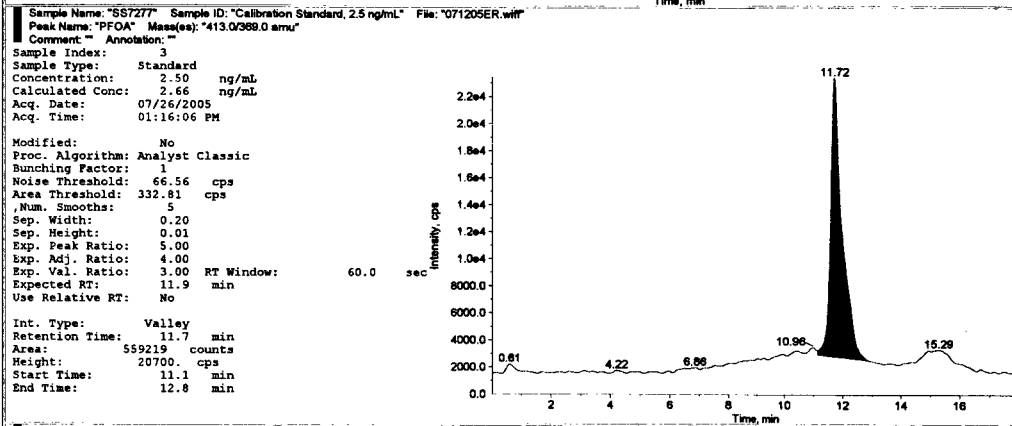
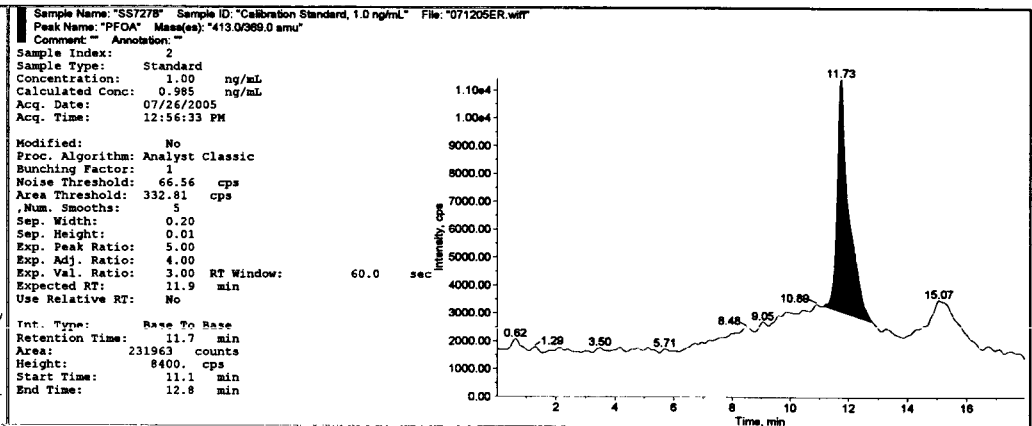
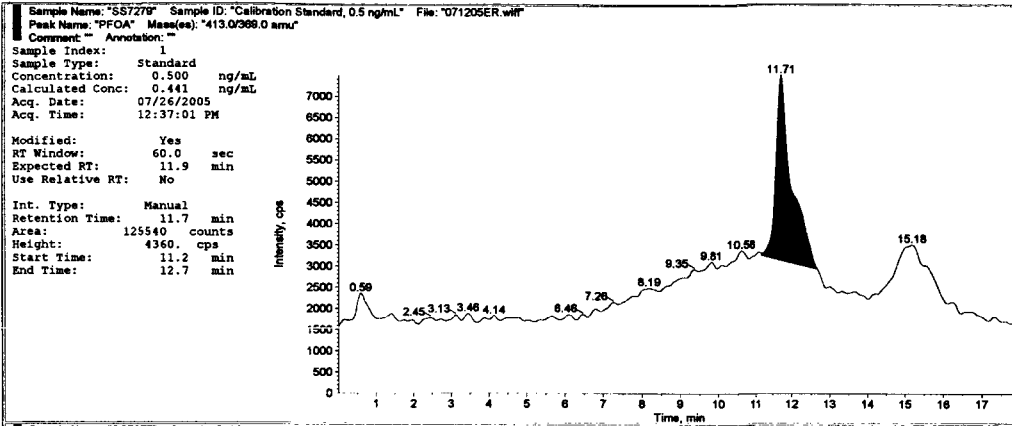
BML 07/28/05

AMB 07/28/05

■ Untitled 1 (PFOA): "Linear" Regression ("1 / x" weighting): $y = 1.96e+005 x + 3.93e+004$ ($r = 0.9990$)

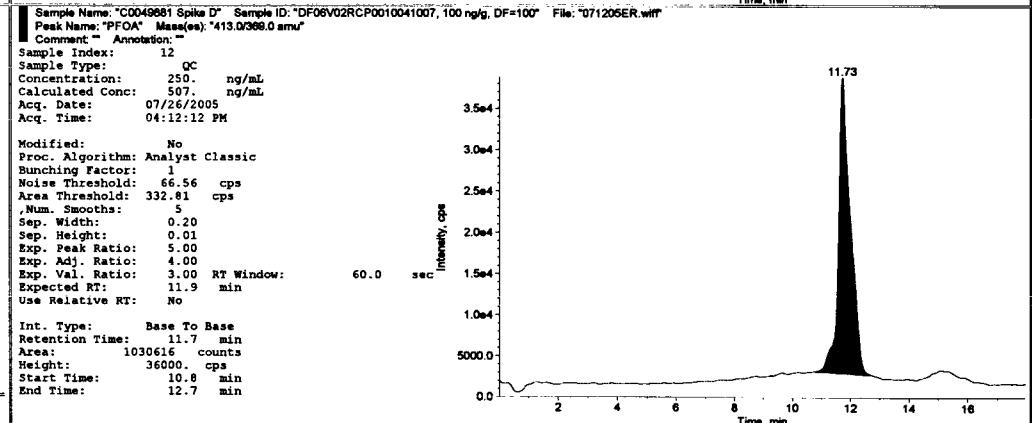
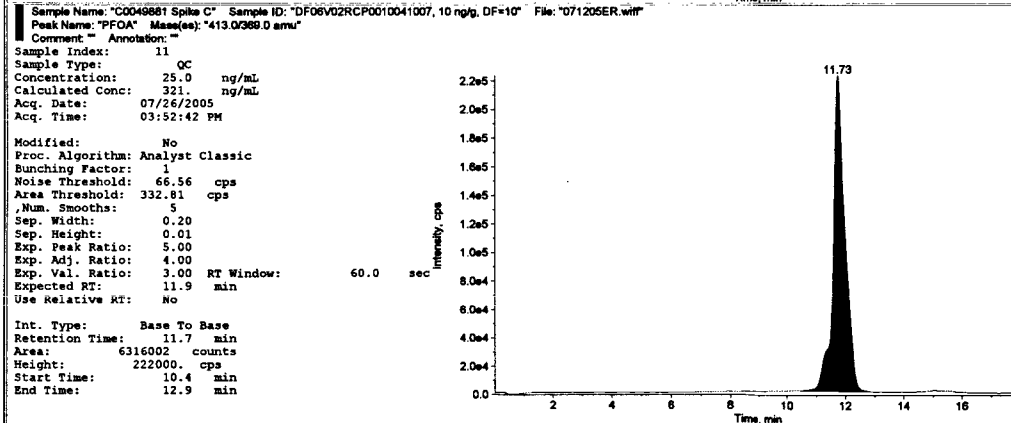
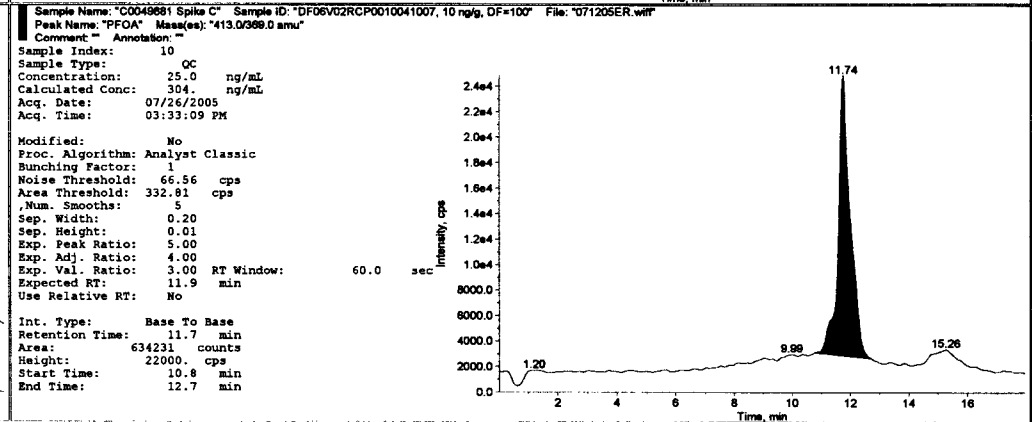
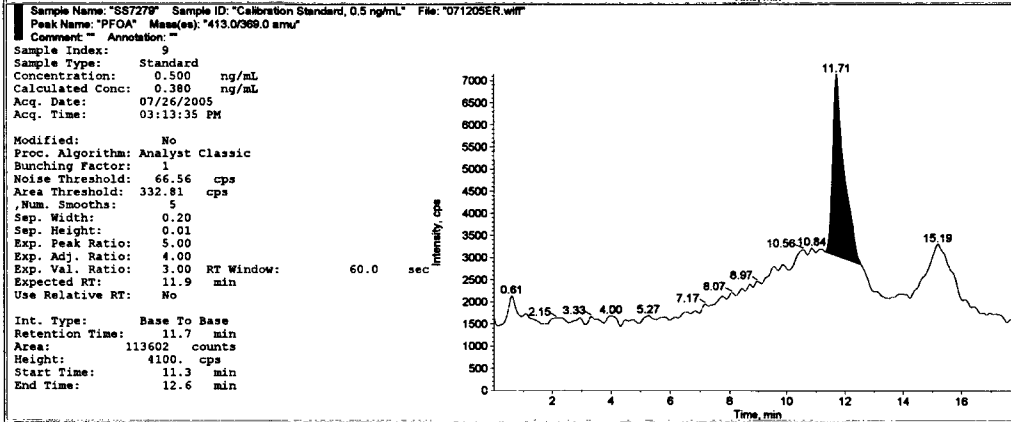
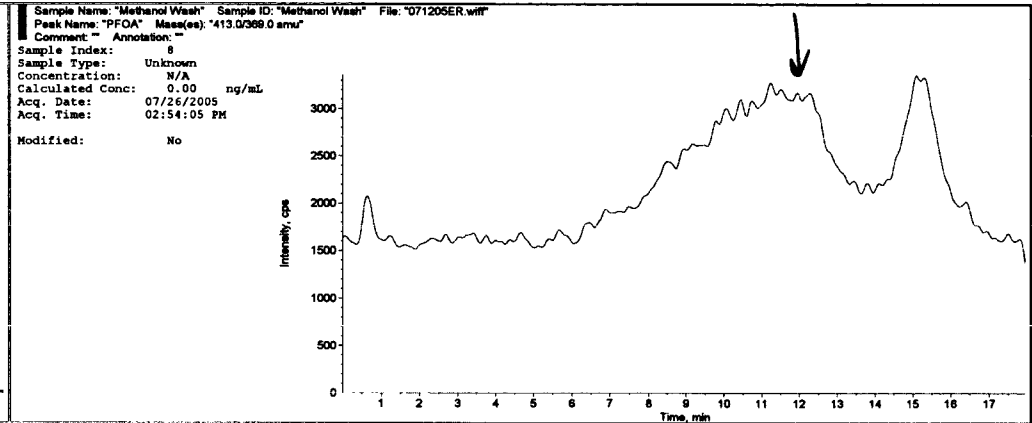
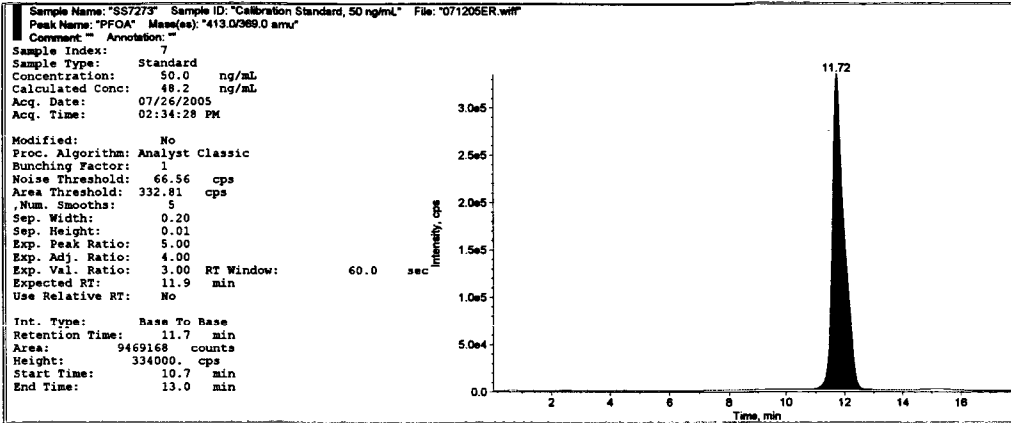


0372

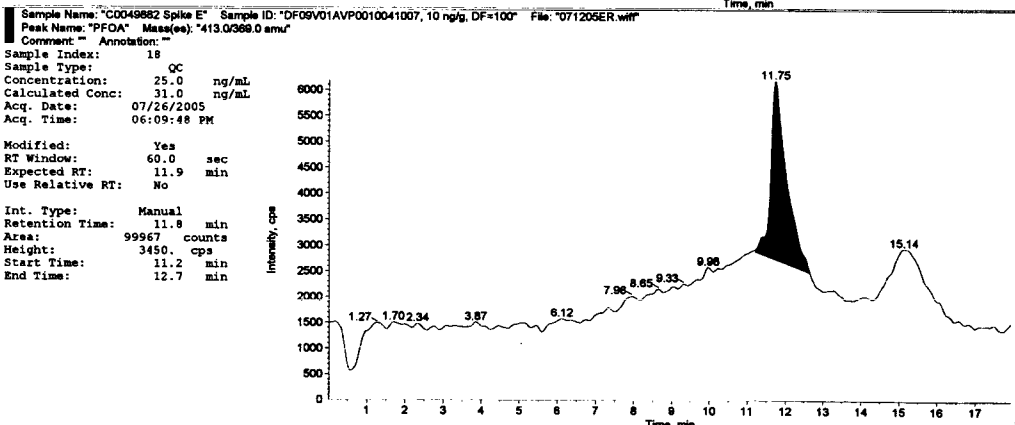
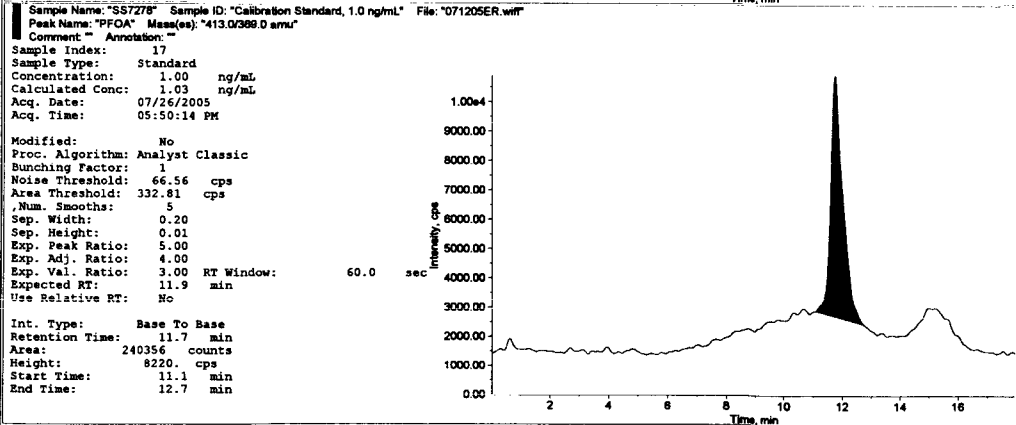
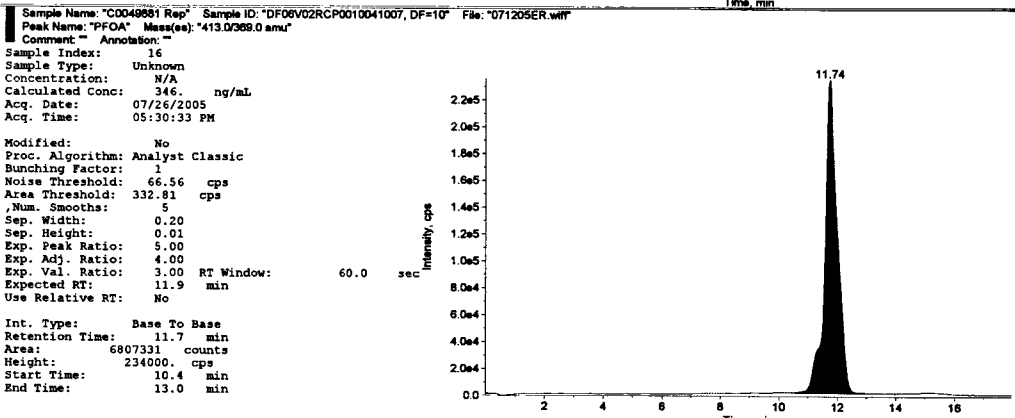
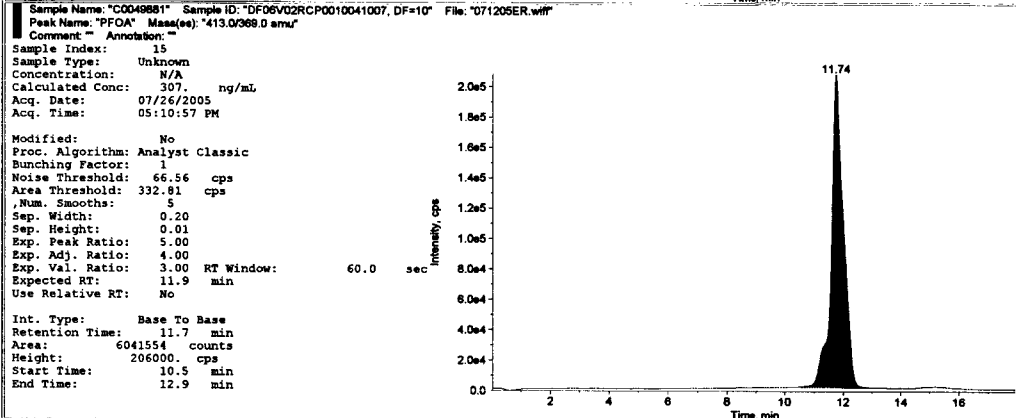
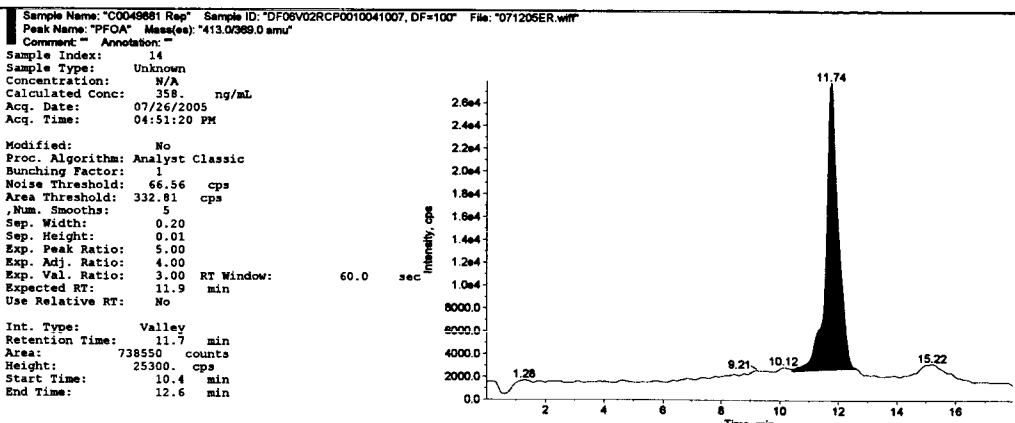
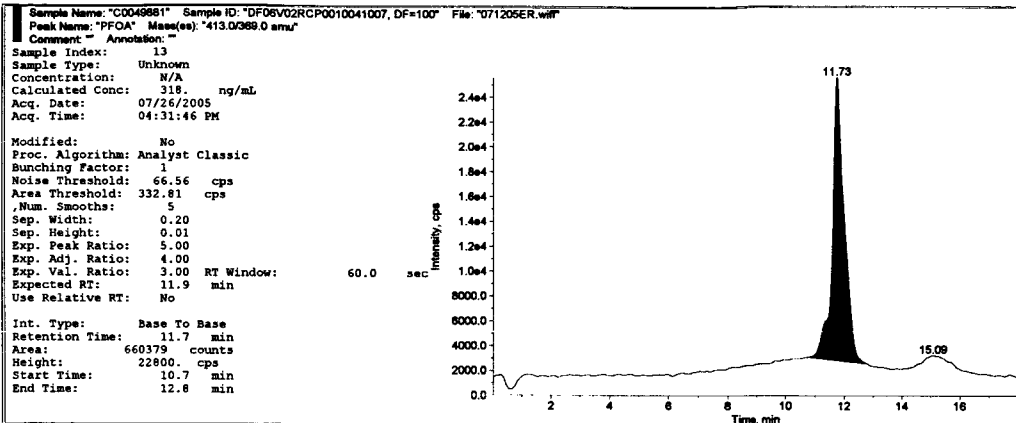


3260 Initials AMB
 Date 07/28/05
 Run# 1 To 50
 Sample Index 1 To 50
 @ AMB 07/28/05

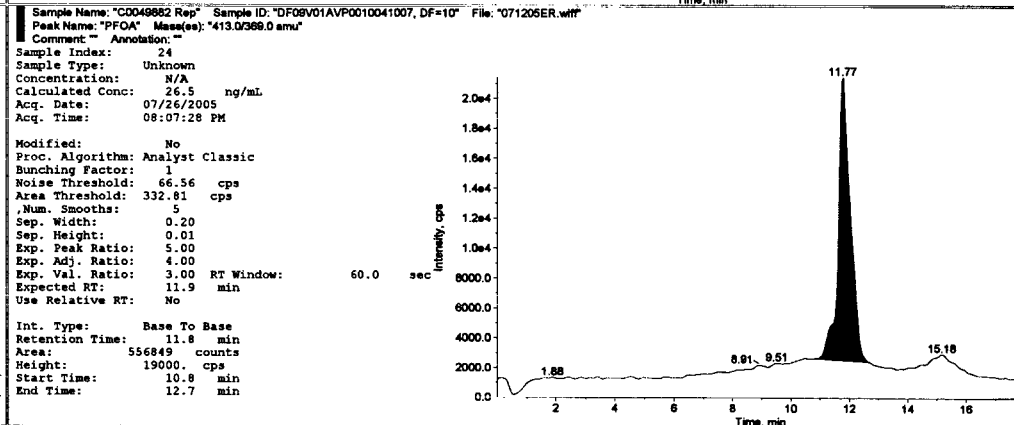
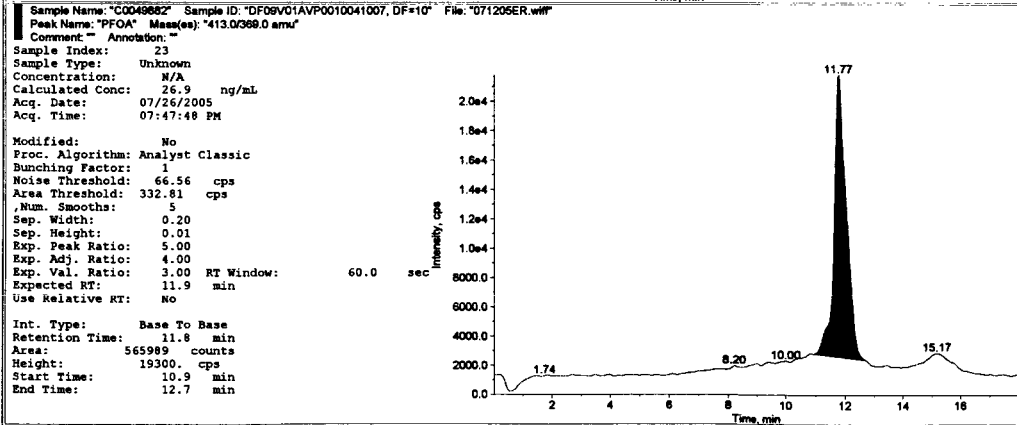
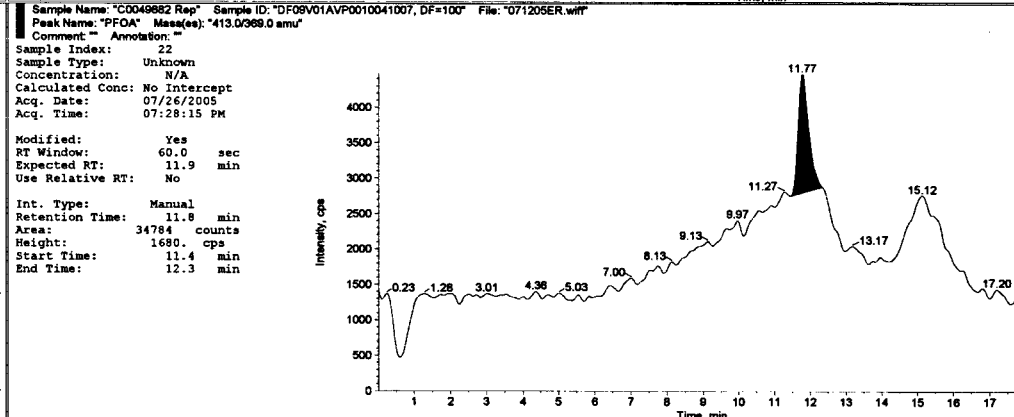
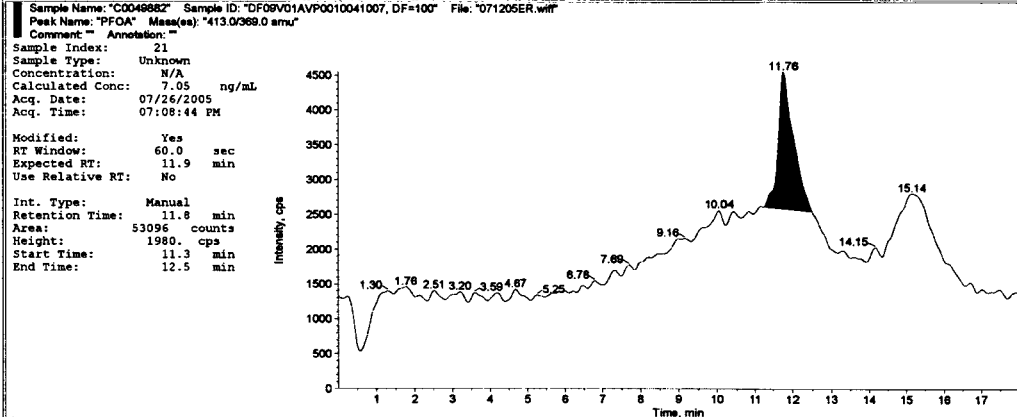
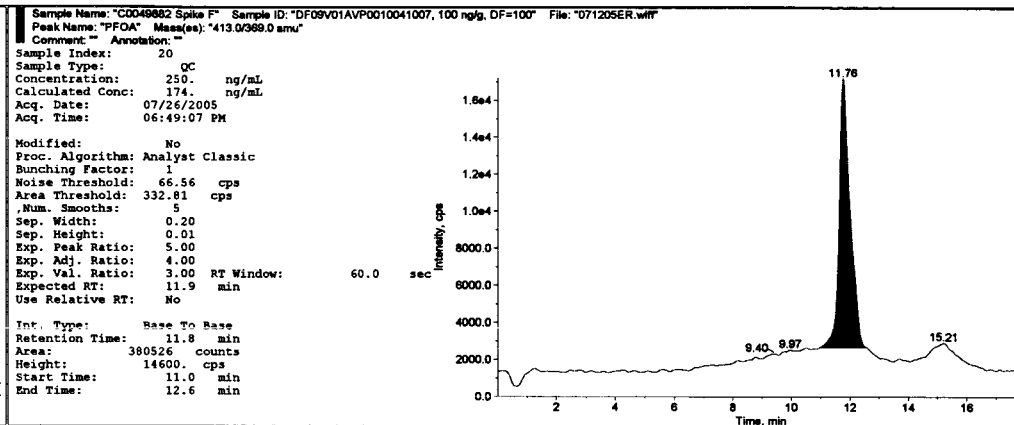
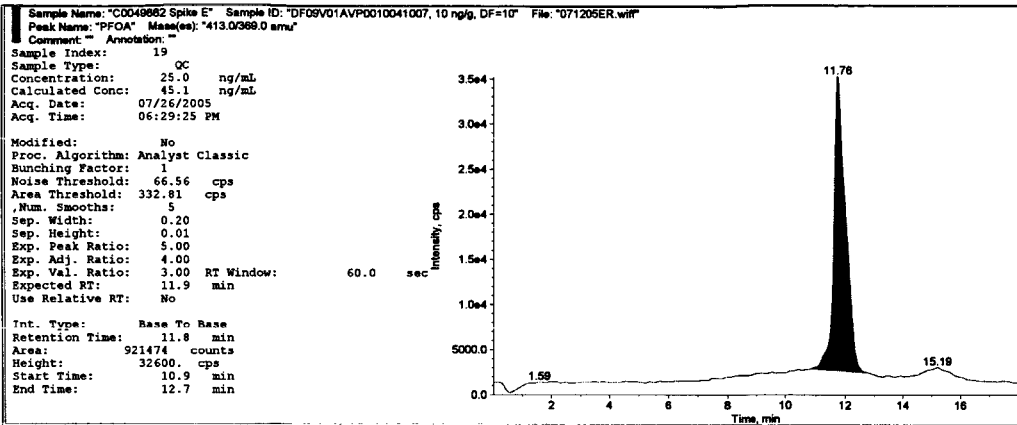
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 Initials: AS Date 09/22/05
 Sample Index: 1 To: 50



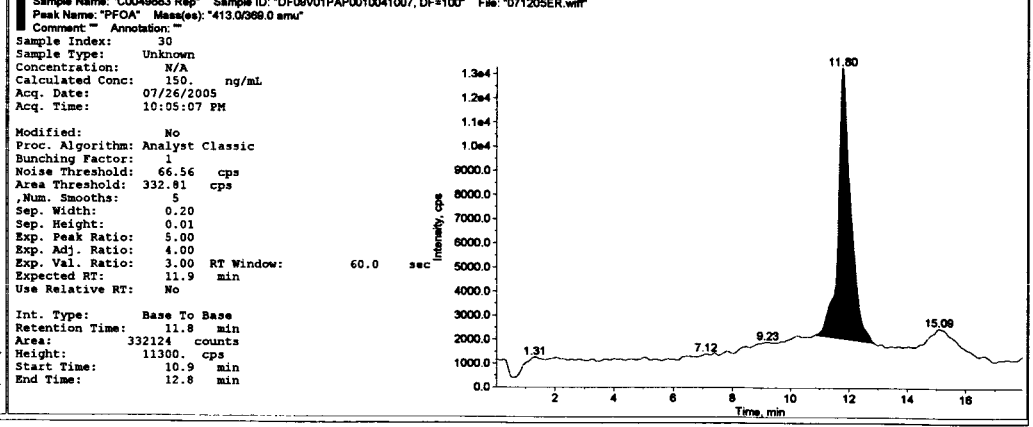
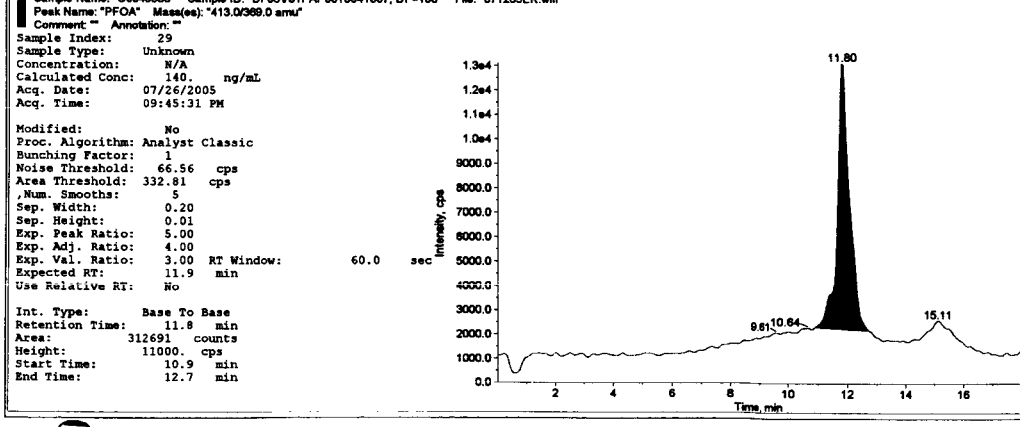
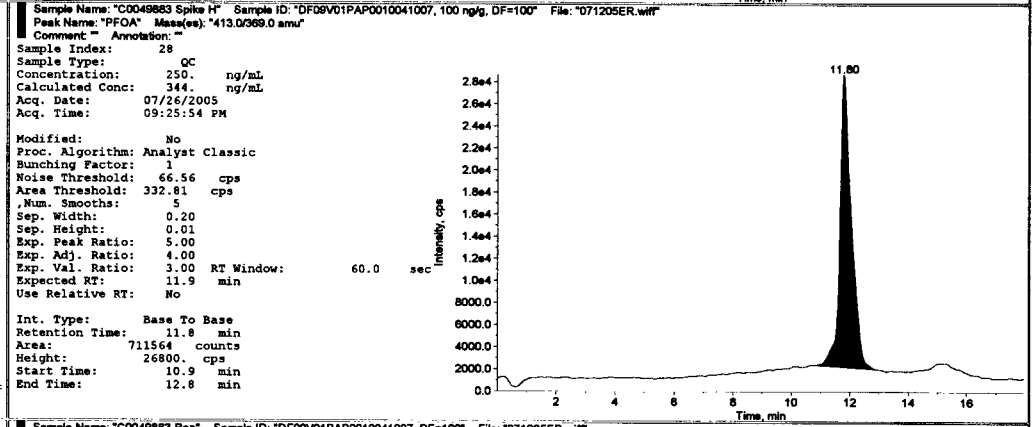
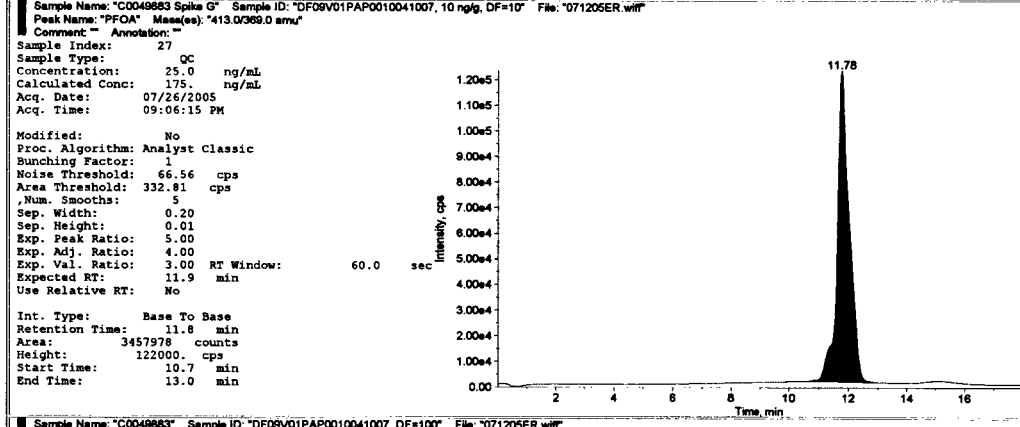
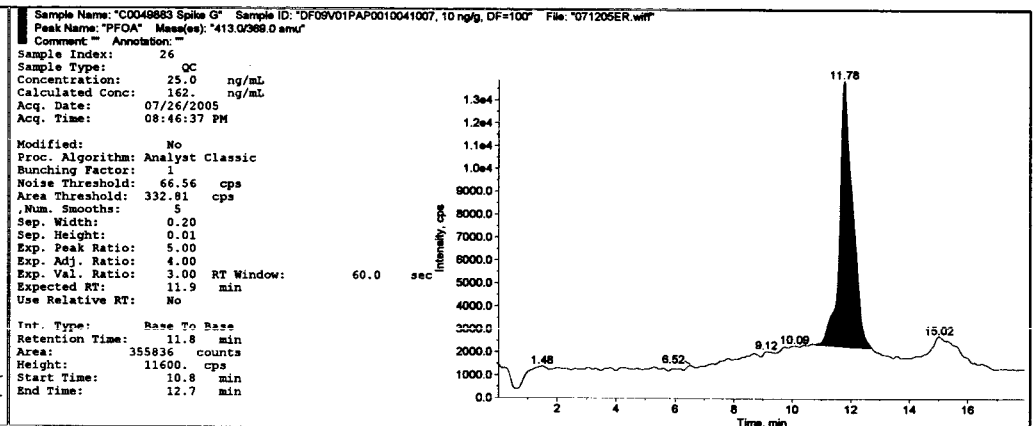
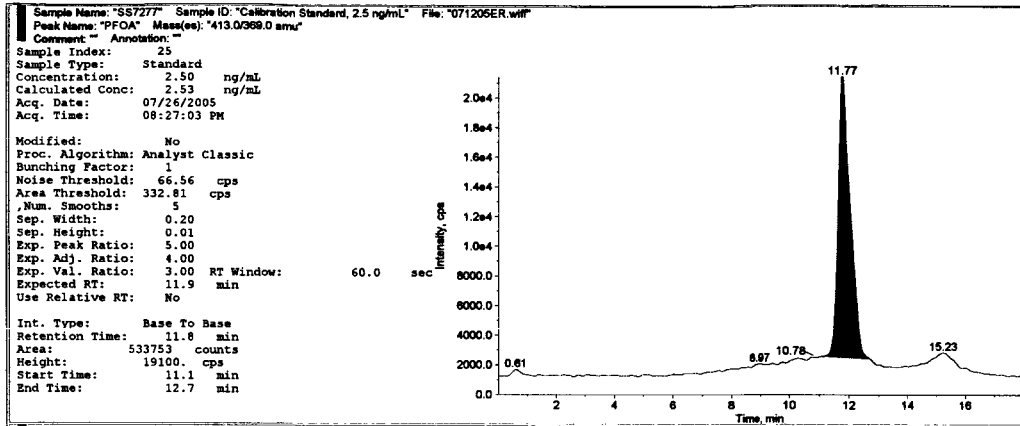
0374



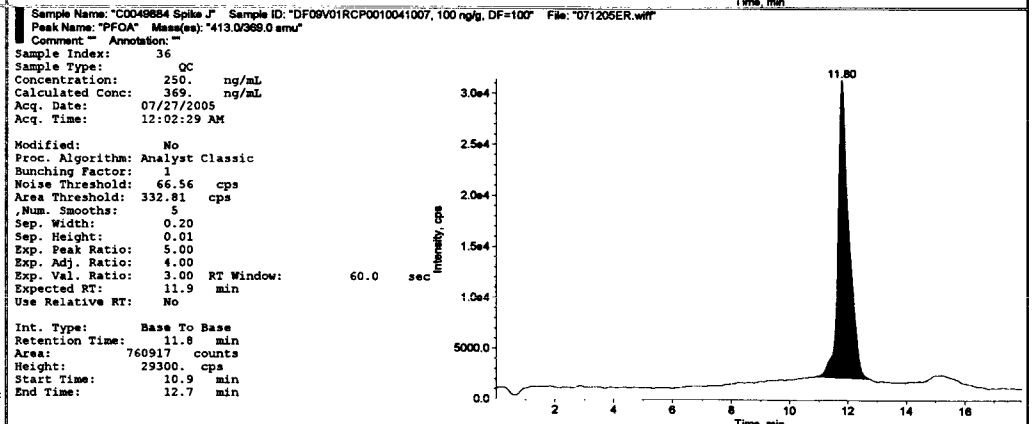
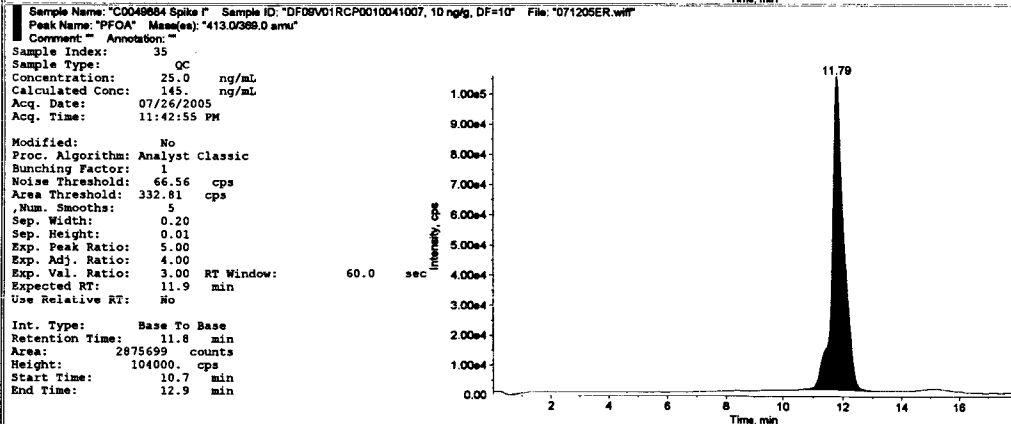
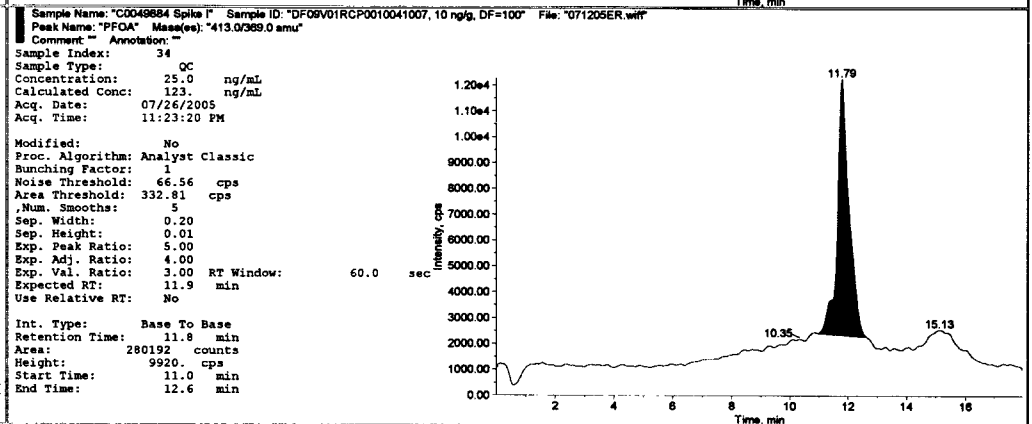
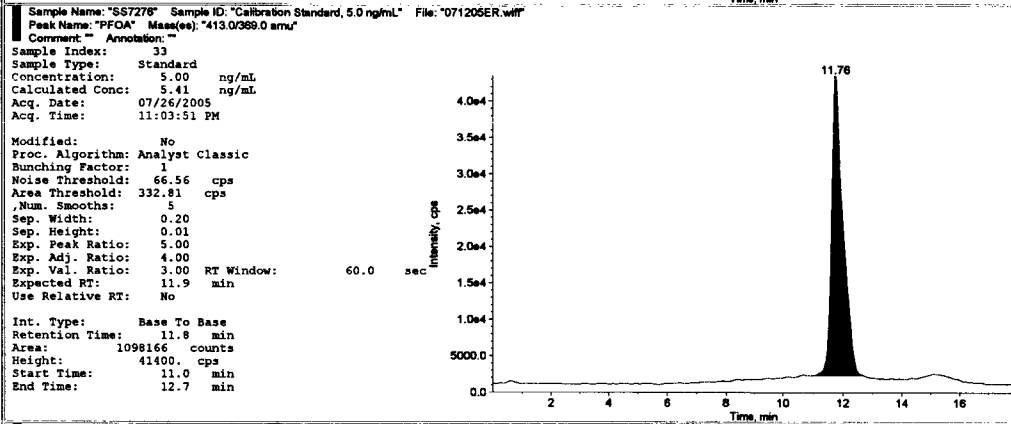
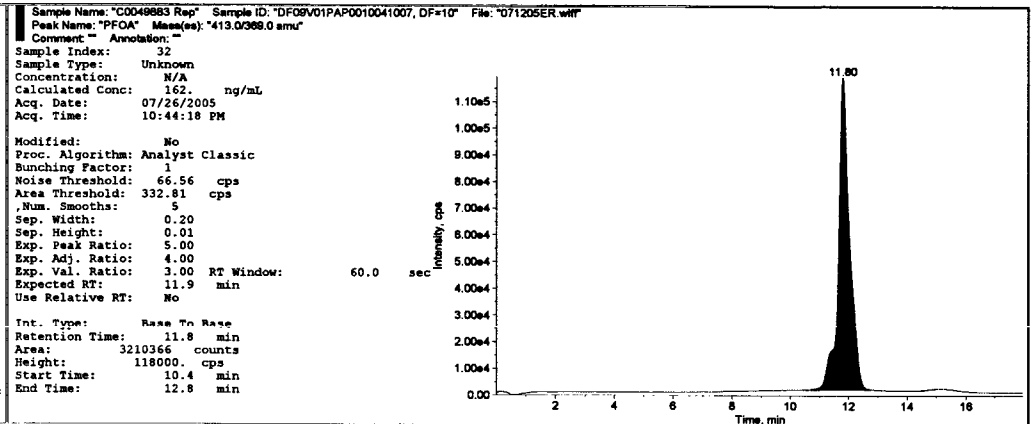
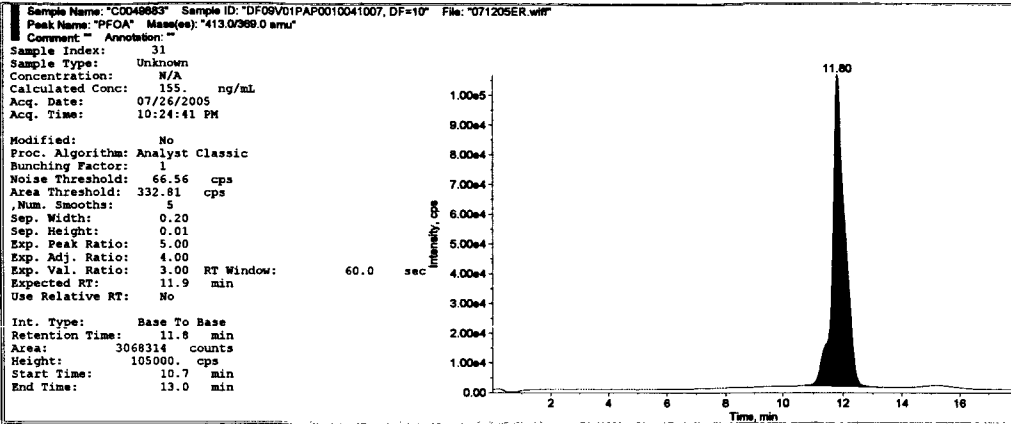
0375



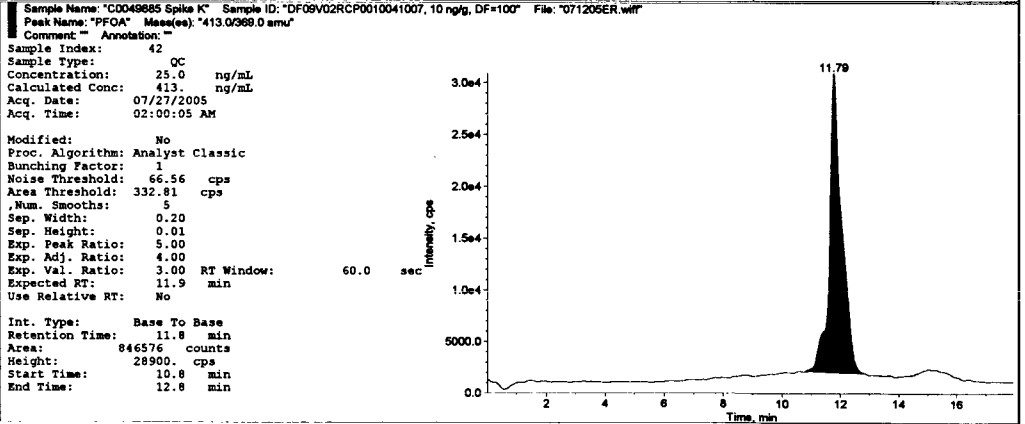
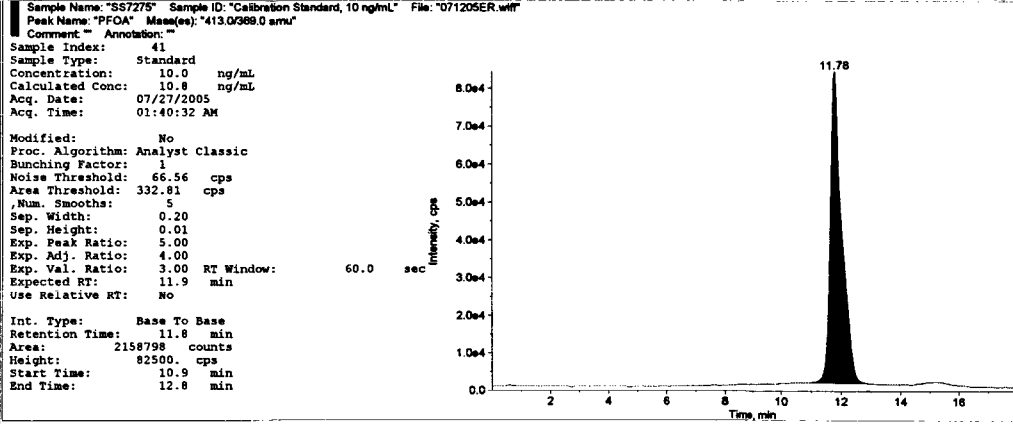
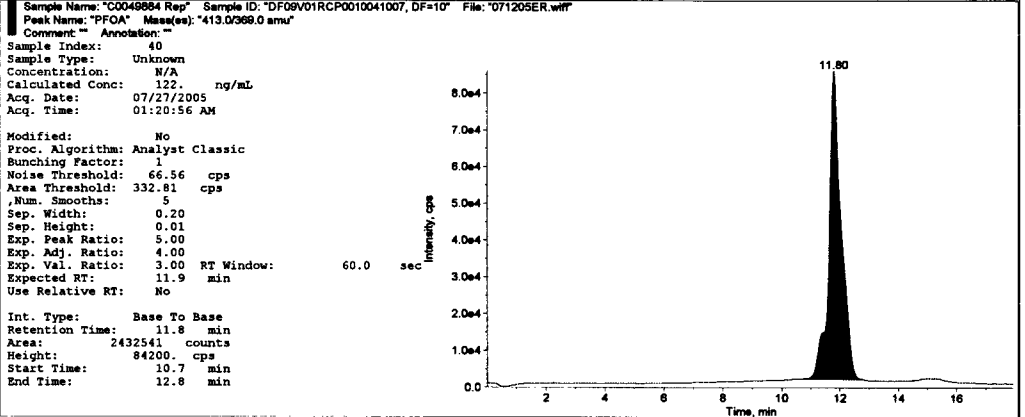
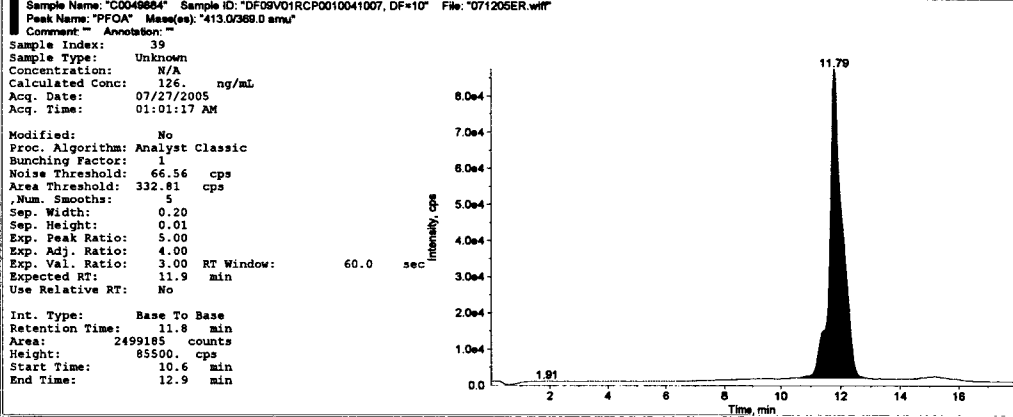
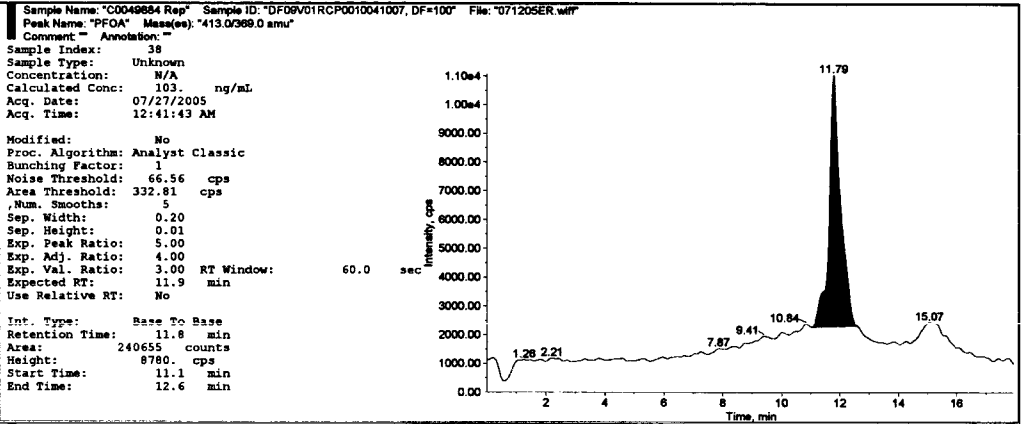
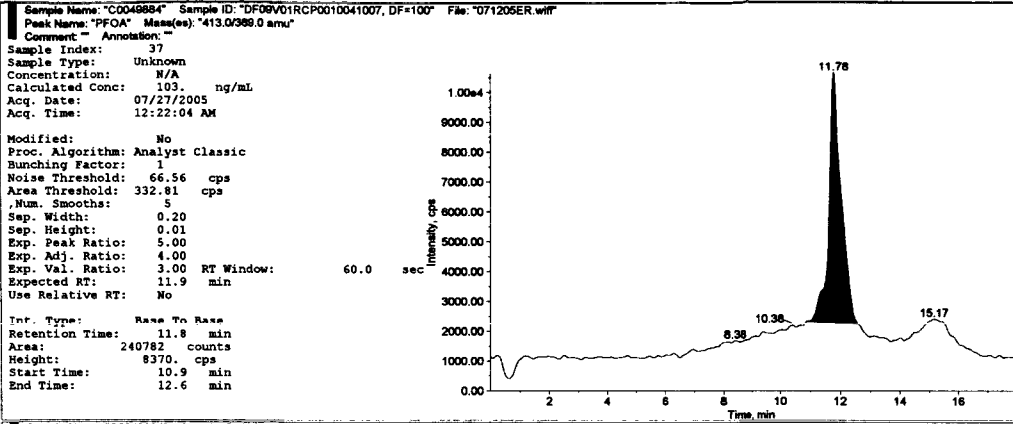
0376



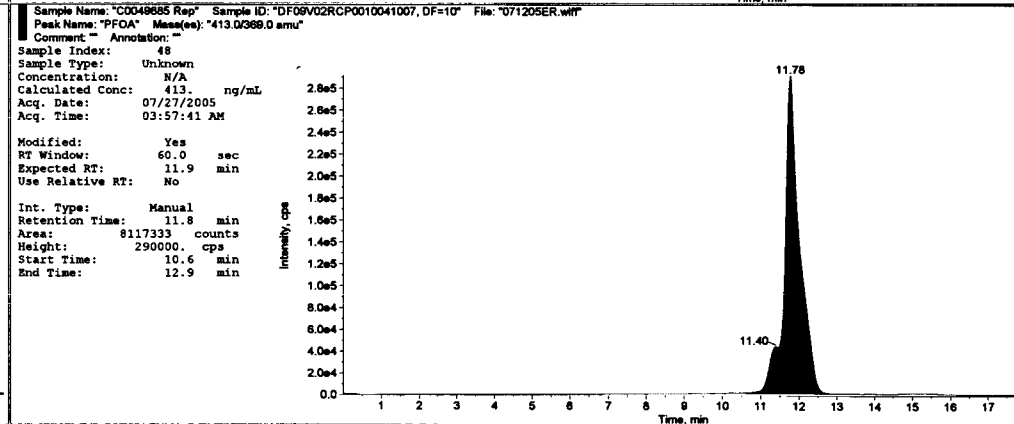
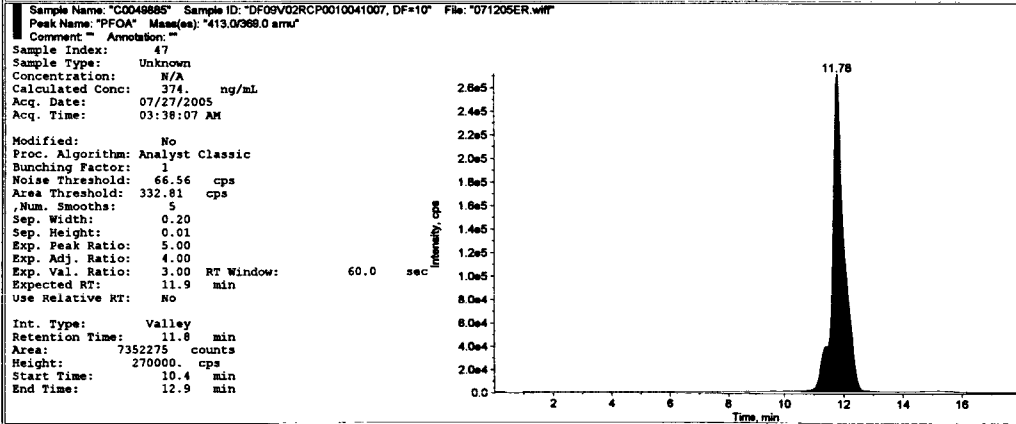
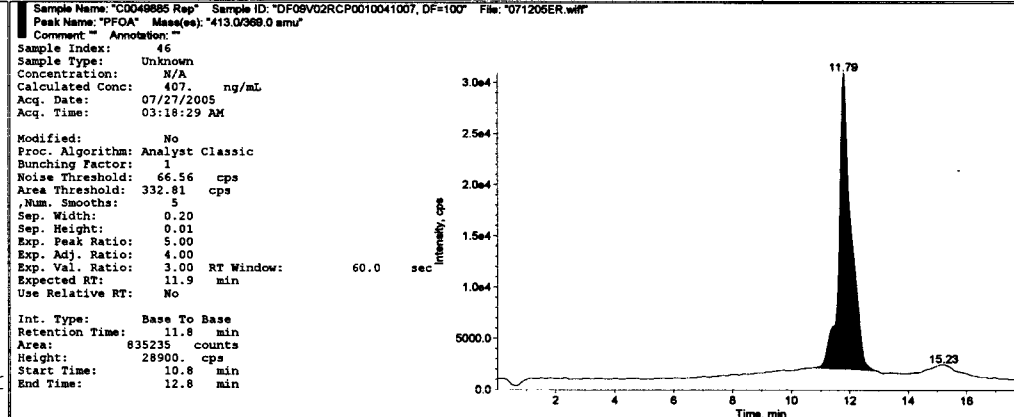
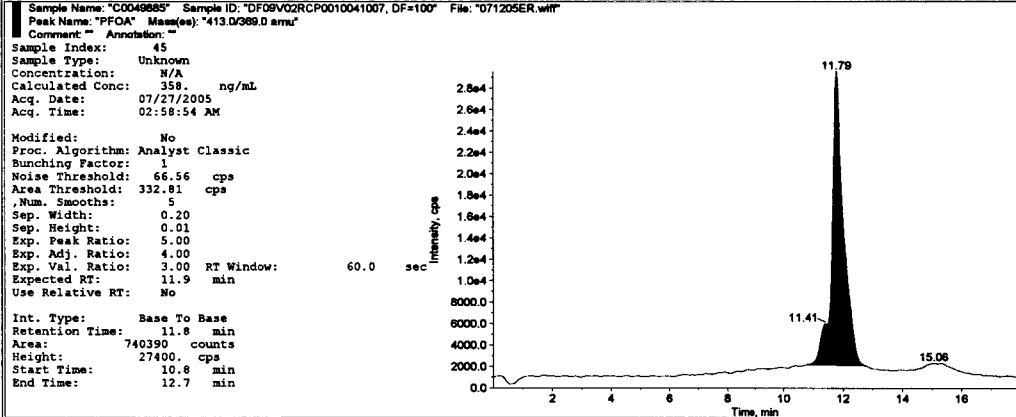
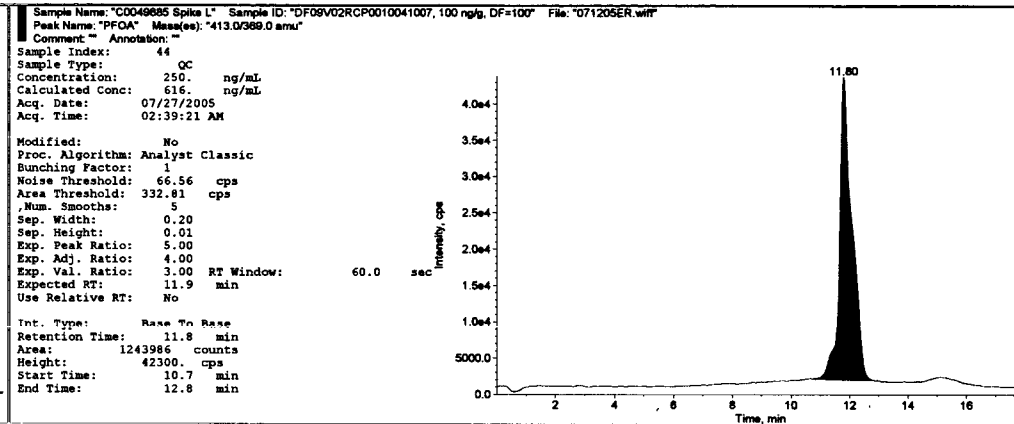
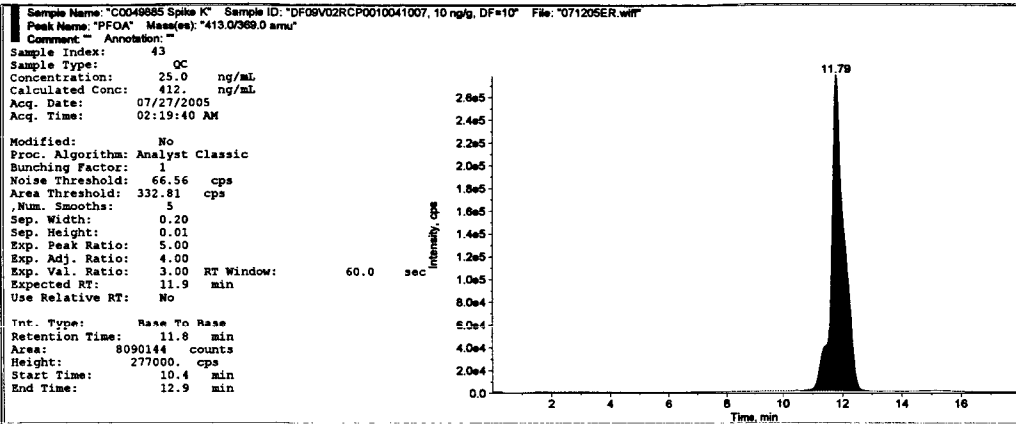
0377



0378



0379



030

Sample Name: "SS7274" Sample ID: "Calibration Standard, 25 ng/mL" File: "071205ER.wif"

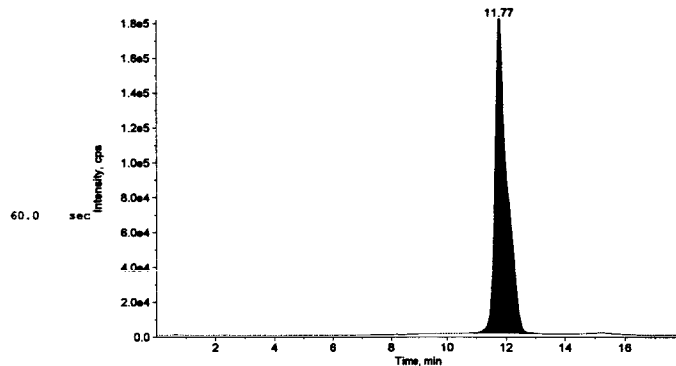
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"

Comment: " Annotation: "

Sample Index: 49
Sample Type: Standard
Concentration: 25.0 ng/mL
Calculated Conc: 25.4 ng/mL
Acq. Date: 07/27/2005
Acq. Time: 04:17:20 AM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 66.56 cps
Area Threshold: 332.81 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.9 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 11.8 min
Area: 5011716 counts
Height: 182000 cps
Start Time: 10.8 min
End Time: 13.0 min



Sample Name: "SS7273" Sample ID: "Calibration Standard, 50 ng/mL" File: "071205ER.wif"

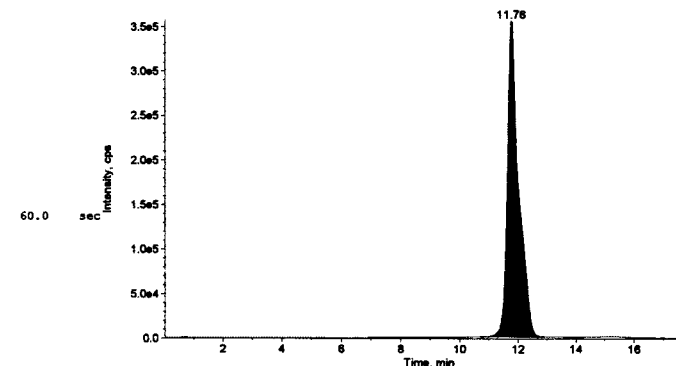
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"

Comment: " Annotation: "

Sample Index: 50
Sample Type: Standard
Concentration: 50.0 ng/mL
Calculated Conc: 48.8 ng/mL
Acq. Date: 07/27/2005
Acq. Time: 04:36:54 AM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 66.56 cps
Area Threshold: 332.81 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.9 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 11.8 min
Area: 9590978 counts
Height: 359000 cps
Start Time: 10.7 min
End Time: 12.9 min



0381

CEE 07/28/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
1 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	2713	0.500	0.617	071205ER-201
2 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	3234	1.00	0.833	071205ER-202
3 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	7510	2.50	2.60	071205ER-203
4 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	12260	5.00	4.57	071205ER-204
5 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	26328	10.0	10.4	071205ER-205
6 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	65267	25.0	26.5	071205ER-206
7 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	120526	50.0	49.4	071205ER-207
8 Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	372	N/A	< 0	071205ER-208
9 SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	2441	0.500	0.505	071205ER-209
10 C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	21883	* 25.0	855.	071205ER-210
11 C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	221450	* 25.0	912.	071205ER-211
12 C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	29002	* 250.	1150.	071205ER-212
13 C0049881	DF06V02RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	25094	* N/A	988.	071205ER-213
14 C0049881 Rep	DF06V02RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	27059	* N/A	1070.	071205ER-214
15 C0049881	DF06V02RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	218549	* N/A	900.	071205ER-215
16 C0049881 Rep	DF06V02RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	247284	* N/A	1020.	071205ER-216
17 SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	3792	1.00	1.06	071205ER-217
18 C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	2847	* 25.0	67.3	071205ER-218
19 C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	22811	* 25.0	89.4	071205ER-219
20 C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	7307	* 250.	252.	071205ER-220
21 C0049882	DF09V01AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	2150	* N/A	38.4	071205ER-221
22 C0049882 Rep	DF09V01AVP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	5168	* N/A	163.	071205ER-222
23 C0049882	DF09V01AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	18584	* N/A	71.9	071205ER-223
24 C0049882 Rep	DF09V01AVP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	20915	* N/A	81.5	071205ER-224
25 SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	6756	2.50	2.29	071205ER-225
26 C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	11632	* 25.0	431.	071205ER-226
27 C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	102006	* 25.0	417.	071205ER-227
28 C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	13256	* 250.	498.	071205ER-228
29 C0049883	DF09V01PAP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	10255	* N/A	374.	071205ER-229
30 C0049883 Rep	DF09V01PAP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	9926	* N/A	360.	071205ER-230
31 C0049883	DF09V01PAP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	102439	* N/A	419.	071205ER-231
32 C0049883 Rep	DF09V01PAP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	106570	* N/A	432.	071205ER-232
33 SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	11299	5.00	4.17	071205ER-233
34 C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	9961	* 25.0	362.	071205ER-234
35 C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	95977	* 25.0	392.	071205ER-235
36 C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	17188	* 250.	661.	071205ER-236
37 C0049884	DF09V01RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	9967	* N/A	362.	071205ER-237
38 C0049884 Rep	DF09V01RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	9446	* N/A	340.	071205ER-238
39 C0049884	DF09V01RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	87602	* N/A	358.	071205ER-239
40 C0049884 Rep	DF09V01RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	92909	* N/A	380.	071205ER-240
41 SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	27085	10.0	10.7	071205ER-241
42 C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	27826	* 25.0	1100.	071205ER-242
43 C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	272325	* 25.0	1120.	071205ER-243
44 C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA Confirm Ion	34228	* 250.	1370.	071205ER-244
45 C0049885	DF09V02RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	24787	* N/A	975.	071205ER-245
46 C0049885 Rep	DF09V02RCP0010041007, DF=100	Unknown	100	PFOA Confirm Ion	27153	* N/A	1070.	071205ER-246
47 C0049885	DF09V02RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	244370	* N/A	1010.	071205ER-247
48 C0049885 Rep	DF09V02RCP0010041007, DF=10	Unknown	10	PFOA Confirm Ion	278198	* N/A	1140.	071205ER-248
49 SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	62848	25.0	25.5	071205ER-249
50 SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	119203	50.0	48.8	071205ER-250

* Analysis not required. BAK 07/28/05

Vegetation Conversion

Compound: PFOA Confirm. Ion

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049881	DF06-V02-RCP001-0-041007	900	360
C0049881 Rep	DF06-V02-RCP001-0-041007	1020	408
C0049881 Spk C	DF06-V02-RCP001-0-041007	912	365
C0049883	DF09-V01-PAP001-0-041007	419	168
C0049883 Rep	DF09-V01-PAP001-0-041007	432	173
C0049883 Spk G	DF09-V01-PAP001-0-041007	417	167
C0049884	DF09-V01-RCP001-0-041007	358	143
C0049884 Rep	DF09-V01-RCP001-0-041007	380	152
C0049884 Spk I	DF09-V01-RCP001-0-041007	392	157
C0049885	DF09-V02-RCP001-0-041007	1010	404
C0049885 Rep	DF09-V02-RCP001-0-041007	1140	456
C0049885 Spk K	DF09-V02-RCP001-0-041007	1120	448
C0049885 Spk L	DF09-V02-RCP001-0-041007	1370	548

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = Peak Area 0 At or Above 0.2ng/g (C)

NQ = Not quantifiable = Measured concentration below Limit of Quantitation (LOQ) which is 0.6 ng/g.

^
BETWEEN 0.2ng/g AND (C)

40

(RE) MLC 9/20/05

0383

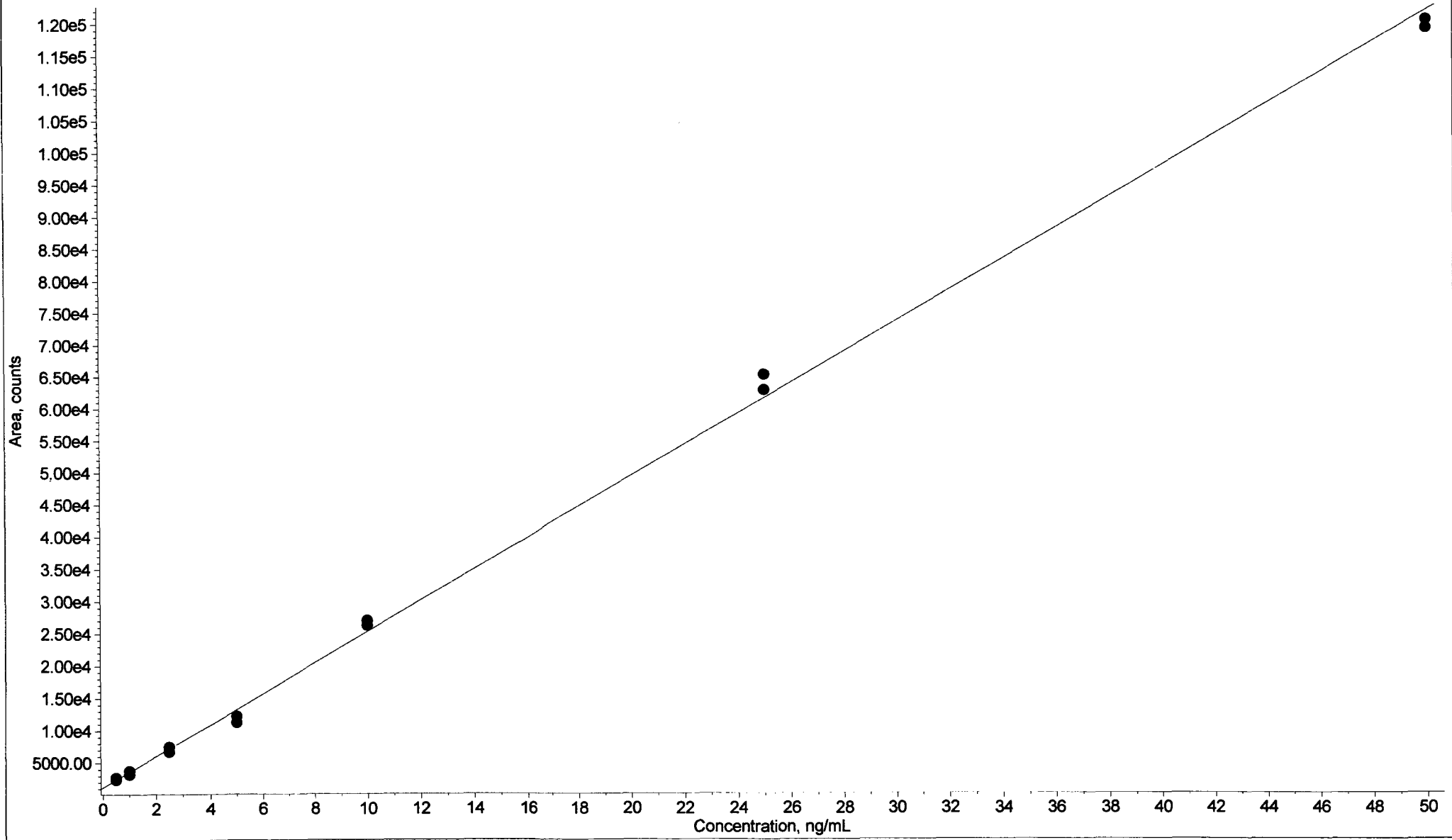
BRK 07/20/05

	A	B	C	D
1	Vegetation Conversion (FORMULAS)			
2				
3	Compound:	PFOA Confirm. Ion	Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049881	DF06-V02-RCP001-0-041007	=Raw Data!H178	=(C9*2)/5
10	C0049881 Rep	DF06-V02-RCP001-0-041007	=Raw Data!H179	=(C10*2)/5
11	C0049881 Spk C	DF06-V02-RCP001-0-041007	=Raw Data!H174	=(C11*2)/5
12				
13	C0049883	DF09-V01-PAP001-0-041007	=Raw Data!H194	=(C13*2)/5
14	C0049883 Rep	DF09-V01-PAP001-0-041007	=Raw Data!H195	=(C14*2)/5
15	C0049883 Spk G	DF09-V01-PAP001-0-041007	=Raw Data!H190	=(C15*2)/5
16				
17	C0049884	DF09-V01-RCP001-0-041007	=Raw Data!H202	=(C17*2)/5
18	C0049884 Rep	DF09-V01-RCP001-0-041007	=Raw Data!H203	=(C18*2)/5
19	C0049884 Spk I	DF09-V01-RCP001-0-041007	=Raw Data!H198	=(C19*2)/5
20				
21	C0049885	DF09-V02-RCP001-0-041007	=Raw Data!H210	=(C21*2)/5
22	C0049885 Rep	DF09-V02-RCP001-0-041007	=Raw Data!H211	=(C22*2)/5
23	C0049885 Spk K	DF09-V02-RCP001-0-041007	=Raw Data!H206	=(C23*2)/5
24	C0049885 Spk L	DF09-V02-RCP001-0-041007	=Raw Data!H207	=(C24*2)/5
25				

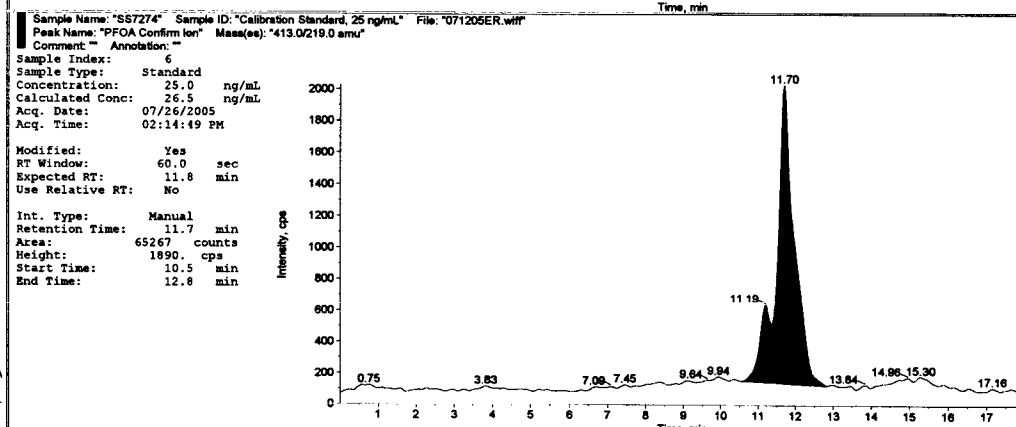
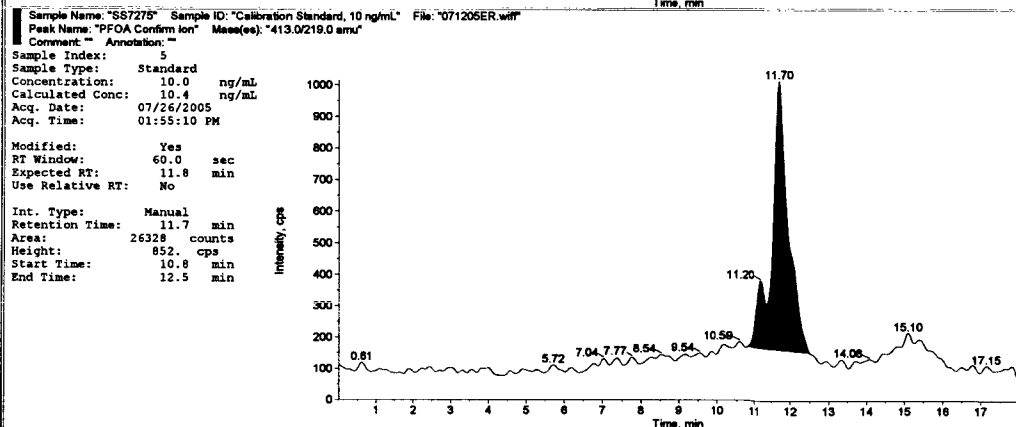
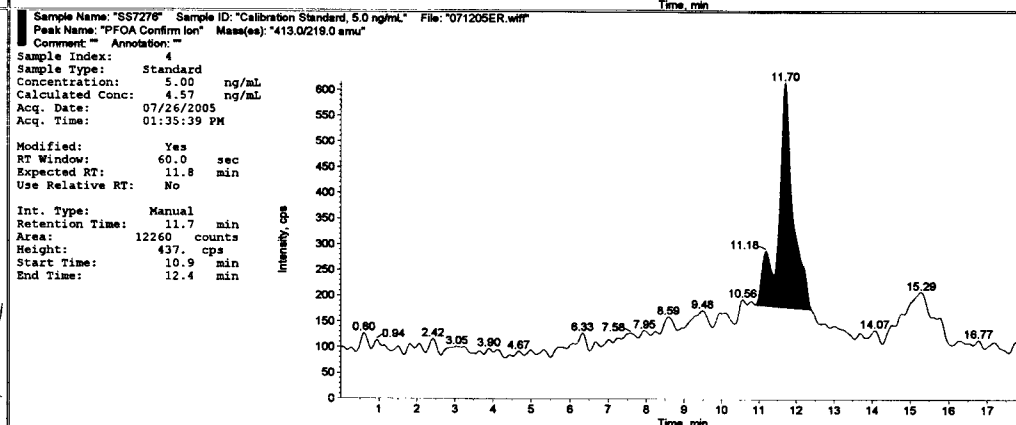
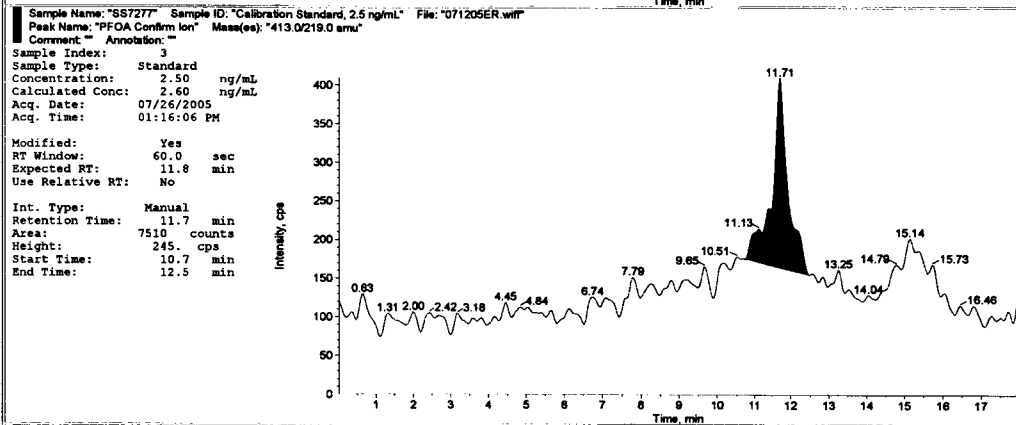
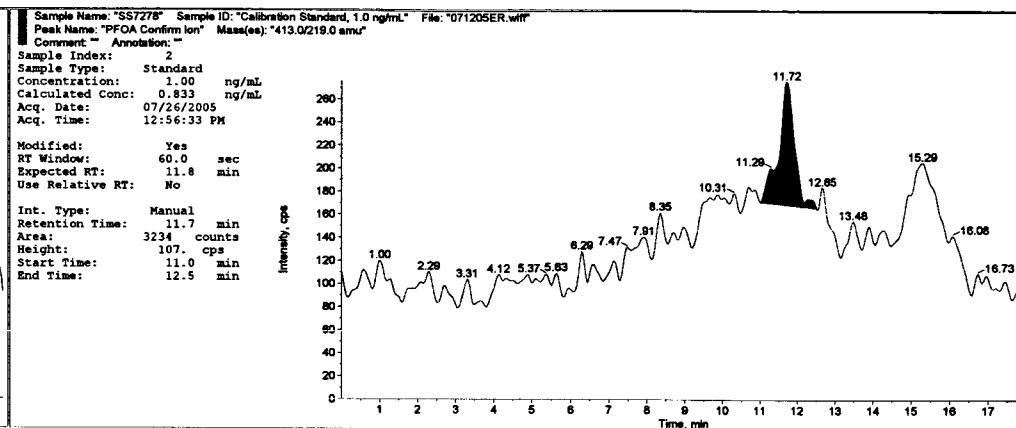
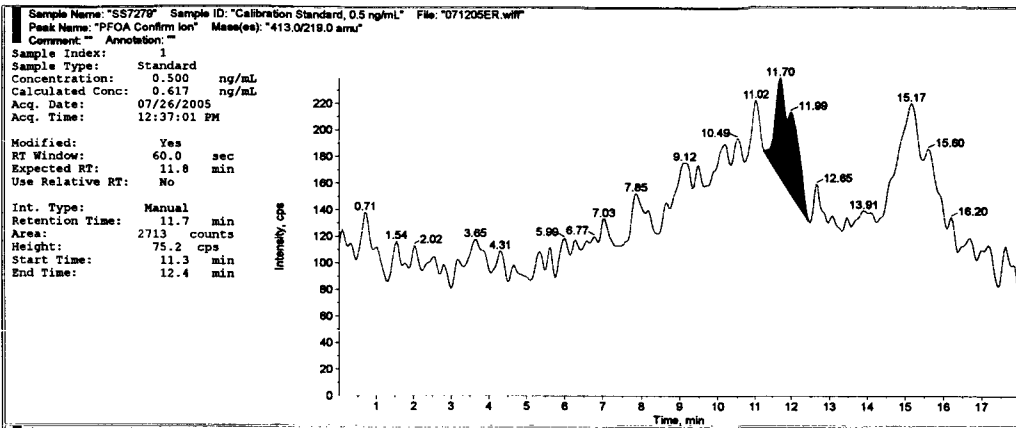
0384
BAC 07/28/15

AMB 07/28/05

■ Untitled 1 (PFOA Confirm Ion): "Linear" Regression ("1 / x" weighting): $y = 2.42e+003 x + 1.22e+003$ ($r = 0.9986$)

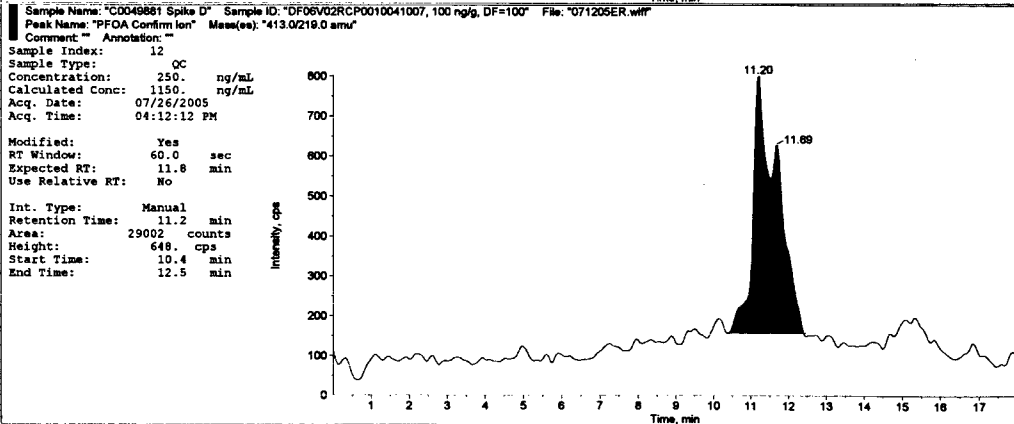
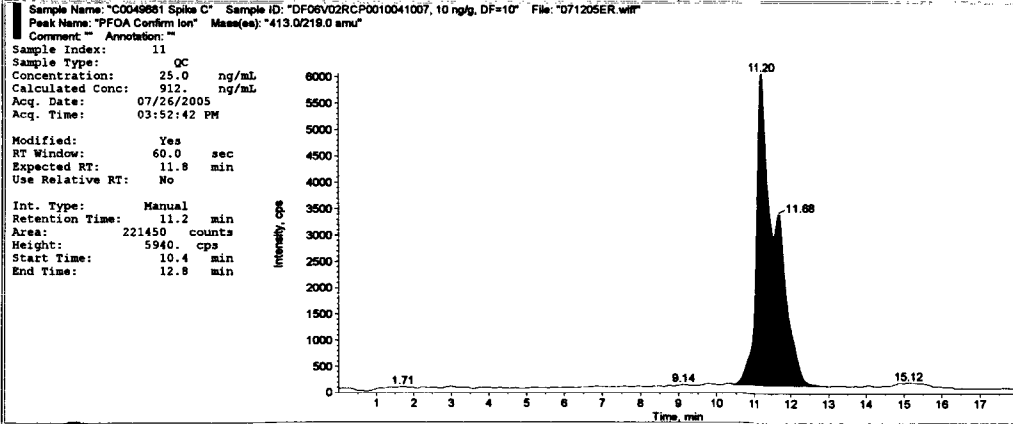
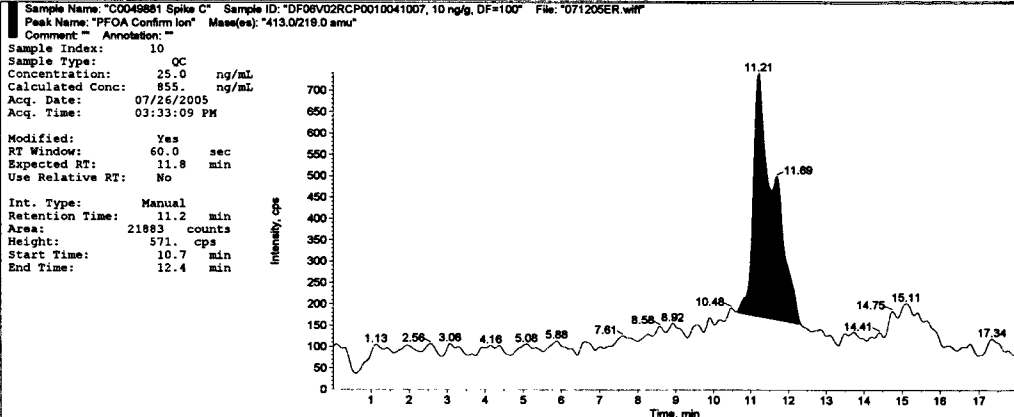
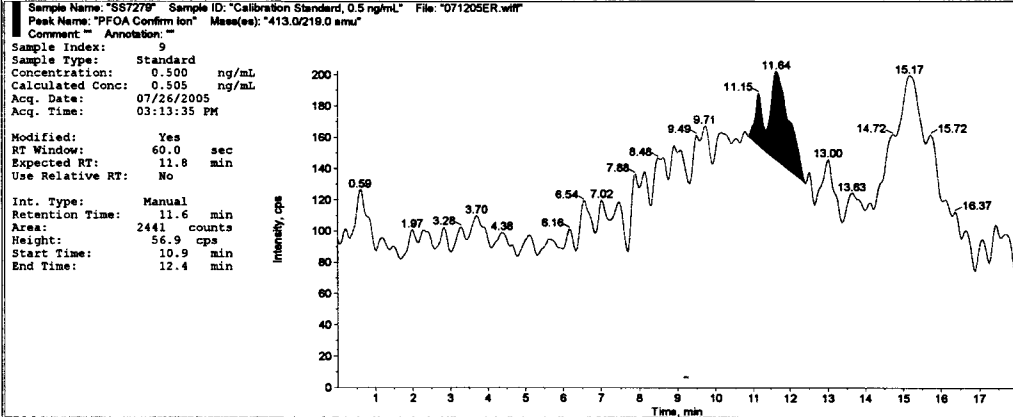
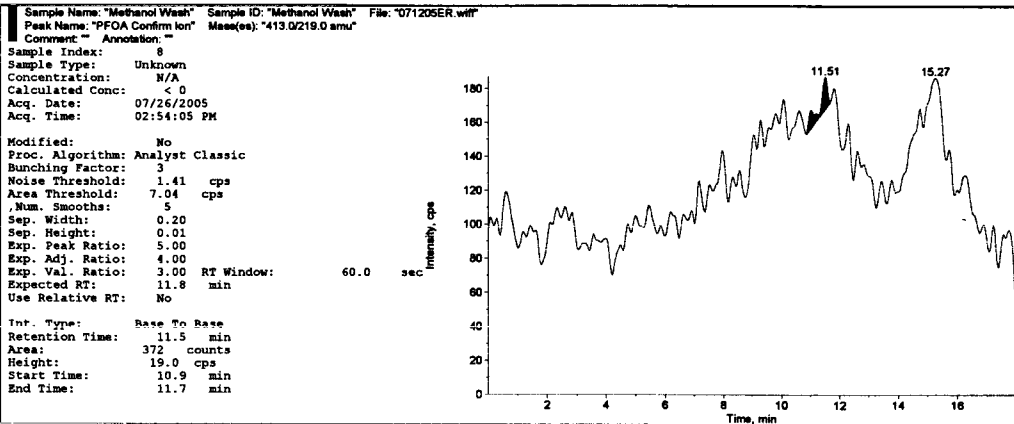
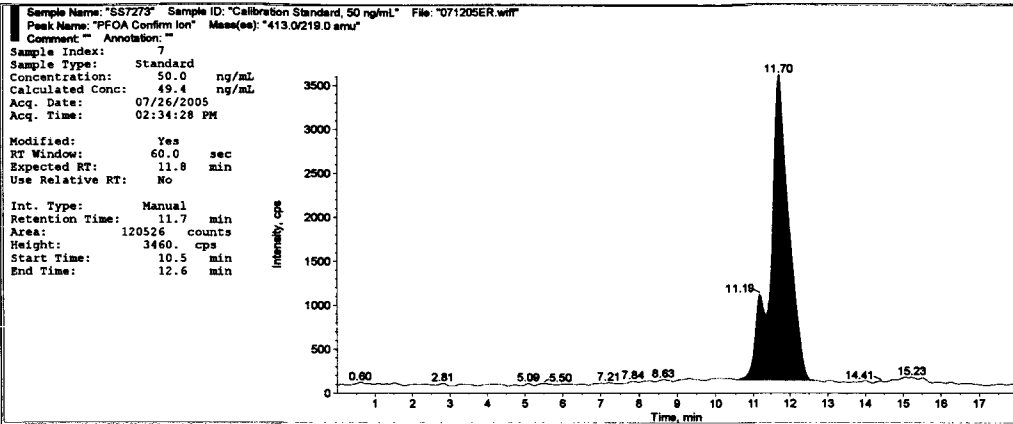


0385

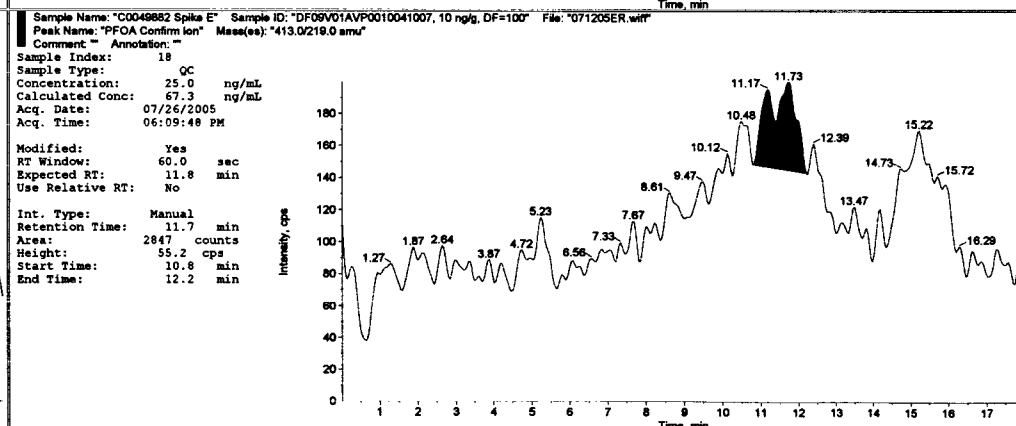
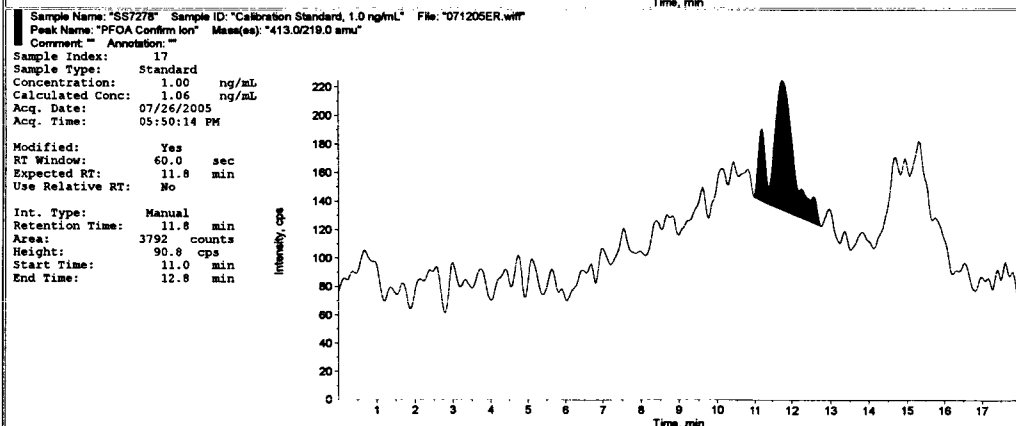
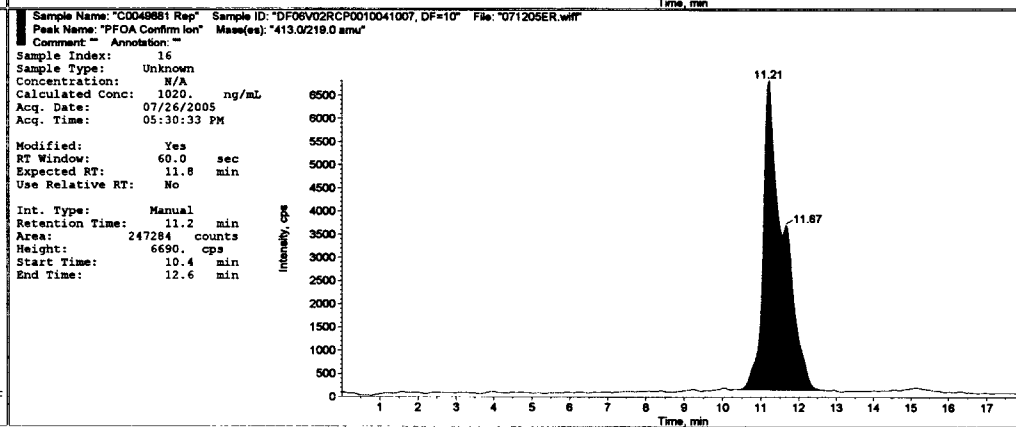
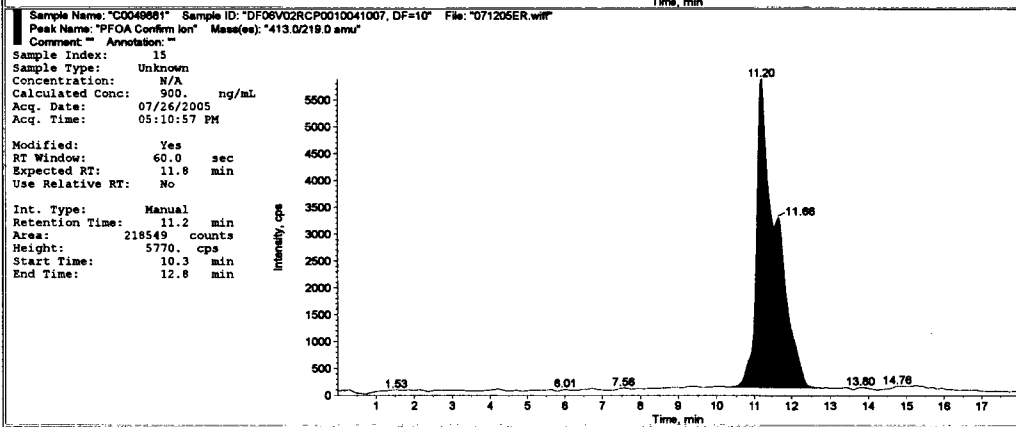
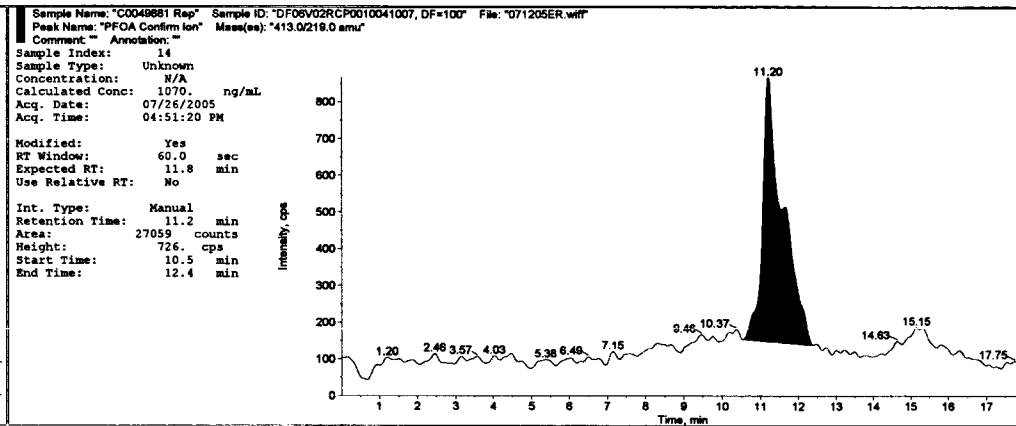
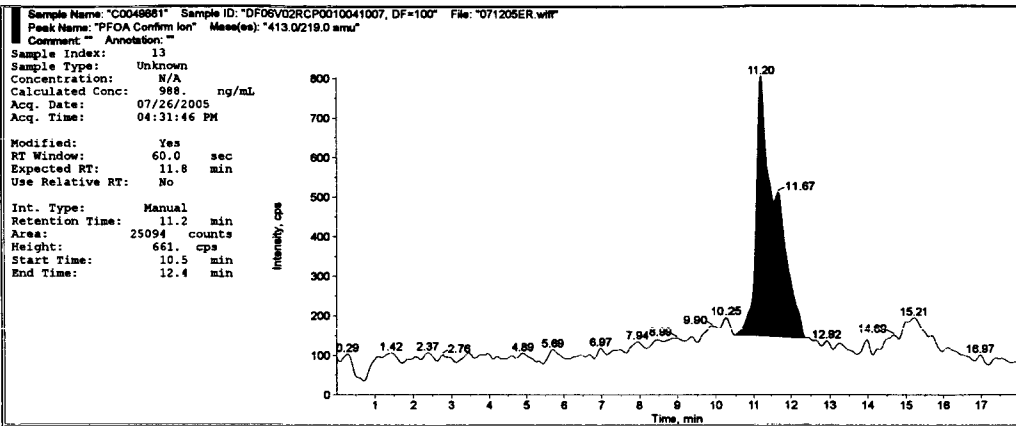


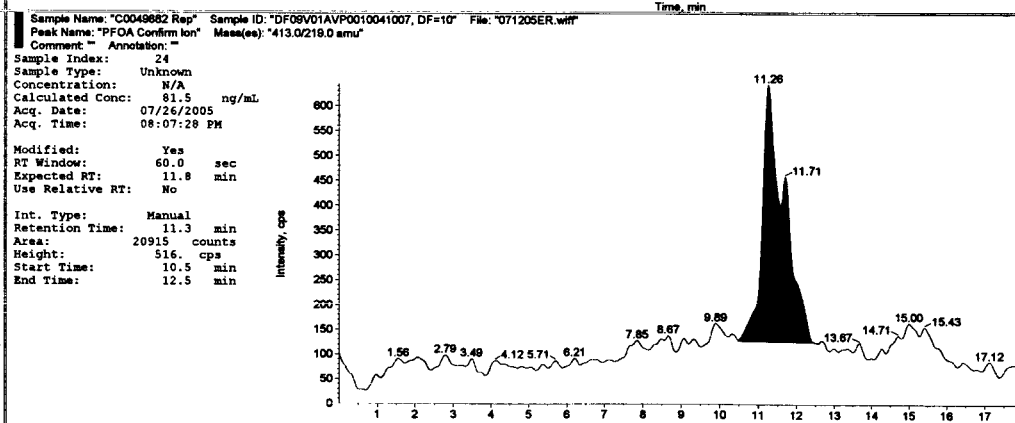
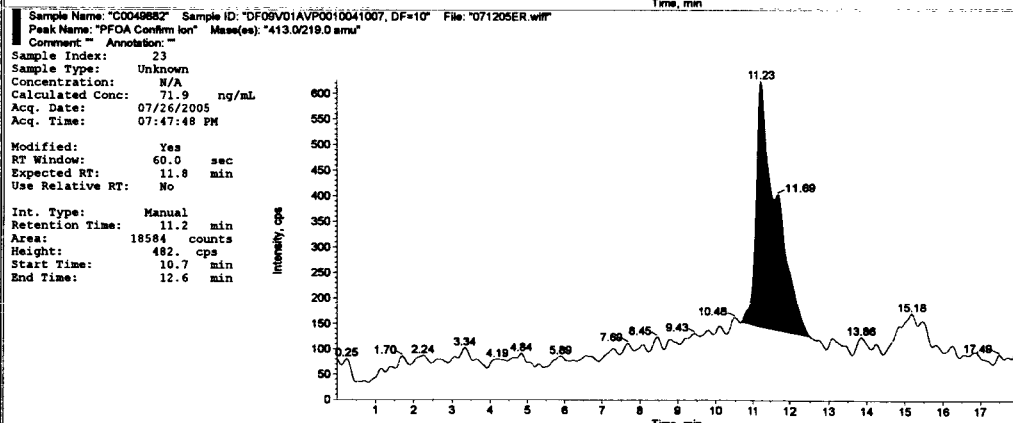
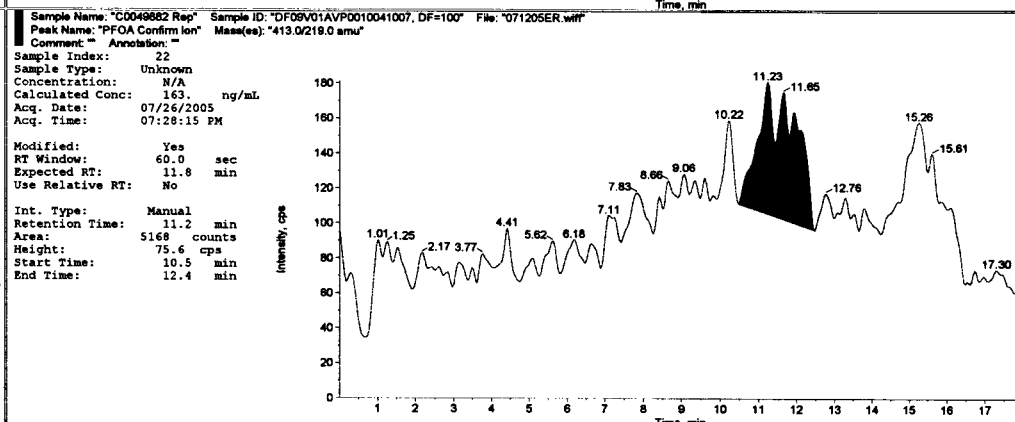
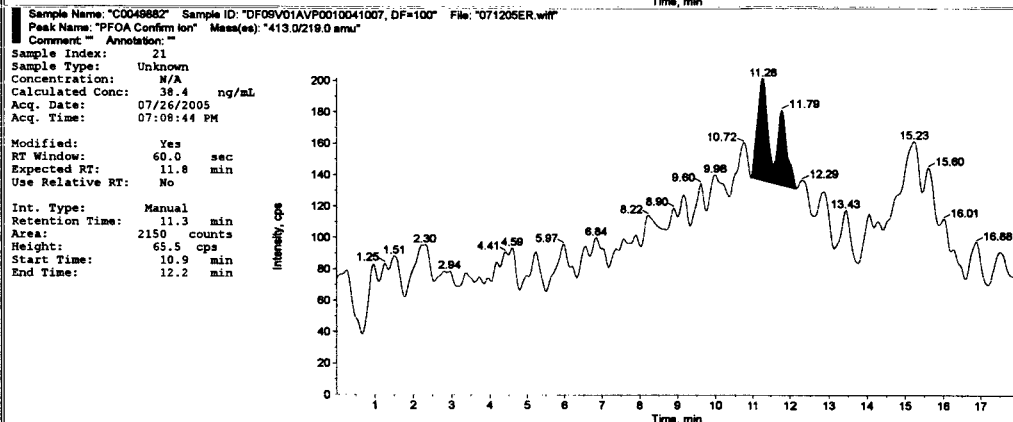
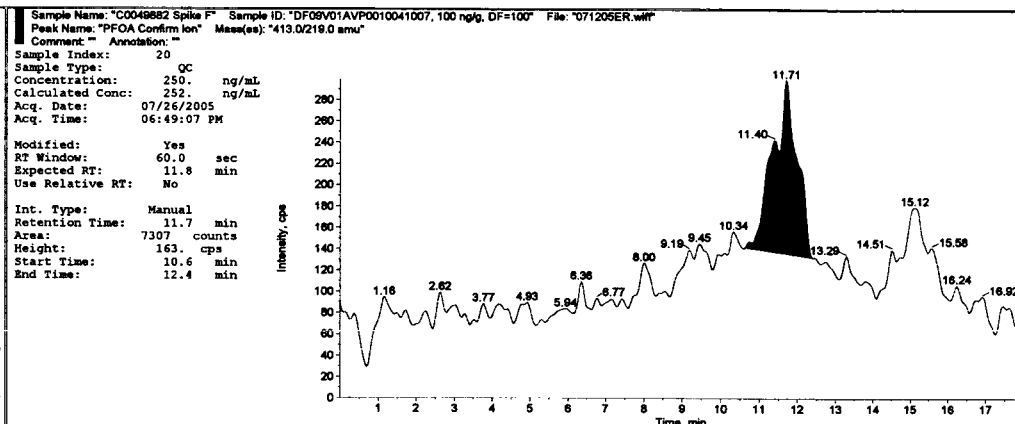
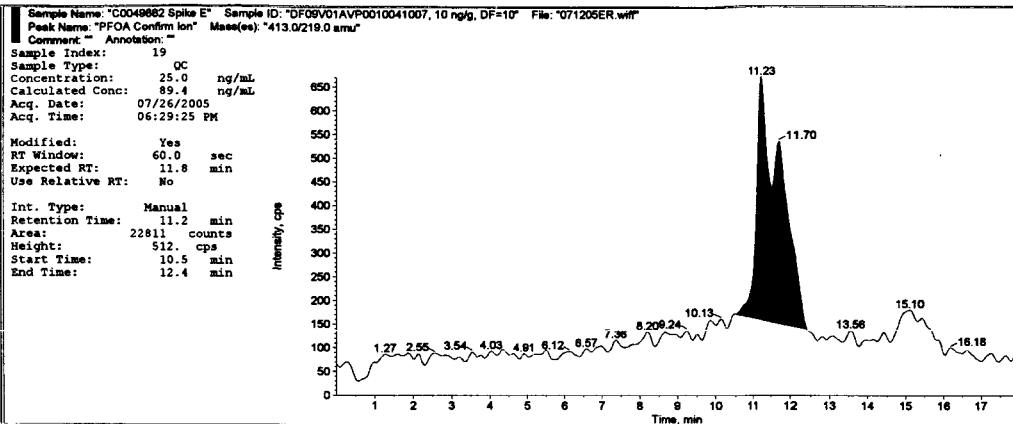
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 Date 07/28/05
 Run# 1 To 50
 Sample Index 1 To 50
AMB 07/28/05

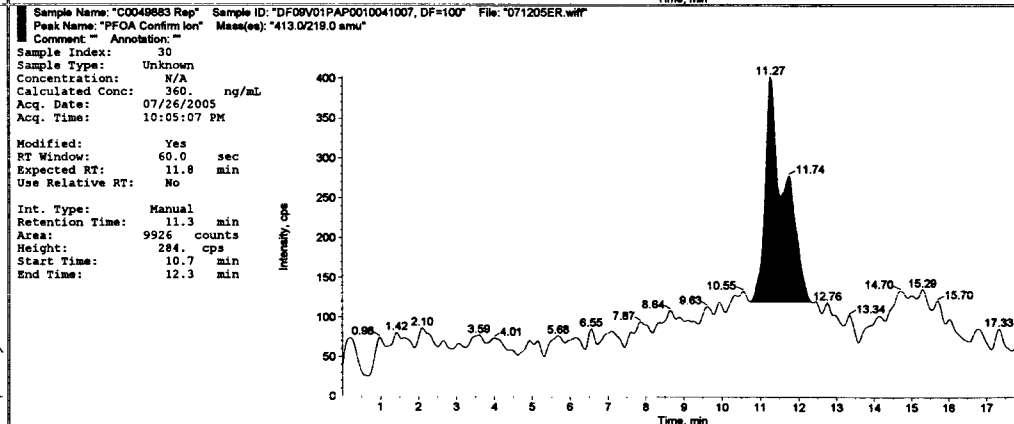
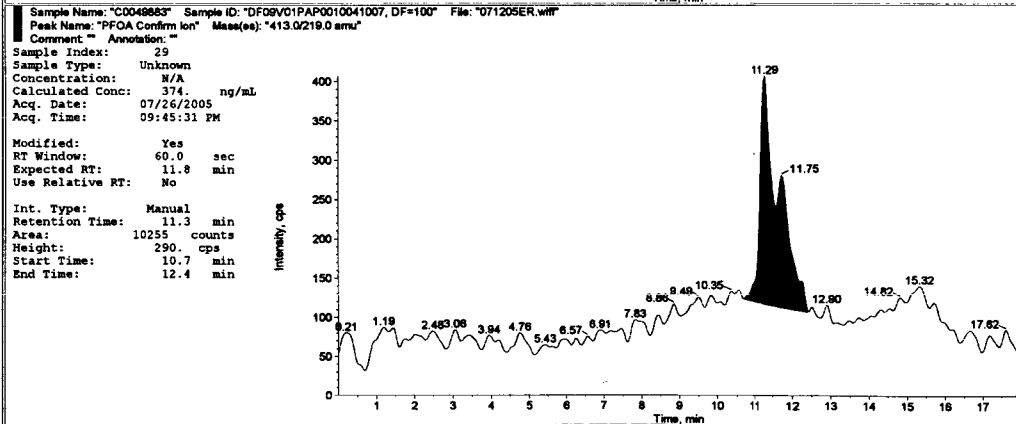
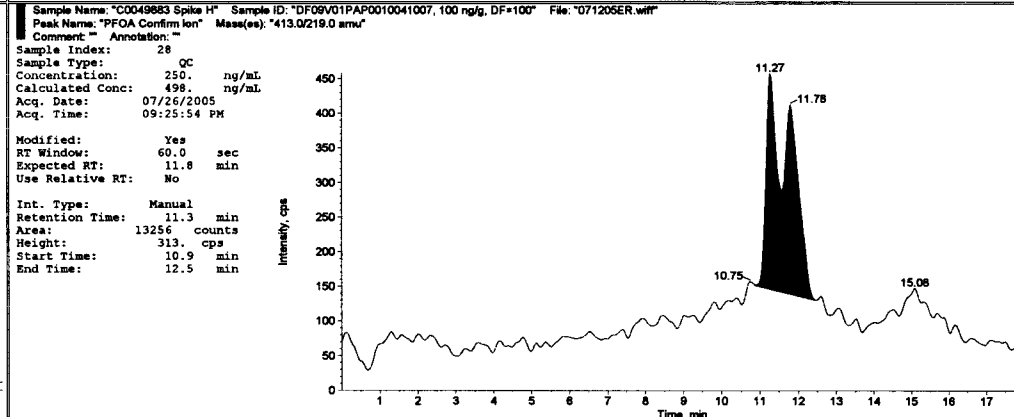
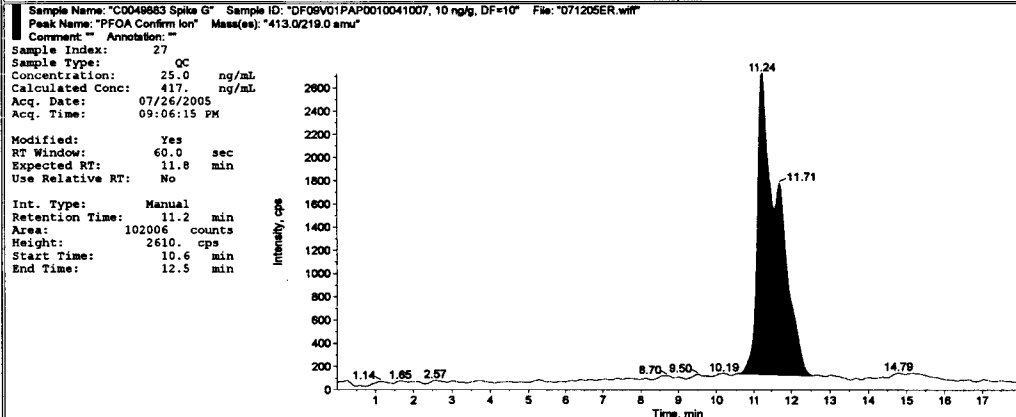
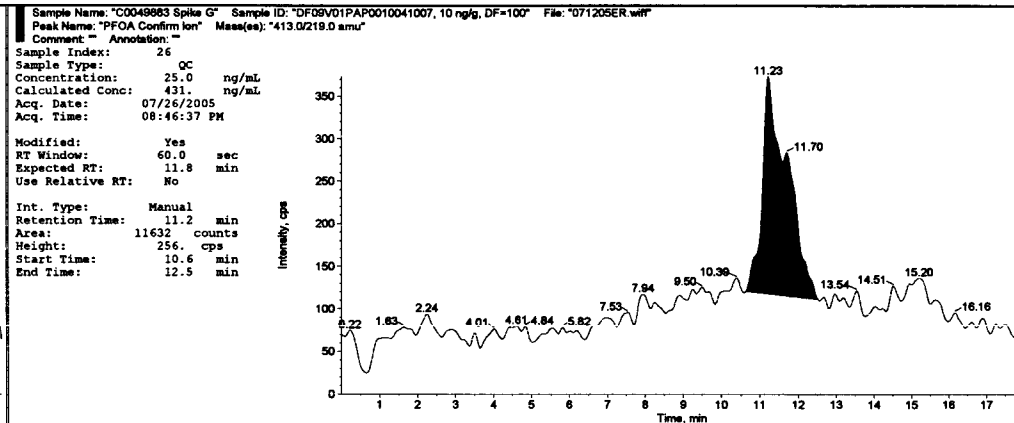
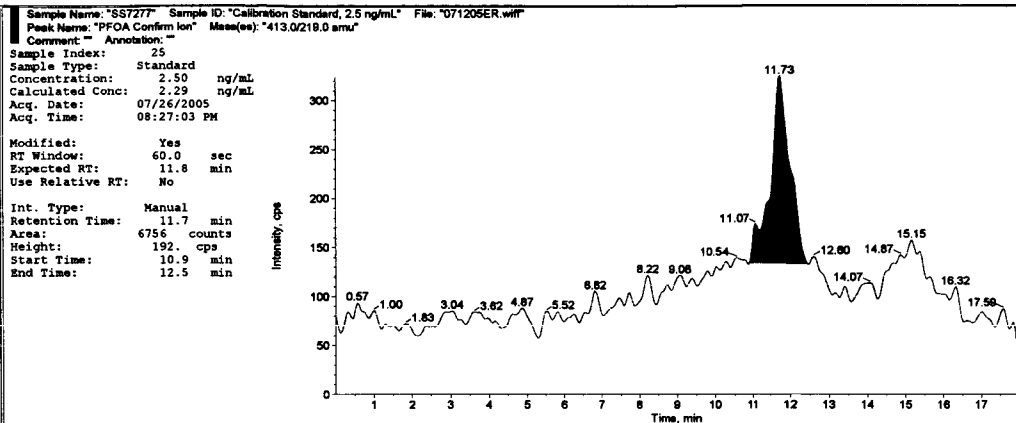
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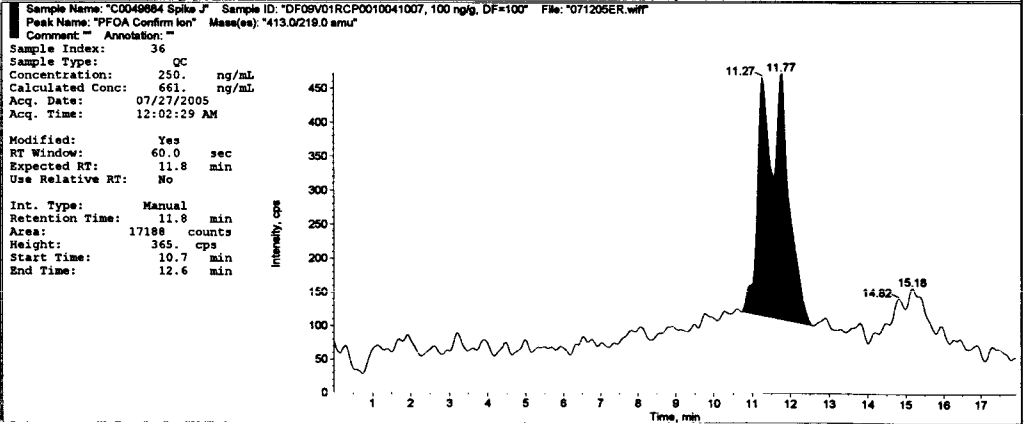
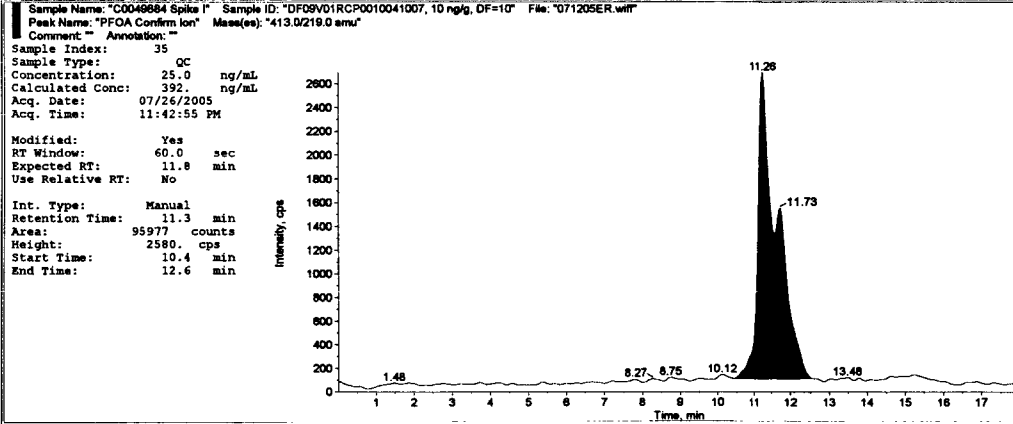
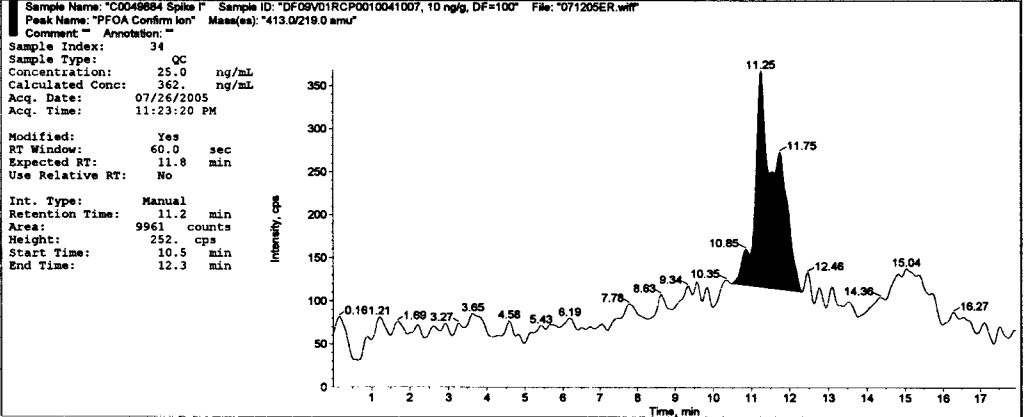
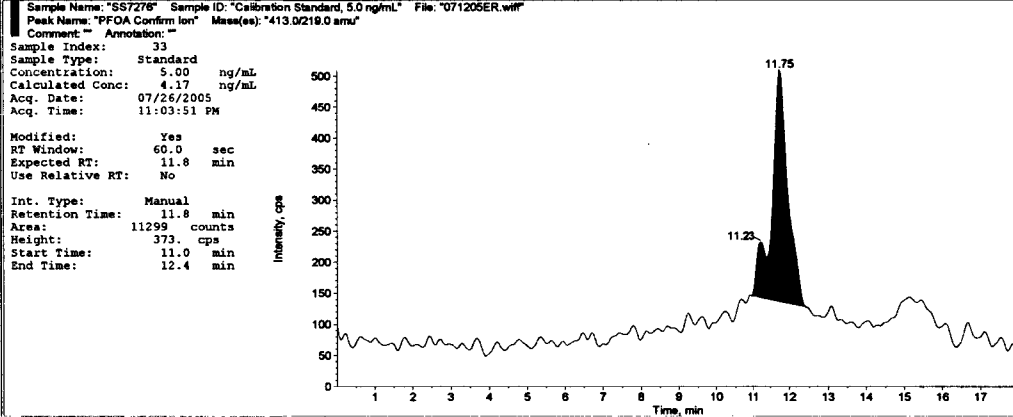
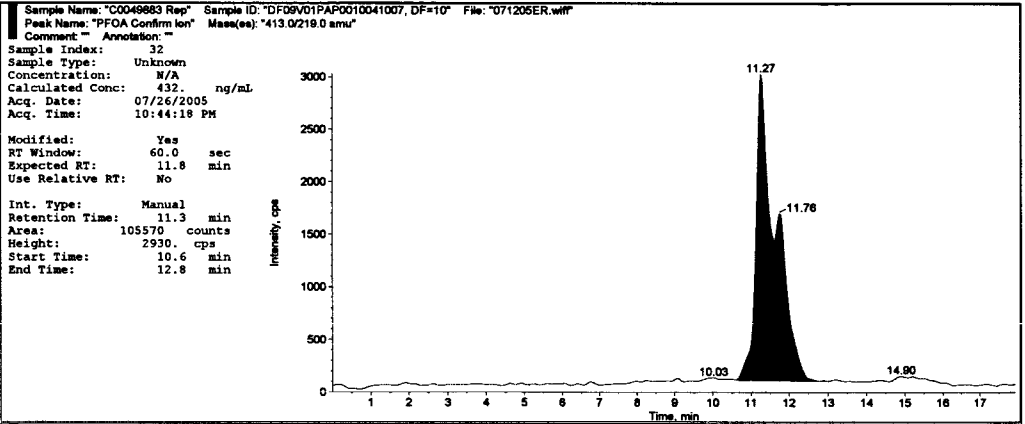
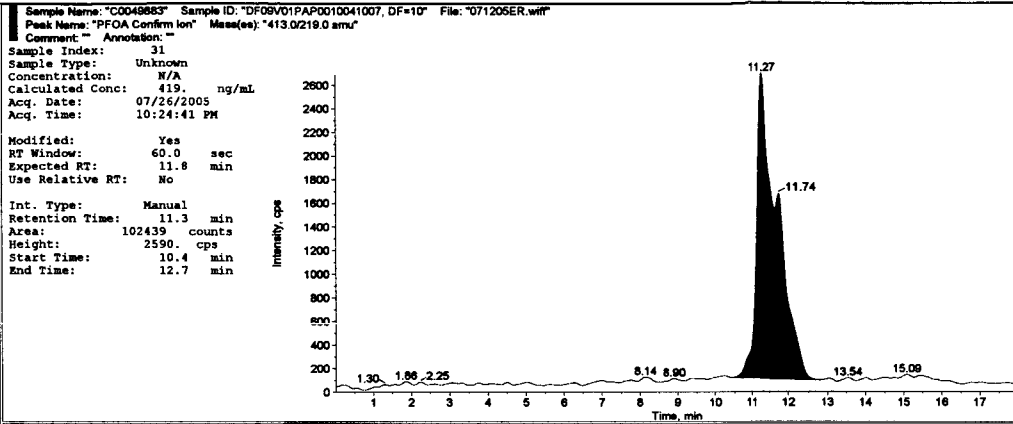
0387



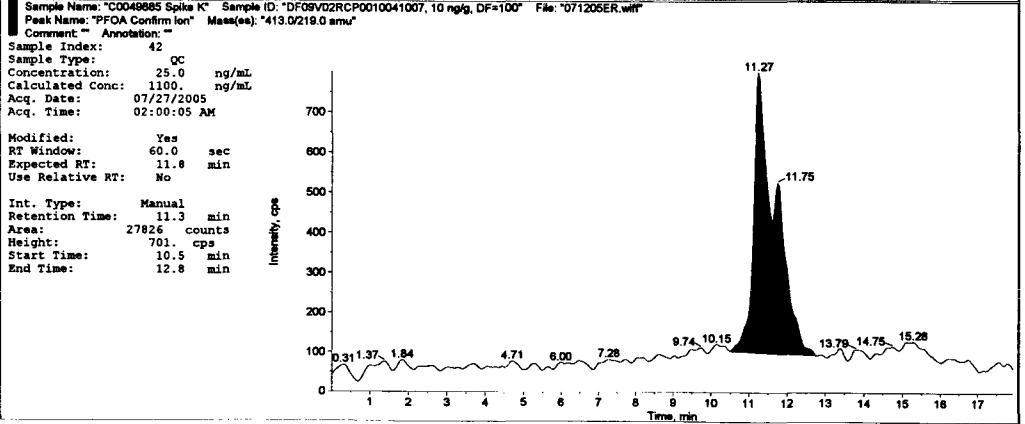
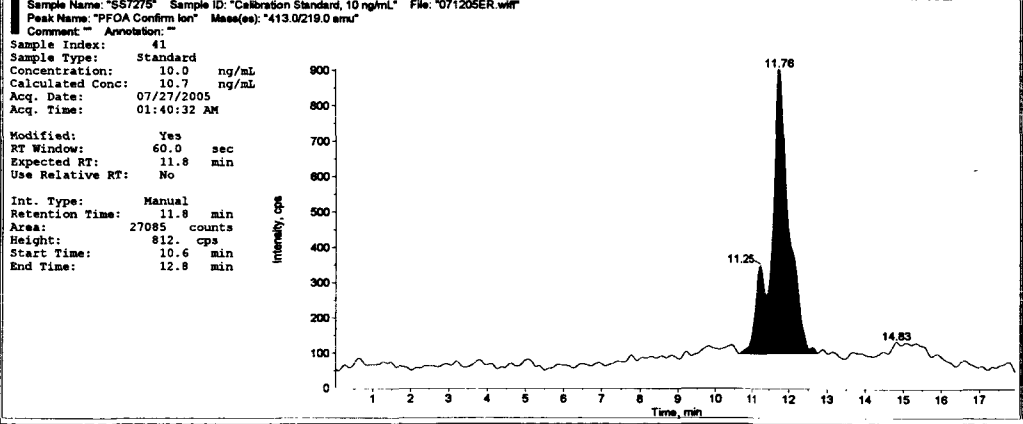
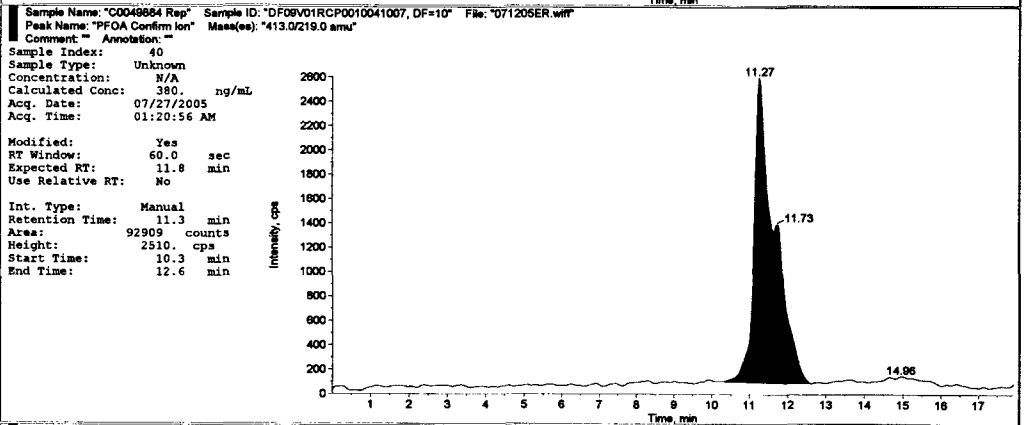
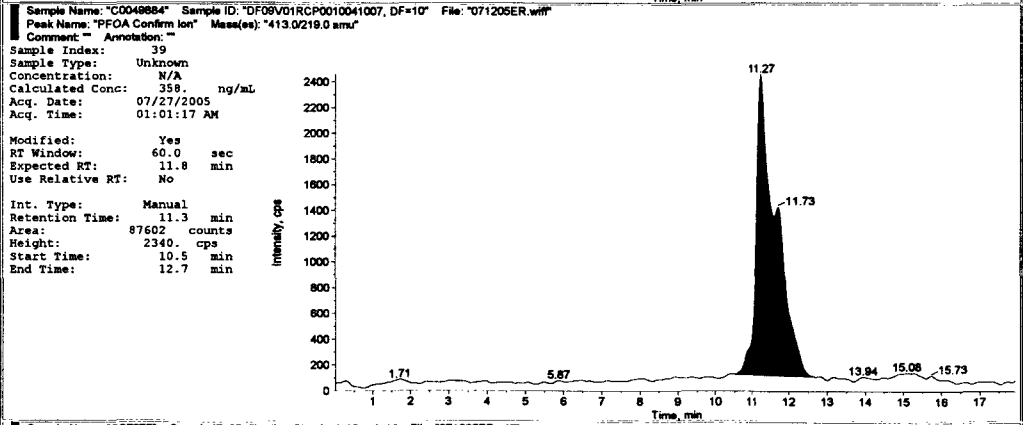
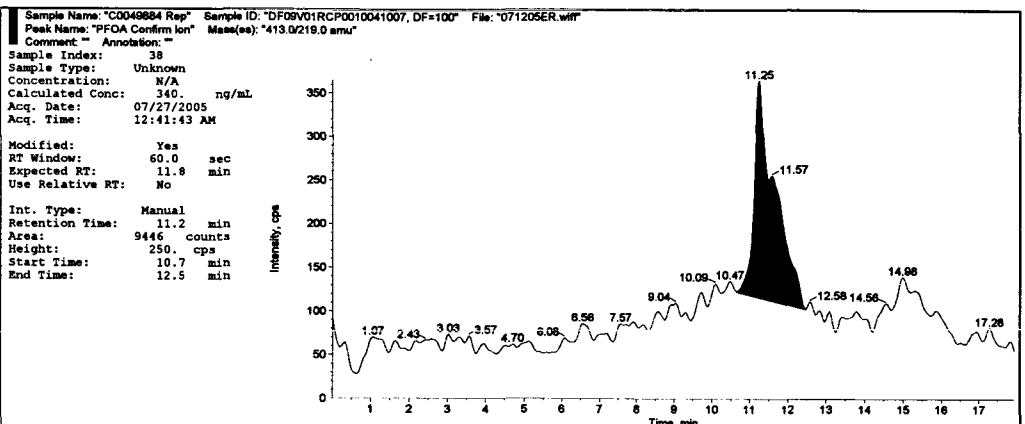
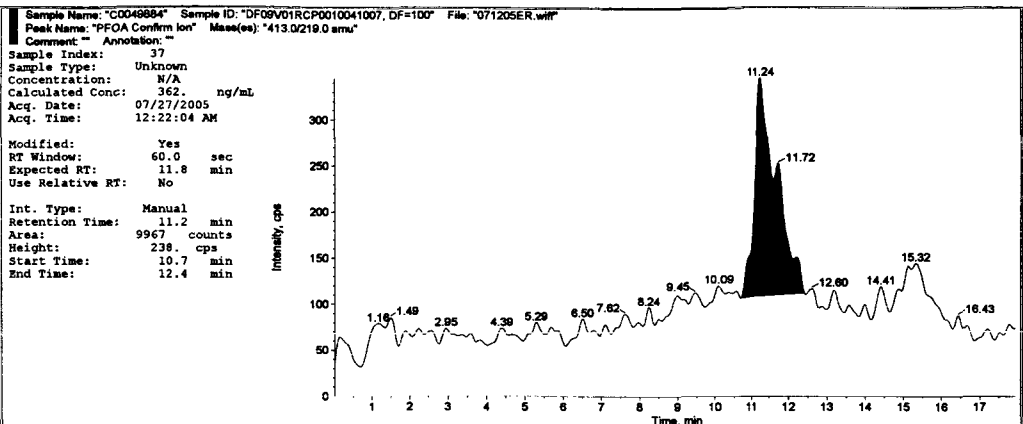




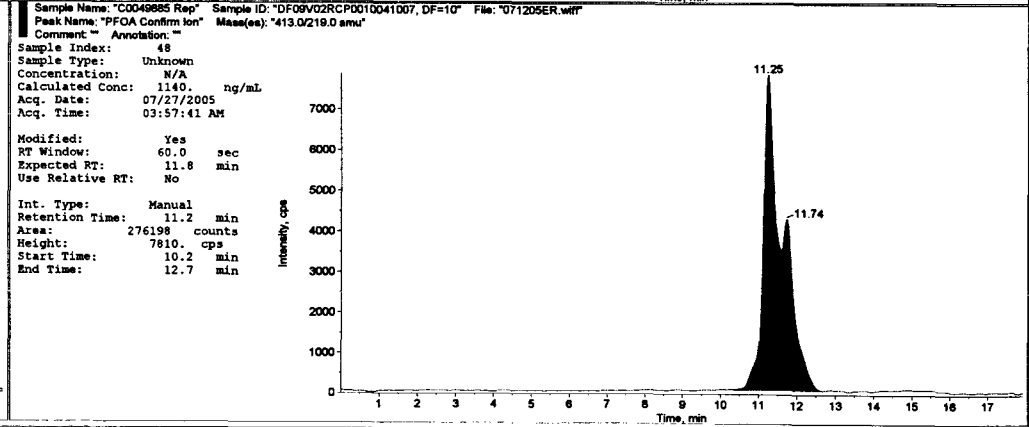
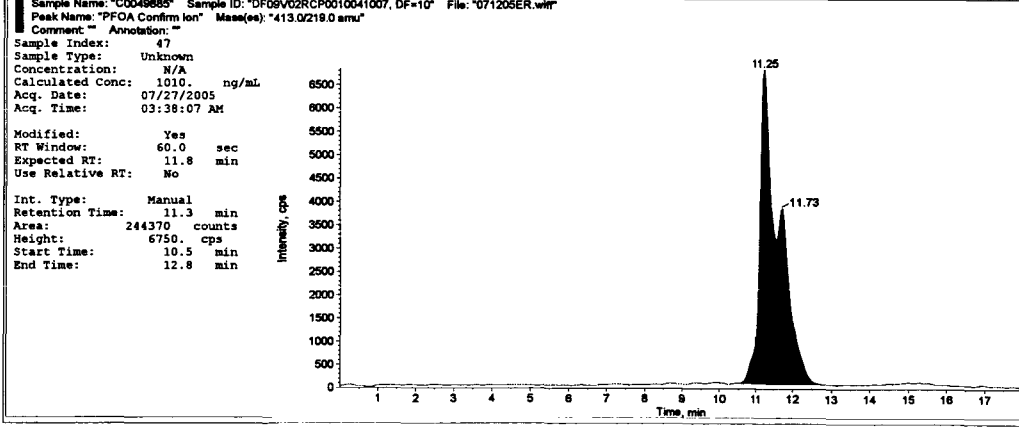
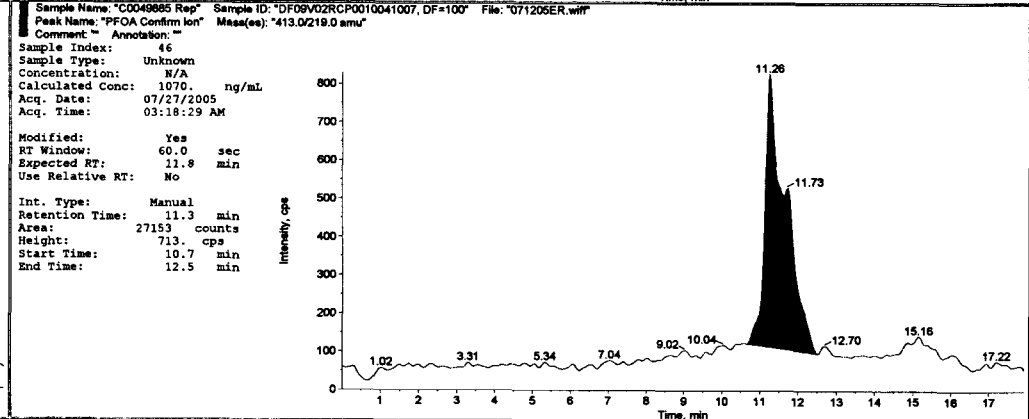
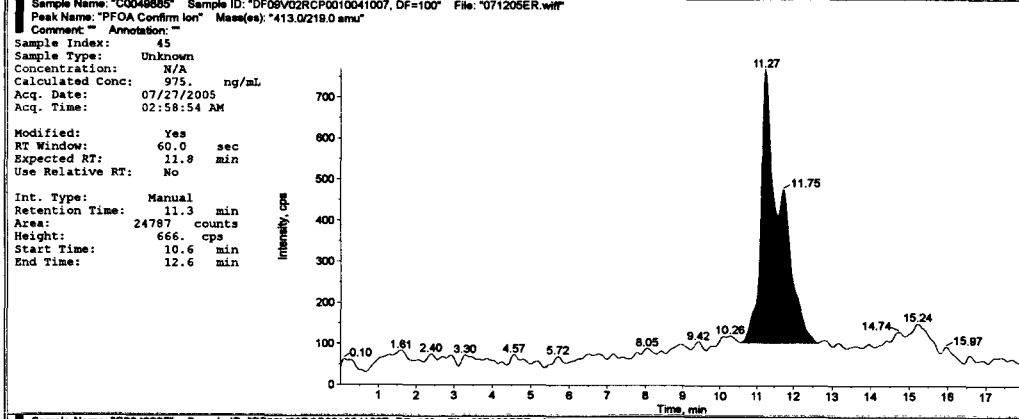
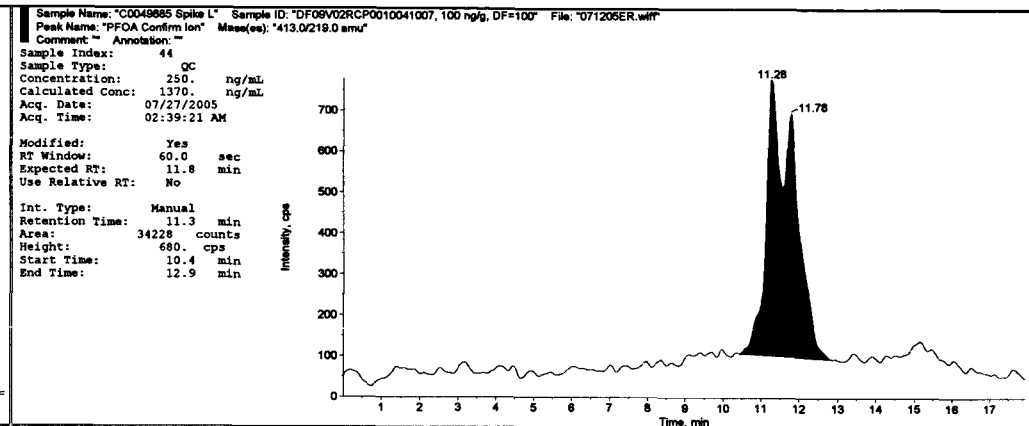
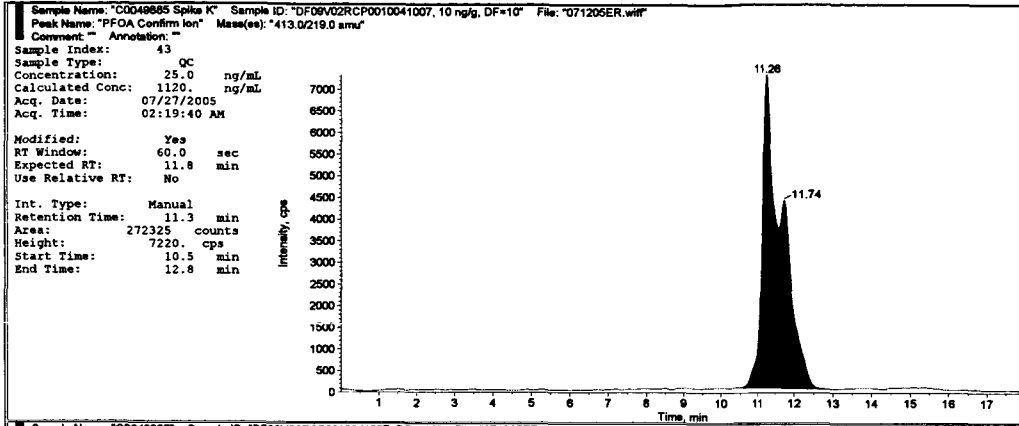
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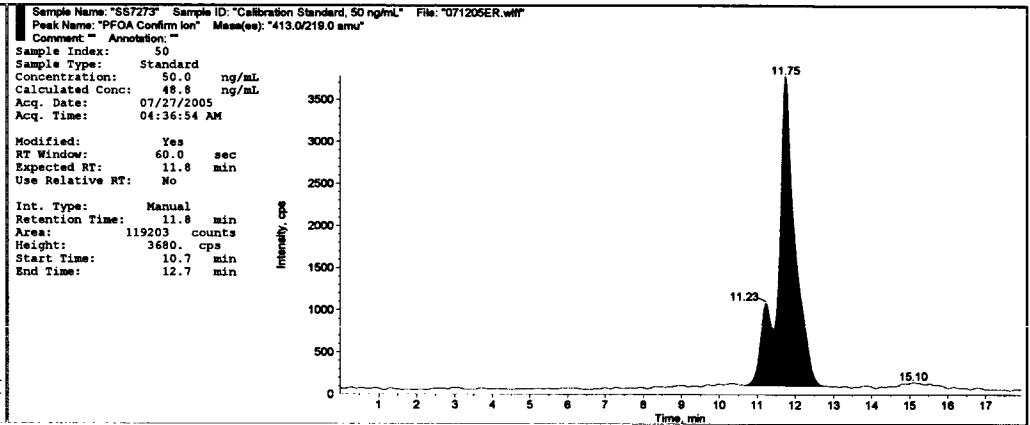
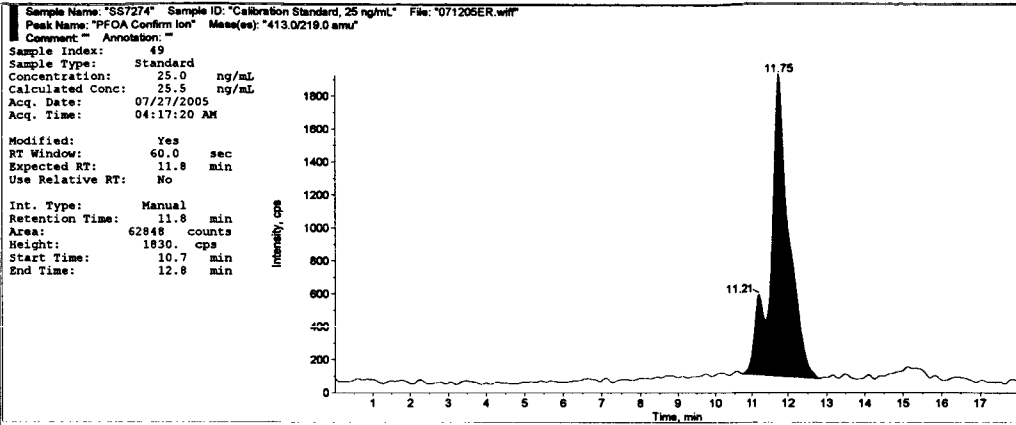


0391



0392





Raw Data

A	B	C	D	E	F	G	H	I	J
PFHS									
Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Peak Area (cc)	Concentration (ng/mL)	Inoculated Concentration (ng/mL)	Run #	
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFHS	14037	0.5	4.55E-01	71205ER-201	
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFHS	28214	1	9.75E-01	71205ER-202	
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFHS	69629	2.5	2.49E+00	71205ER-203	
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFHS	140033	5	5.08E+00	71205ER-204	
SS7275	Calibration Standard, 10 ng/mL	Standard	1.0	PFHS	276951	10	1.01E+01	71205ER-205	
SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PFHS	666894	25	2.44E+01	71205ER-206	
SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PFHS	1301736	50	4.77E+01	71205ER-207	
Methanol Wash	Methanol Wash	Unknown	1.0	PFHS	326	N/A	< 0	71205ER-208	
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFHS	13525	0.5	4.36E-01	71205ER-209	
C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFHS	11705	25	3.69E+01	71205ER-210	
C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PFHS	114567	25	4.14E+01	71205ER-211	
C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	100.0	PFHS	65317	250	2.34E+02	71205ER-212	
C0049881	DF06V02RCP0010041007, DF=100	Unknown	100.0	PFHS	6454	N/A	1.77E+01	71205ER-213	
C0049881 Rep	DF06V02RCP0010041007, DF=100	Unknown	100.0	PFHS	6824	N/A	1.90E+01	71205ER-214	
C0049881	DF06V02RCP0010041007, DF=10	Unknown	10.0	PFHS	58007	N/A	2.07E+01	71205ER-215	
C0049881 Rep	DF06V02RCP0010041007, DF=10	Unknown	10.0	PFHS	64852	N/A	2.32E+01	71205ER-216	
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFHS	29372	1	1.02E+00	71205ER-217	
C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFHS	19361	25	6.50E+01	71205ER-218	
C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PFHS	194708	25	7.08E+01	71205ER-219	
C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	100.0	PFHS	60312	250	2.15E+02	71205ER-220	
C0049882	DF09V01AVP0010041007, DF=100	Unknown	100.0	PFHS	13341	N/A	4.29E+01	71205ER-221	
C0049882 Rep	DF09V01AVP0010041007, DF=100	Unknown	100.0	PFHS	14726	N/A	4.80E+01	71205ER-222	
C0049882	DF09V01AVP0010041007, DF=10	Unknown	10.0	PFHS	142687	N/A	5.18E+01	71205ER-223	
C0049882 Rep	DF09V01AVP0010041007, DF=10	Unknown	10.0	PFHS	143865	N/A	5.22E+01	71205ER-224	
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFHS	75340	2.5	2.70E+00	71205ER-225	
C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFHS	15671	25	5.15E+01	71205ER-226	
C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PFHS	159529	25	5.79E+01	71205ER-227	
C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	100.0	PFHS	70329	250	2.52E+02	71205ER-228	
C0049883	DF09V01PAP0010041007, DF=100	Unknown	100.0	PFHS	9028	N/A	2.71E+01	71205ER-229	
C0049883 Rep	DF09V01PAP0010041007, DF=100	Unknown	100.0	PFHS	10028	N/A	3.08E+01	71205ER-230	
C0049883	DF09V01PAP0010041007, DF=10	Unknown	10.0	PFHS	94146	N/A	3.39E+01	71205ER-231	
C0049883 Rep	DF09V01PAP0010041007, DF=10	Unknown	10.0	PFHS	100205	N/A	3.62E+01	71205ER-232	
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFHS	147936	5	5.37E+00	71205ER-233	
C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFHS	12987	25	4.16E+01	71205ER-234	
C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PFHS	127987	25	4.64E+01	71205ER-235	
C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	100.0	PFHS	81443	250	2.93E+02	71205ER-236	
C0049884	DF09V01RCP0010041007, DF=100	Unknown	100.0	PFHS	7022	N/A	1.97E+01	71205ER-237	
C0049884 Rep	DF09V01RCP0010041007, DF=100	Unknown	100.0	PFHS	7333	N/A	2.09E+01	71205ER-238	
C0049884	DF09V01RCP0010041007, DF=10	Unknown	10.0	PFHS	70145	N/A	25.1	71205ER-239	
C0049884 Rep	DF09V01RCP0010041007, DF=10	Unknown	10	PFHS	72045	N/A	25.8	71205ER-240	
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFHS	292820	10	10.7	71205ER-241	
C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFHS	32256	25	1.12E+02	71205ER-242	
C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PFHS	329677	25	1.20E+02	71205ER-243	
C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	100.0	PFHS	90488	250	3.26E+02	71205ER-244	
C0049885	DF09V02RCP0010041007, DF=100	Unknown	100.0	PFHS	24274	N/A	8.31E+01	71205ER-245	
C0049885 Rep	DF09V02RCP0010041007, DF=100	Unknown	100.0	PFHS	28153	N/A	9.73E+01	71205ER-246	
C0049885	DF09V02RCP0010041007, DF=10	Unknown	10.0	PFHS	242023	N/A	8.82E+01	71205ER-247	
C0049885 Rep	DF09V02RCP0010041007, DF=10	Unknown	10.0	PFHS	269178	N/A	9.82E+01	71205ER-248	
SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PFHS	704456	25	2.58E+01	71205ER-249	
SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PFHS	1385196	50	5.08E+01	71205ER-250	
PPOS									
Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Peak Area (cc)	Concentration (ng/mL)	Inoculated Concentration (ng/mL)	Run #	
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PPOS	9914	0.5	4.78E-01	71205ER-201	
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PPOS	17494	1	9.54E-01	71205ER-202	
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PPOS	41912	2.5	2.49E+00	71205ER-203	
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PPOS	83369	5	5.09E+00	71205ER-204	
SS7275	Calibration Standard, 10 ng/mL	Standard	1.0	PPOS	166035	10	1.03E+01	71205ER-205	
SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PPOS	392283	25	2.45E+01	71205ER-206	
SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PPOS	772162	50	4.83E+01	71205ER-207	
Methanol Wash	Methanol Wash	Unknown	1.0	PPOS	1114	N/A	< 0	71205ER-208	
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PPOS	9096	0.5	4.27E-01	71205ER-209	
C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PPOS	85860	25	5.24E+02	71205ER-210	
C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PPOS	888493	25	5.56E+02	71205ER-211	
C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	100.0	PPOS	122194	250	7.52E+02	71205ER-212	
C0049881	DF06V02RCP0010041007, DF=100	Unknown	100.0	PPOS	91689	N/A	5.61E+02	71205ER-213	
C0049881 Rep	DF06V02RCP0010041007, DF=100	Unknown	100.0	PPOS	102763	N/A	6.30E+02	71205ER-214	
C0049881	DF06V02RCP0010041007, DF=10	Unknown	10.0	PPOS	828699	N/A	5.19E+02	71205ER-215	
C0049881 Rep	DF06V02RCP0010041007, DF=10	Unknown	10.0	PPOS	982956	N/A	6.15E+02	71205ER-216	
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PPOS	18462	1	1.01E+00	71205ER-217	
C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PPOS	83233	25	5.08E+02	71205ER-218	
C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PPOS	809218	25	5.06E+02	71205ER-219	
C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	100.0	PPOS	102065	250	6.26E+02	71205ER-220	
C0049882	DF09V01AVP0010041007, DF=100	Unknown	100.0	PPOS	78607	N/A	4.79E+02	71205ER-221	
C0049882 Rep	DF09V01AVP0010041007, DF=100	Unknown	100.0	PPOS	74573	N/A	4.54E+02	71205ER-222	
C0049882	DF09V01AVP0010041007, DF=10	Unknown	10.0	PPOS	790057	N/A	4.94E+02	71205ER-223	
C0049882 Rep	DF09V01AVP0010041007, DF=10	Unknown	10.0	PPOS	747136	N/A	4.67E+02	71205ER-224	
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PPOS	43078	2.5	2.56E+00	71205ER-225	
C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PPOS	148880	25	9.20E+02	71205ER-226	
C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10.0	PPOS	1455868	25	9.12E+02	71205ER-227	
C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=10	Quality Control	100.0	PPOS	186822	250	1.16E+03	71205ER-228	

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	A	B	C	D	E	F	G	H	I	J
84	C0049883	DF09V01PAP0010041007, DF=100	Unknown	100.0	PFOS	152047	N/A	9.40E+02	71205ER-229	
85	C0049883 Rep	DF09V01PAP0010041007, DF=100	Unknown	100.0	PFOS	149257	N/A	9.22E+02	71205ER-230	
86	C0049883	DF09V01PAP0010041007, DF=10	Unknown	10.0	PFOS	1468074	N/A	9.20E+02	71205ER-231	
87	C0049883 Rep	DF09V01PAP0010041007, DF=10	Unknown	10	PFOS	1513664	N/A	948	71205ER-232	
88	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOS	90840	5	5.56	71205ER-233	
89	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOS	42220	25	251	71205ER-234	
90	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOS	430353	25	269	71205ER-235	
91	C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	100	PFOS	85752	250	524	71205ER-236	
92	C0049884	DF09V01RCP0010041007, DF=100	Unknown	100	PFOS	38624	N/A	228	71205ER-237	
93	C0049884 Rep	DF09V01RCP0010041007, DF=100	Unknown	100	PFOS	40271	N/A	238	71205ER-238	
94	C0049884	DF09V01RCP0010041007, DF=10	Unknown	10	PFOS	398692	N/A	249	71205ER-239	
95	C0049884 Rep	DF09V01RCP0010041007, DF=10	Unknown	10	PFOS	401822	N/A	251	71205ER-240	
96	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOS	172848	10	10.7	71205ER-241	
97	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOS	620731	25	3880	71205ER-242	
98	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOS	5714739	25	3580	71205ER-243	
99	C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	100	PFOS	659318	250	4120	71205ER-244	
100	C0049885	DF09V02RCP0010041007, DF=100	Unknown	100	PFOS	569525	N/A	3560	71205ER-245	
101	C0049885 Rep	DF09V02RCP0010041007, DF=100	Unknown	100	PFOS	641284	N/A	4010	71205ER-246	
102	C0049885	DF09V02RCP0010041007, DF=10	Unknown	10	PFOS	5163293	N/A	3240	71205ER-247	
103	C0049885 Rep	DF09V02RCP0010041007, DF=10	Unknown	10	PFOS	5862055	N/A	3680	71205ER-248	
104	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOS	416543	25	26	71205ER-249	
105	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOS	793983	50	49.7	71205ER-250	

PFOA

109	Sample Name	Sample ID	Sample Type	Injection Factor	Analyte Peak Name	Peak Area (cc)	Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
110	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFOA	125540	0.5	0.441	71205ER-201
111	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFOA	231963	1	0.985	71205ER-202
112	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFOA	559219	2.5	2.66	71205ER-203
113	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFOA	1075916	5	5.3	71205ER-204
114	SS7275	Calibration Standard, 10 ng/mL	Standard	1.0	PFOA	2152125	10	10.8	71205ER-205
115	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	4958601	25	25.2	71205ER-206
116	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	9469168	50	48.2	71205ER-207
117	Methanol Wash	Methanol Wash	Unknown	1	PFOA	0	N/A	No Peak	71205ER-208
118	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	113602	0.5	0.38	71205ER-209
119	C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	634231	25	304	71205ER-210
120	C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	6316002	25	321	71205ER-211
121	C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=10	Quality Control	100	PFOA	1030616	250	507	71205ER-212
122	C0049881	DF06V02RCP0010041007, DF=100	Unknown	100	PFOA	660379	N/A	318	71205ER-213
123	C0049881 Rep	DF06V02RCP0010041007, DF=100	Unknown	100	PFOA	738550	N/A	358	71205ER-214
124	C0049881	DF06V02RCP0010041007, DF=10	Unknown	10	PFOA	6041554	N/A	307	71205ER-215
125	C0049881 Rep	DF06V02RCP0010041007, DF=10	Unknown	10	PFOA	6807331	N/A	346	71205ER-216
126	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	240356	1	1.03	71205ER-217
127	C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	99967	25	31	71205ER-218
128	C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	921474	25	45.1	71205ER-219
129	C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=10	Quality Control	100	PFOA	380526	250	174	71205ER-220
130	C0049882	DF09V01AVP0010041007, DF=100	Unknown	100	PFOA	53096	N/A	7.05	71205ER-221
131	C0049882 Rep	DF09V01AVP0010041007, DF=100	Unknown	100	PFOA	34784	N/A	< 0	71205ER-222
132	C0049882	DF09V01AVP0010041007, DF=10	Unknown	10	PFOA	565989	N/A	26.9	71205ER-223
133	C0049882 Rep	DF09V01AVP0010041007, DF=10	Unknown	10	PFOA	556849	N/A	26.5	71205ER-224
134	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	533753	2.5	2.53	71205ER-225
135	C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	355836	25	162	71205ER-226
136	C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	3457978	25	175	71205ER-227
137	C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100	PFOA	711564	250	344	71205ER-228
138	C0049883	DF09V01PAP0010041007, DF=100	Unknown	100	PFOA	312691	N/A	140	71205ER-229
139	C0049883 Rep	DF09V01PAP0010041007, DF=100	Unknown	100	PFOA	332124	N/A	150	71205ER-230
140	C0049883	DF09V01PAP0010041007, DF=10	Unknown	10	PFOA	3068314	N/A	155	71205ER-231
141	C0049883 Rep	DF09V01PAP0010041007, DF=10	Unknown	10	PFOA	3210366	N/A	162	71205ER-232
142	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1098166	5	5.41	71205ER-233
143	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	280192	25	123	71205ER-234
144	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	2875699	25	145	71205ER-235
145	C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=10	Quality Control	100	PFOA	760917	250	369	71205ER-236
146	C0049884	DF09V01RCP0010041007, DF=100	Unknown	100	PFOA	240782	N/A	103	71205ER-237
147	C0049884 Rep	DF09V01RCP0010041007, DF=100	Unknown	100	PFOA	240655	N/A	103	71205ER-238
148	C0049884	DF09V01RCP0010041007, DF=10	Unknown	10	PFOA	2499185	N/A	126	71205ER-239
149	C0049884 Rep	DF09V01RCP0010041007, DF=10	Unknown	10	PFOA	2432541	N/A	122	71205ER-240
150	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2158798	10	10.8	71205ER-241
151	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100	PFOA	846576	25	413	71205ER-242
152	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=10	Quality Control	10	PFOA	8090144	25	412	71205ER-243
153	C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=10	Quality Control	100	PFOA	1243986	250	616	71205ER-244
154	C0049885	DF09V02RCP0010041007, DF=100	Unknown	100	PFOA	740390	N/A	358	71205ER-245
155	C0049885 Rep	DF09V02RCP0010041007, DF=100	Unknown	100	PFOA	835235	N/A	407	71205ER-246
156	C0049885	DF09V02RCP0010041007, DF=10	Unknown	10	PFOA	7352275	N/A	374	71205ER-247
157	C0049885 Rep	DF09V02RCP0010041007, DF=10	Unknown	10	PFOA	8117333	N/A	413	71205ER-248
158	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5011716	25	25.4	71205ER-249
159	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	9590978	50	48.8	71205ER-250

PFOA Confirm. Ion

163	Sample Name	Sample ID	Sample Type	Injection Factor	Analyte Peak Name	Peak Area (cc)	Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
164	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFOA Confirm Ion	2713	0.5	0.617	71205ER-201
165	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFOA Confirm Ion	3234	1	0.833	71205ER-202
166	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFOA Confirm Ion	7510	2.5	2.6	71205ER-203

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Raw Data

	A	B	C	D	E	F	G	H	I	J
167	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFOA Confirm for	12260	5	4.57		71205ER-204
168	SS7275	Calibration Standard, 10 ng/mL	Standard	1.0	PFOA Confirm for	26328	10	10.4		71205ER-205
169	SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PFOA Confirm for	65267	25	26.5		71205ER-206
170	SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PFOA Confirm for	120526	50	49.4		71205ER-207
171	Methanol Wash	Methanol Wash	Unknown	1.0	PFOA Confirm for	372	N/A	< 0		71205ER-208
172	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFOA Confirm for	2441	0.5	0.505		71205ER-209
173	C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	21883	25	855		71205ER-210
174	C0049881 Spike C	DF06V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	221450	25	912		71205ER-211
175	C0049881 Spike D	DF06V02RCP0010041007, 100 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	29002	250	1150		71205ER-212
176	C0049881	DF06V02RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	25094	N/A	988		71205ER-213
177	C0049881 Rep	DF06V02RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	27059	N/A	1070		71205ER-214
178	C0049881	DF06V02RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	218549	N/A	900		71205ER-215
179	C0049881 Rep	DF06V02RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	247284	N/A	1020		71205ER-216
180	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFOA Confirm for	3792	1	1.06		71205ER-217
181	C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	2847	25	67.3		71205ER-218
182	C0049882 Spike E	DF09V01AVP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	22811	25	89.4		71205ER-219
183	C0049882 Spike F	DF09V01AVP0010041007, 100 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	7307	250	252		71205ER-220
184	C0049882	DF09V01AVP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	2150	N/A	38.4		71205ER-221
185	C0049882 Rep	DF09V01AVP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	5168	N/A	163		71205ER-222
186	C0049882	DF09V01AVP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	18584	N/A	71.9		71205ER-223
187	C0049882 Rep	DF09V01AVP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	20915	N/A	81.5		71205ER-224
188	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFOA Confirm for	6756	2.5	2.29		71205ER-225
189	C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	11632	25	431		71205ER-226
190	C0049883 Spike G	DF09V01PAP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	102006	25	417		71205ER-227
191	C0049883 Spike H	DF09V01PAP0010041007, 100 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	13256	250	498		71205ER-228
192	C0049883	DF09V01PAP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	10255	N/A	374		71205ER-229
193	C0049883 Rep	DF09V01PAP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	9926	N/A	360		71205ER-230
194	C0049883	DF09V01PAP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	102439	N/A	419		71205ER-231
195	C0049883 Rep	DF09V01PAP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	105570	N/A	432		71205ER-232
196	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFOA Confirm for	11299	5	4.17		71205ER-233
197	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	9961	25	362		71205ER-234
198	C0049884 Spike I	DF09V01RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	95977	25	392		71205ER-235
199	C0049884 Spike J	DF09V01RCP0010041007, 100 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	17188	250	661		71205ER-236
200	C0049884	DF09V01RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	9967	N/A	362		71205ER-237
201	C0049884 Rep	DF09V01RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	9446	N/A	340		71205ER-238
202	C0049884	DF09V01RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	87602	N/A	358		71205ER-239
203	C0049884 Rep	DF09V01RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	92909	N/A	380		71205ER-240
204	SS7275	Calibration Standard, 10 ng/mL	Standard	1.0	PFOA Confirm for	27085	10	10.7		71205ER-241
205	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	27826	25	1100		71205ER-242
206	C0049885 Spike K	DF09V02RCP0010041007, 10 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	272325	25	1120		71205ER-243
207	C0049885 Spike L	DF09V02RCP0010041007, 100 ng/g, DF=100	Quality Control	100.0	PFOA Confirm for	34228	250	1370		71205ER-244
208	C0049885	DF09V02RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	24787	N/A	975		71205ER-245
209	C0049885 Rep	DF09V02RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	27153	N/A	1070		71205ER-246
210	C0049885	DF09V02RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	244370	N/A	1010		71205ER-247
211	C0049885 Rep	DF09V02RCP0010041007, DF=100	Unknown	100.0	PFOA Confirm for	276198	N/A	1140		71205ER-248
212	SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PFOA Confirm for	62848	25	25.5		71205ER-249
213	SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PFOA Confirm for	119203	50	48.8		71205ER-250

0397

BMC 07/28/15

Vegetation Set #3:
Internal Chain of Custody/Fortification
Sheets

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.:	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	0829	07/06/05	CEH

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0073483 Control	5.0	—
—	C0073483 Spike A	5.0	2.5 ng/g
—	C0073483 Spike B	5.0	10 ng/g
BAK 07/19/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (µL)	Micro pipet Syringe ① Volume (µL)	Initials/Date
Spike A	SS0007404 (0.1 µg/mL)	125	25/50	BAK 07/13/05
Spike B	SS0007404 (0.1 µg/mL)	500	100/200	BAK 07/13/05
Spike — ②	—	—	—	—
Spike —	—	—	—	—

All samples were measured on:			
Balance ID:	Time:	Date:	Initials:
BALANCE 20	1100	07/06/05	CEH

After measuring samples were returned to:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1225	07/06/05	CEH

Comments: ① (CC) BAK 07/13/05
 ② (RE) BAK 07/13/05

Analysis Summary	Data Set ID:	071305C	Initials/Date:	AMB / 07/19/05
	Data Set ID:	071305CR	Initials/Date:	AMB / 07/26/05
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:
	MB / 08/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Oxygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Oxygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	0073403 Control	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	-	↓	Methanol	RE0020194
---	0073403 Spk A	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	-	↓	Acetonitrile	RE0020233
---	0073403 Spk B	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	-	↓	Carbon	RE0019757
															Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019970
															2% Ascorbic Acid in Methanol	SLO014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SLO014640
															Toluene	RE0007267
Initials/Date	-	See ICOC page.	BAK 07/13/05	BAK 07/12/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/14/05	BAK 07/14/05	---	AMB 07/19/05	-	AMB 07/24/05	Extraction Items Initials/Date	BAK 07/13/05
															Analysis Items Initials/Date	* AMB 07/19/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
 STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: 1N0000605
 STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
 STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: 1N0000605 Glassware silanized: initials/Date 07/13/05 BAK
 STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: 1N0000605
 STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: 1N0000605
 STEP 7 = Repeat steps 5 through 6 again.
 STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C. *After this step, samples were covered w/ foil and stored in Refrigerator 32 (1N0000708) overnight.*
 STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
 STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
 STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/14/05

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ : FREEZER 34	Time: 1002	Date: 07/06/05	Initials: <u>CEB</u>

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
_____	C0049886 Spike C	5.0	10 ^{ng} /g
_____	C0049886 Spike D	5.0	100 ^{ng} /g
_____	C0049886	5.0	10 ^{ng} /g*
_____	C0049886 Rep.	5.0	10 ^{ng} /g*

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	^{Micropipet} Syringe ① Volume (μL)	Initials/Date
Spike C	SS0007404 (0.1 ^{μg} /mL)	500	100/200	BAK 07/13/05
Spike D	SS0007403 (1.0 ^{μg} /mL)	500	100/200	BAK 07/13/05
Spike *	SS0007505 (0.1 ^{μg} /mL ^{13C})	500	100/200	BAK 07/13/05
Spike _____	_____	_____	_____	_____

All samples were measured on:			
Balance ID: BALANCE 20	Time: 1100	Date: 07/06/05	Initials: <u>CEB</u>

After measuring samples were returned to:			
CED ID ¹ : FREEZER 34	Time: 1225	Date: 07/06/05	Initials: <u>CEB</u>

Comments: ① ② BAK 07/13/05

Analysis Summary	Data Set ID:	071305C	Initials/Date:	AMB / 07/19/05
	Data Set ID:	071305CR	Initials/Date:	AMB / 07/26/05
	Data Set ID:	_____	Initials/Date:	/ / _____

Set extraction/analysis data verified by	Initials/Date:	<u>RF</u> / 08/22/05
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¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Oxygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Oxygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used *	ExyLIMS Inventory ID
---	CO049006 Spk C	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	① 0.1 0.1 0.1 10 10 10	↓	Methanol	R#0020194
---	CO049006 Spk D	↓	↓	↓	↓	↓	↓	↓	↓	↓	0.1/1.0	↓	② 0.1 0.1 10 10	↓	Acetonitrile	R#0020233
---	CO049006	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	② 0.1 0.1 10 10	↓	Carbon	R#0019757
---	CO049006 Rep.	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	② 0.1 0.1 10 10	↓	Silica gel	R#0000639
Florisil R#0000641																
LC-NH2 R#0019970																
2% Ascorbic Acid in Methanol S#0014702																
1-Octanol R#0016118																
30% Dimethyldichlorosilane in Toluene Solution S#0014640																
Toluene R#0007267																
Initials/Date	-	See ICOC page.	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/14/05	BAK 07/14/05	AMB 07/19/05	AMB 07/19/05	AMB 07/26/05	AMB 07/26/05	Extraction Items Initials/Date	BAK 07/13/05
															Analysis Items Initials/Date	* AMB 07/19/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: 1N0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: 1N0000182 Glassware silanized: initials/Date BAK 07/19/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: 1N0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: 1N0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

After this step, samples were covered w/ foil and stored in Refrigerator 32 (1N0000708) overnight - BAK 07/13/05

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/14/05

0402

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1002	07/06/05	CEH

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
_____	C0049887 Spike E	5.0	10 ^{ng/g}
_____	C0049887 Spike F	5.0	100 ^{ng/g}
_____	C0049887	5.0	10 ^{ng/g} *
_____	C0049887 Rep.	5.0	10 ^{ng/g} *
BAK 07/13/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (µL)	Micropipet		Initials/Date
			Syringe ^① Volume (µL)		
Spike E	SS0007404 (0.1 ^{µg} /mL)	500	100/200		BAK 07/13/05
Spike F	SS0007403 (1.0 ^{µg} /mL)	500	100/200		BAK 07/13/05
Spike *	SS0007505 (0.1 ^{µg} /mL ^{12C})	500	100/200		BAK 07/13/05
Spike _____	_____	_____	_____	_____	_____

All samples were measured on:			
Balance ID:	Time:	Date:	Initials:
BALANCE 20	1100	07/06/05	CEH

After measuring samples were returned to:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1225	07/06/05	CEH

Comments: (100) BAK 07/13/05

Analysis Summary	Data Set ID:	07/305C	Initials/Date:	AMB / 07/19/05
	Data Set ID:	07/305CR	Initials/Date:	AMB / 07/26/05
	Data Set ID:	_____	Initials/Date:	_____ / _____

Set extraction/analysis data verified by	Initials/Date:
	BAK / 08/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Oxygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Oxygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	COO49887	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	① 0.1 0.1 0.1 T.D T.D T.D	↓	Methanol	RE0020194
---	COO49887	↓	↓	↓	↓	↓	↓	↓	↓	↓	0.1/1.0	↓	② 0.1 0.1 T.D T.D	↓	Acetonitrile	RE0020233
---	COO49887	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	③ 0.1 0.1 0.1 T.D T.D T.D	↓	Carbon	RE0019751
---	COO49887	↓	↓	↓	↓	↓	↓	↓	↓	↓	---	↓	④ 0.1 0.1 0.1 T.D T.D T.D	↓	Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019970
															2% Ascorbic Acid in Methanol	SLO014702
															1-Octanol	RE0016118
															30% Dimethylchlorosilane in Toluene Solution	SLO014640
															Toluene	RE0007267
Initials/Date	-	See ICOC page.	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/14/05	BAK 07/14/05	AMB 07/19/05	AMB 07/19/05	AMB 07/20/05	AMB 07/20/05	Extraction Items Initials/Date	BAK 07/13/05
															Analysis Items Initials/Date	*AMB 07/19/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
 STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605
 STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
 STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/13/05
 STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605
 STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182
 STEP 7 = Repeat steps 5 through 6 again. → After this step, samples were covered w/ foil
 STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
 STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial. and stored in Refrigerator 32 (IN0000708) overnight.
 STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
 STEP 11 = Perform LC/MS/MS re-analysis.
 COMMENTS: BAK 07/13/05

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/14/05

F0404

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1002	07/06/05	SLB

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	C0049888 Spike G	5.0	10 ^{ng/g}
—	C0049888 Spike H	5.0	100 ^{ng/g}
—	C0049888	5.0	10 ^{ng/g} *
—	C0049888 Rep.	5.0	10 ^{ng/g} *
BRK 07/13/05			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (µL)	Microplet	
			-Syringe (D) Volume (µL)	Initials/Date
Spike G	SS0007404 (0.1 ^{µg/mL})	500	100/200	BRK 07/13/05
Spike H	SS0007403 (1.0 ^{µg/mL})	500	100/200	BRK 07/13/05
Spike *	SS0007505 (0.1 ^{µg/mL} ?)	500	100/200	BRK 07/13/05
Spike —	—	—	—	—

All samples were measured on:			
Balance ID:	Time:	Date:	Initials:
BALANCE 20	1100	07/06/05	SLB

After measuring samples were returned to:			
CED ID ¹ :	Time:	Date:	Initials:
FREEZER 34	1225	07/06/05	SLB

Comments: (1) (C) BRK 07/13/05

Analysis Summary	Data Set ID:	071305C	Initials/Date:	AMB / 07/19/05
	Data Set ID:	071305CR	Initials/Date:	AMB / 07/26/05
	Data Set ID:	—	Initials/Date:	— / —

Set extraction/analysis data verified by	Initials/Date:
	DF / 08/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID	
---	CO049008 Spk G										---		① 0.1 T.D 0.1 T.D 0.1 T.D		Methanol	RE0020194	
---	CO049008 Spk H										0.1/1.0		② 0.1 T.D 0.1 T.D		Acetonitrile	RE0020233	
---	CO049008										---		① 0.1 T.D 0.1 T.D 0.1 T.D		Carbon	RE0019751	
---	CO049008 Rep.	↓	↓	↓	↓	↓	↓	↓	↓	↓	---		① 0.1 T.D 0.1 T.D 0.1 T.D	↓	Silica gel	RE0000639	
															Florisil	RE0000641	
															LC-NH2	RE0019970	
															2% Ascorbic Acid in Methanol	SL0014702	
															1-Octanol	RE0016118	
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640	
															Toluene	RE0007267	
Initials/Date	-	See ICOC page.	BRK 07/13/05	BRK 07/12/05	BRK 07/13/05	BRK 07/13/05	BRK 07/13/05	BRK 07/13/05	BRK 07/13/05	BRK 07/14/05	BRK 07/14/05	AMB 07/19/05	AMB 07/19/05	AMB 07/26/05	AMB 07/26/05	Extraction Items Initials/Date	BRK 07/13/05
																Analysis Items Initials/Date	* AMB 07/19/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: 1N0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: 1N0000182 Glassware silanized: initials/Date BRK 07/13/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: 1N0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: 1N0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BRK Date: 07/14/05

① DF = 10, 100, 1000 > AMB 07/26/05
② DF = 100, 1000 > AMB 07/26/05

After this step, samples were covered w/ foil and stored in Refrigerator 32 (1N0000708) overnight. BRK 07/13/05

0406

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Exygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID ¹ : FREEZER 34	Time: 1002	Date: 07/06/05	Initials: <i>CEH</i>

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
—	CO049889 Spike I	5.0	10 ^{ng/g}
—	CO049889 Spike J	5.0	100 ^{ng/g}
—	CO049889	5.0	10 ^{ng/g} *
—	CO049889 Rep.	5.0	10 ^{ng/g} *
<i>BRK 07/13/05</i>			

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (μL)	^{Micro pipet} Syringe ① Volume (μL)	Initials/Date
Spike I	SS0007404 (0.1 ^{μg/mL})	500	100/200	<i>BRK 07/13/05</i>
Spike J	SS0007403 (1.0 ^{μg/mL})	500	100/200	<i>BRK 07/13/05</i>
Spike *	SS0007505 (0.1 ^{μg/mL} ¹³ C)	500	100/200	<i>BRK 07/13/05</i>
Spike —	—	—	—	—

All samples were measured on:			
Balance ID: BALANCE 20	Time: 1100	Date: 07/06/05	Initials: <i>CEH</i>

After measuring samples were returned to:			
CED ID ¹ : FREEZER 34	Time: 1225	Date: 07/06/05	Initials: <i>CEH</i>

Comments: ①② *BRK 07/13/05*

Analysis Summary	Data Set ID:	071305C	Initials/Date:	<i>AMB 1 07/19/05</i>
	Data Set ID:	071305R	Initials/Date:	<i>AMB 1 07/26/05</i>
	Data Set ID:	—	Initials/Date:	—

Set extraction/analysis data verified by	Initials/Date:	<i>BRK 1 08/22/05</i>
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¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Oxygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Oxygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
///	00049889 Spk 1	↓	↓	↓	↓	↓	↓	↓	↓	↓	—	↓	① 0.1 0.1 0.1 1.0 1.0 1.0	↓	Methanol	RE0020194
///	00049889 Spk 2	↓	↓	↓	↓	↓	↓	↓	↓	↓	0.1/1.0	↓	② 0.1 0.1 1.0 1.0	↓	Acetonitrile	RE0020233
///	00049889 Spk 3	↓	↓	↓	↓	↓	↓	↓	↓	↓	—	↓	① 0.1 0.1 0.1 1.0 1.0 1.0	↓	Carbon	RE0019751
///	00049889 Rep	↓	↓	↓	↓	↓	↓	↓	↓	↓	—	↓	① 0.1 0.1 0.1 1.0 1.0 1.0	↓	Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019970
															2% Ascorbic Acid in Methanol	SL0014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640
															Toluene	RE0007267
Initials/Date	—	See ICOC page.	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/14/05	BAK 07/14/05	AMB 07/19/05	AMB 07/19/05	AMB 07/20/05	AMB 07/20/05	Extraction Items Initials/Date	BAK 07/13/05
															Analysis Items Initials/Date	AMB 07/19/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

① DF = 10, 100, 1000
② DF = 100, 1000
AMB 07/20/05

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
 STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605
 STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
 STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/13/05
 STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605
 STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182
 STEP 7 = Repeat steps 5 through 6 again.
 STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C. → After this step, samples were covered w/ foil and stored in Refrigerator 32 (IN0000708) overnight.
 STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
 STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
 STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/14/05

0408

INTERNAL CHAIN OF CUSTODY/FORTIFICATION SHEET FOR SOLID SAMPLES

Oxygen Study/Project No.:	Protocol No.	Matrix:
P760 and P1131	P0000760 and P0001131	Vegetation

The samples listed below were removed from:			
CED ID:	Time:	Date:	Initials:
FREEZER 34	1002	07/06/05	CBH

CLIENT SAMPLE ID	EXYGEN ID NUMBER	WEIGHT (g)	FORTIFICATION
_____	C0049890 Spike K	5.0	10 mg/g
_____	C0049890 Spike L	5.0	100 mg/g
_____	C0049890	5.0	10 mg/g*
_____	C0049890 Rep.	5.0	10 mg/g*

Spike Sample ID Number	Spiking Solution Used (ID and Concentration)	Volume Used for Spiking (µL)	Microinjector		Initials/Date
			Syringe	Volume (µL)	
Spike K	SS0007404 (0.1 µg/mL)	500	100/200	100/200	BAL 07/13/05
Spike L	SS0007403 (1.0 µg/mL)	500	100/200	100/200	BAL 07/13/05
Spike *	SS0007505 (0.1 µg/mL ¹³ C)	500	100/200	100/200	BAL 07/13/05
Spike _____	_____	_____	_____	_____	_____

All samples were measured on:			
Balance ID:	Time:	Date:	Initials:
BALANCE 20	1100	07/06/05	CBH

After measuring samples were returned to:			
CED ID:	Time:	Date:	Initials:
FREEZER 34	1225	07/06/05	CBH

Comments: ① ② BAL 07/13/05

Analysis Summary	Data Set ID:	071305C	Initials/Date:	AMB / 07/19/05
	Data Set ID:	071305CR	Initials/Date:	AMB / 07/20/05
	Data Set ID:		Initials/Date:	

Set extraction/analysis data verified by	Initials/Date:
	EC / 08/22/05

¹ CED = Controlled environment device; specify the type of device and number (e.g., Walkin Freezer 8)

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

FOR METHOD: "Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Vegetation by LC/MS/MS" V0001784

Exygen Study/Project Number: P760/P1131

Matrix: Vegetation

Client Sample ID	Exygen Sample ID	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9	Dilutions (ml/ml)	Step 10	Dilutions (ml/ml)	Step 11	Reagents, Solutions, and Materials Used	ExyLIMS Inventory ID
---	00049890 SPK										---		① 0.1 0.1 0.1 T.D T.D T.D		Methanol	RE0020194
---	00049890 SPK										0.1/1.0		② T.D T.D T.D T.D		Acetonitrile	RE0020233
---	00049890										---		① 0.1 0.1 0.1 T.D T.D T.D		Carbon	RE0019751
---	00049890 Rep.	✓	✓	✓	✓	✓	✓	✓	✓	✓	---		① 0.1 0.1 0.1 T.D T.D T.D	✓	Silica gel	RE0000639
															Florisil	RE0000641
															LC-NH2	RE0019970
															2% Ascorbic Acid in Methanol	SL0014702
															1-Octanol	RE0016118
															30% Dimethyldichlorosilane in Toluene Solution	SL0014640
															Toluene	RE0007267
Initials/Date	-	See ICOC page.	BAK 07/13/05	BAK 07/12/05	BAK 07/13/05	BAK 07/13/05	BAK 07/13/05	BAK 07/14/05	BAK 07/14/05	BAK 07/14/05	AMB 07/19/05	AMB 07/19/05	AMB 07/20/05	AMB 07/20/05	Extraction Items Initials/Date	BAK 07/13/05
															Analysis Items Initials/Date	*AMB 07/19/05

* See LC/MS/MS conditions pages for reagent and solution information for mobile phase.

- STEP 1 = Measure 5 g of frozen sample into a 50 mL polypropylene centrifuge tube.
- STEP 2 = Add 30 mL ACN and shake for ~15 min. Shaker ID: IN0000605
- STEP 3 = Pack and condition SPE Columns. (Pack with ~2g of florisil, silica gel, carbon, and ~1g of LC-NH2) (Condition with 20 mL methanol, 20 mL ACN) Do not let column go dry.
- STEP 4 = Centrifuge at ~2000 rpm for ~10 minutes, Decant onto SPE and collect in silanized flask. Centrifuge ID: IN0000182 Glassware silanized: initials/Date BAK 07/13/05
- STEP 5 = Add 20 mL ACN to 50 mL centrifuge tube, shake for ~10 min. Shaker ID: IN0000605
- STEP 6 = Centrifuge at ~2000 rpm for ~5 minutes, Decant onto same SPE column and collect in the same silanized flask. Centrifuge ID: IN0000182
- STEP 7 = Repeat steps 5 through 6 again.
- STEP 8 = Add 3-4 drops of 1-octanol, evaporate using rotovap at <40°C.
- STEP 9 = Add 2 mL 2% ascorbic acid in methanol, mix, transfer to HPLC vial.
- STEP 10 = Perform LC/MS/MS analysis for PFOA, PFBS, PFHS, and PFOS.
- STEP 11 = Perform LC/MS/MS re-analysis.

COMMENTS:

After this step, the samples were covered w/ foil and stored in Refrigerator 32 (IN0000708) overnight.
BAK 07/13/05

Final extracts stored in Refrigerator # 32 Initials: BAK Date: 07/14/05

0410

Vegetation Set #3

Data Set 071305C

Extracted on 07/13/05

(Analyzed on 07/19-20/05)

AMB 07/19/05

File: \\Abbeyroad\PE Sciex Data\Projects\P1131 Decatur Monitoring Study Batch:071305C VEG P760 Tab:Sample Set:SET1 AcqMethod:P760-1131 Isomer Separation

Sample Name	Sample ID	Vial Position	Data File	Run #
SS7279	Calibration Standard, 0.5 ng/mL	1	08_071305C\071305C	071305C-301
SS7278	Calibration Standard, 1.0 ng/mL	2	08_071305C\071305C	071305C-302
SS7277	Calibration Standard, 2.5 ng/mL	3	08_071305C\071305C	071305C-303
SS7276	Calibration Standard, 5.0 ng/mL	4	08_071305C\071305C	071305C-304
SS7275	Calibration Standard, 10 ng/mL	5	08_071305C\071305C	071305C-305
SS7274	Calibration Standard, 25 ng/mL	6	08_071305C\071305C	071305C-306
SS7273	Calibration Standard, 50 ng/mL	7	08_071305C\071305C	071305C-307
Methanol Wash	Methanol Wash	92	08_071305C\071305C	071305C-308
Reagent Control	C0054391 Control	11	08_071305C\071305C	071305C-309
Reagent Spk A	C0054391 Spike A, 2.5 ng/g	12	08_071305C\071305C	071305C-310
Reagent Spk B	C0054391 Spike B, 10 ng/g	13	08_071305C\071305C	071305C-311
SS7279	Calibration Standard, 0.5 ng/mL	1	08_071305C\071305C	071305C-312
C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	14	08_071305C\071305C	071305C-313
C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=10	15	08_071305C\071305C	071305C-314
C0049886	DF09V02PAP0010041007	16	08_071305C\071305C	071305C-315
C0049886 Rep	DF09V02PAP0010041007	17	08_071305C\071305C	071305C-316
SS7278	Calibration Standard, 1.0 ng/mL	2	08_071305C\071305C	071305C-317
C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	18	08_071305C\071305C	071305C-318
C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=10	19	08_071305C\071305C	071305C-319
C0049887	DF8bV01PAP0010041007	20	08_071305C\071305C	071305C-320
C0049887 Rep	DF8bV01PAP0010041007	21	08_071305C\071305C	071305C-321
SS7277	Calibration Standard, 2.5 ng/mL	3	08_071305C\071305C	071305C-322
C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	22	08_071305C\071305C	071305C-323
C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=10	23	08_071305C\071305C	071305C-324
C0049888	DF8bV01RCP0010041007	24	08_071305C\071305C	071305C-325
C0049888 Rep	DF8bV01RCP0010041007	25	08_071305C\071305C	071305C-326
SS7276	Calibration Standard, 5.0 ng/mL	4	08_071305C\071305C	071305C-327
C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	26	08_071305C\071305C	071305C-328
C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=10	27	08_071305C\071305C	071305C-329
C0049889	DF8bV02PAP0010041007	28	08_071305C\071305C	071305C-330
C0049889 Rep	DF8bV02PAP0010041007	29	08_071305C\071305C	071305C-331
SS7275	Calibration Standard, 10 ng/mL	5	08_071305C\071305C	071305C-332
C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	30	08_071305C\071305C	071305C-333
C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=10	31	08_071305C\071305C	071305C-334
C0049890	DF8bV02RCP0010041007	32	08_071305C\071305C	071305C-335
C0049890 Rep	DF8bV02RCP0010041007	33	08_071305C\071305C	071305C-336
SS7274	Calibration Standard, 25 ng/mL	6	08_071305C\071305C	071305C-337
SS7273	Calibration Standard, 50 ng/mL	7	08_071305C\071305C	071305C-338

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Study No: NA

Exygen Study No: P760/P1131

Instrument: API 4000 LC/MS/MS System, (LC/MS/MS #8)
Turbo Ion Spray Liquid Introduction Interface

Computer: DELL OptiPlex GX400

Software: Windows NT, Analyst 1.4

HPLC Equipment: 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 (Exygen ID MA0018925)
Column Temperature: 35°C

Mobile Phase (A) : 2 mM ammonium acetate in water (SL0014984)
Mobile Phase (B) : Methanol (RE0020196)

<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

Injected Volume: 15 µL

Ions monitored:

<u>Analyte</u>	<u>Mode</u>	<u>Transition Monitored</u>
PFBS	negative	299 → 99
PFHS	negative	399 → 80
PFOS	negative	499 → 80
PFOA	negative	413 → 369
PFOA Confirm Ion	negative	413 → 219
¹³ C PFOA	negative	415 → 370

Analyst: Amy Sheehan *AMB 07/19/05*
Exygen Research
3058 Research Drive, State College, PA 16801
Phone : (814) 272-1039 FAX : (814) 231-1580

NOTE: ALL HANDWRITTEN PEAK IDENTIFICATIONS BY *AMB* / *07/22/05*

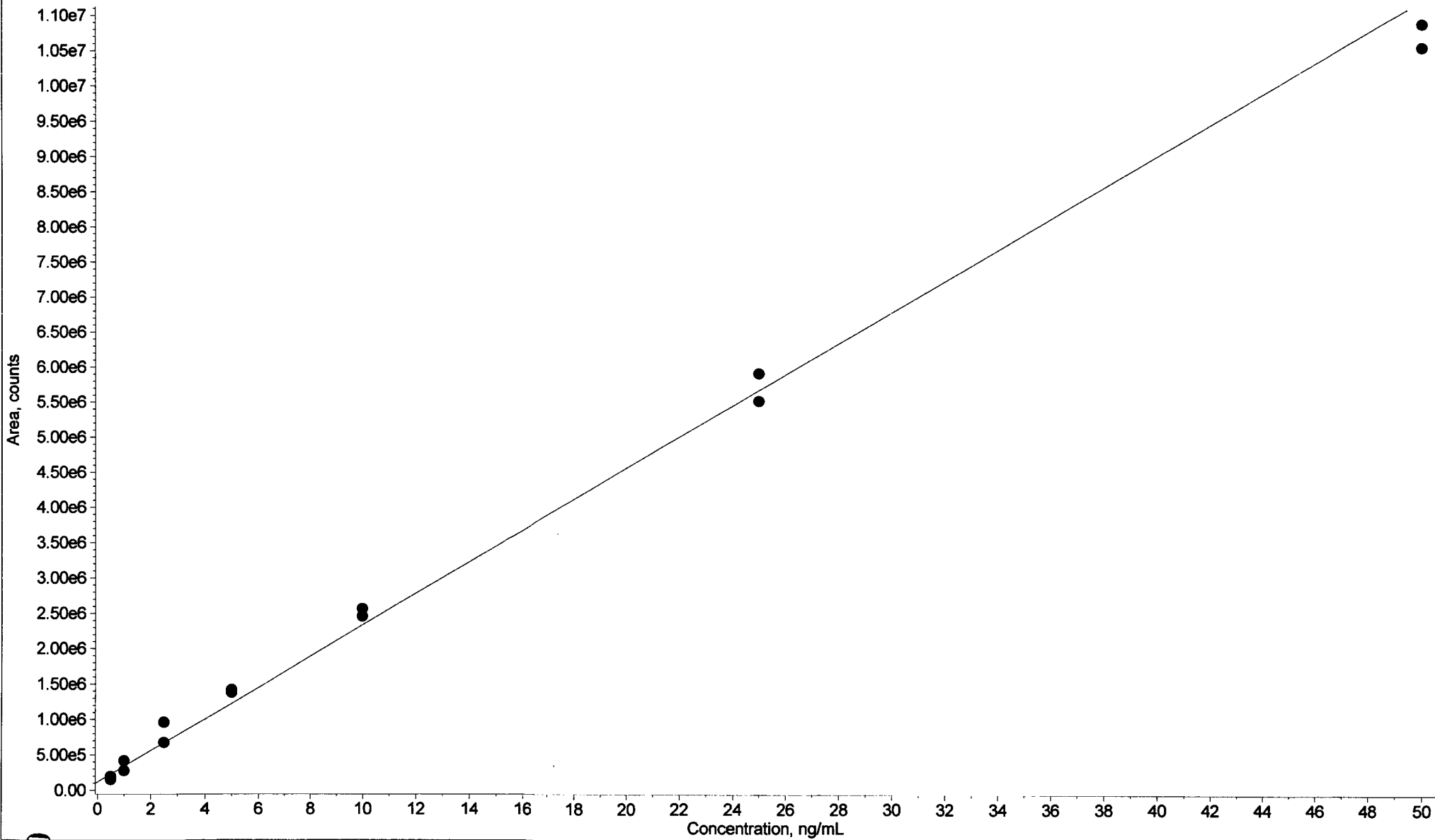
BAK 08/15/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	152030	0.500	0.158	071305C-301
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	276132	1.00	0.716	071305C-302
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	676478	2.50	2.51	071305C-303
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1386295	5.00	5.70	071305C-304
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2572205	10.0	11.0	071305C-305
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5922677	25.0	26.1	071305C-306
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10918506	50.0	48.5	071305C-307
Methanol Wash	Methanol Wash	Unknown	1	PFOA	80114	N/A	< 0	071305C-308
Reagent Control	C0054391 Control	Unknown	1	PFOA	75087	N/A	< 0	071305C-309
Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1	PFOA	1617762	6.25	6.74	071305C-310
Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1	PFOA	5972528	25.0	26.3	071305C-311
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	194021	0.500	0.347	071305C-312
C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1	PFOA	74691305	25.0	335.	071305C-313
C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	20958666	250.	936.	071305C-314
C0049886	DF09V02PAP0010041007	Unknown	1	PFOA	71568996	N/A	321.	071305C-315
C0049886 Rep	DF09V02PAP0010041007	Unknown	1	PFOA	69835640	N/A	313.	071305C-316
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	417242	1.00	1.35	071305C-317
C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1	PFOA	96938481	25.0	435.	071305C-318
C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	32606764	250.	1460.	071305C-319
C0049887	DF8bV01PAP0010041007	Unknown	1	PFOA	94931069	N/A	426.	071305C-320
C0049887 Rep	DF8bV01PAP0010041007	Unknown	1	PFOA	90939799	N/A	408.	071305C-321
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	961364	2.50	3.79	071305C-322
C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1	PFOA	49847301	25.0	223.	071305C-323
C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	13495888	250.	601.	071305C-324
C0049888	DF8bV01RCP0010041007	Unknown	1	PFOA	49634130	N/A	222.	071305C-325
C0049888 Rep	DF8bV01RCP0010041007	Unknown	1	PFOA	47262880	N/A	212.	071305C-326
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1424412	5.00	5.88	071305C-327
C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1	PFOA	86680092	25.0	389.	071305C-328
C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	29165507	250.	1310.	071305C-329
C0049889	DF8bV02PAP0010041007	Unknown	1	PFOA	85792086	N/A	385.	071305C-330
C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	PFOA	81391265	N/A	365.	071305C-331
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2463709	10.0	10.5	071305C-332
C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	PFOA	52748335	25.0	236.	071305C-333
C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA	16202334	250.	723.	071305C-334
C0049890	DF8bV02RCP0010041007	Unknown	1	PFOA	52117601	N/A	234.	071305C-335
C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	PFOA	50090193	N/A	225.	071305C-336
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5527250	25.0	24.3	071305C-337
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10584455	50.0	47.0	071305C-338

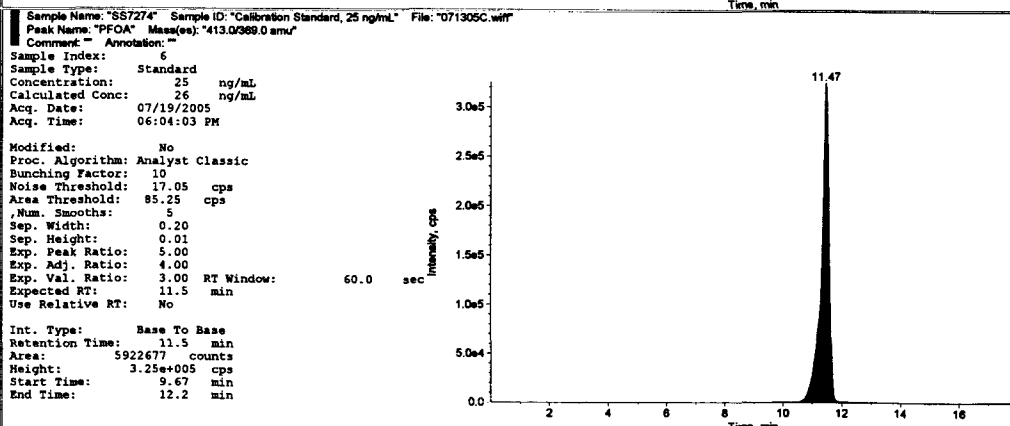
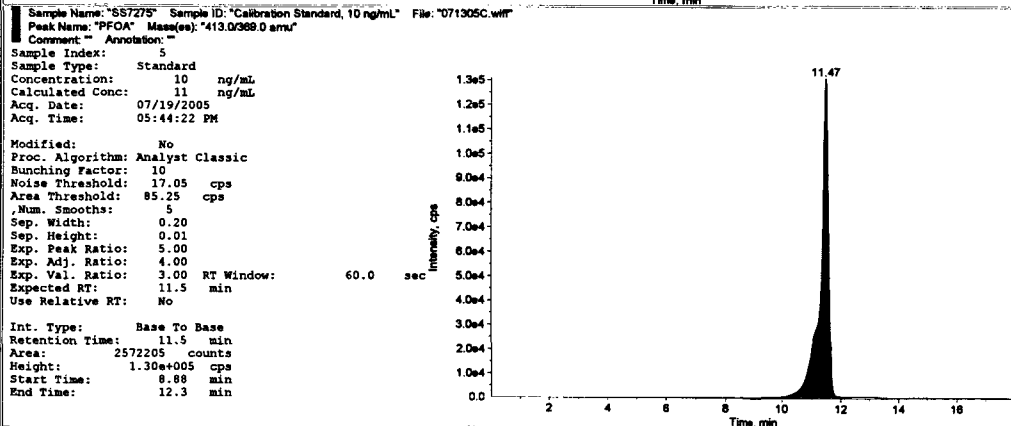
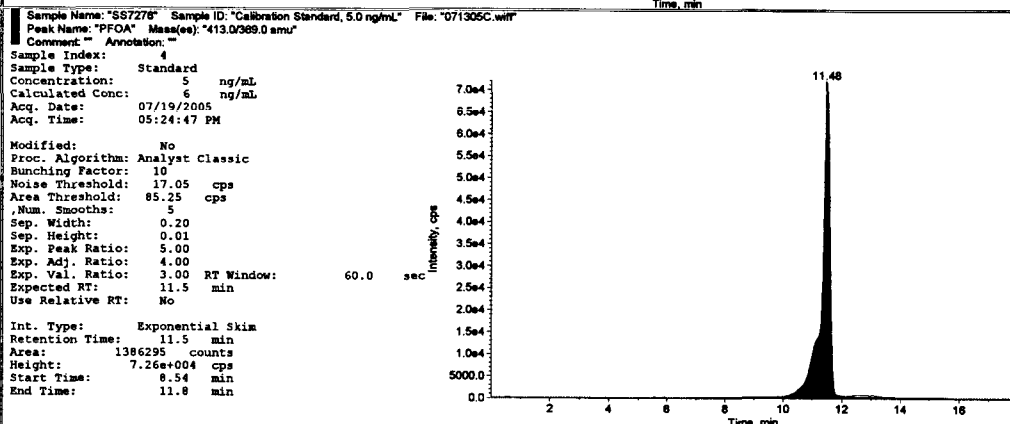
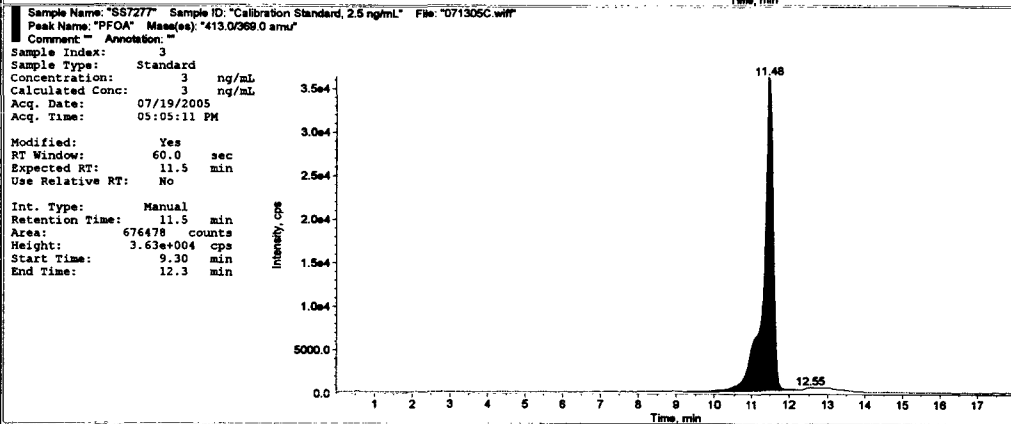
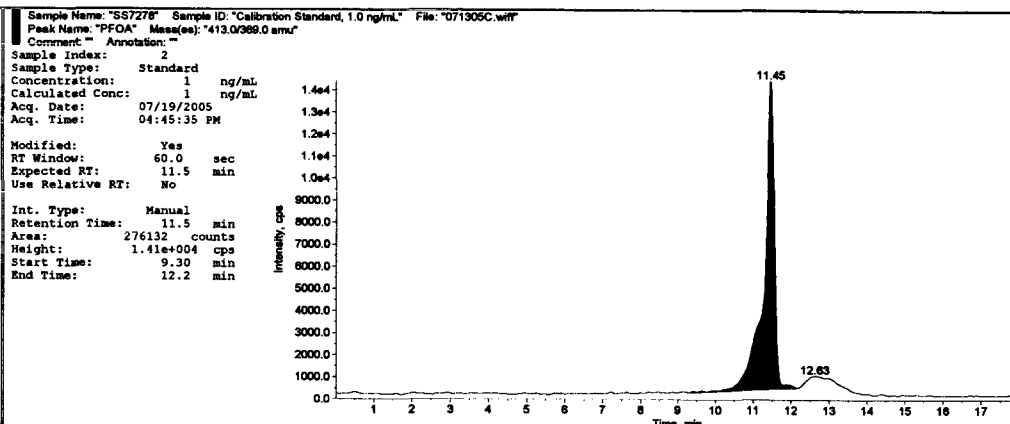
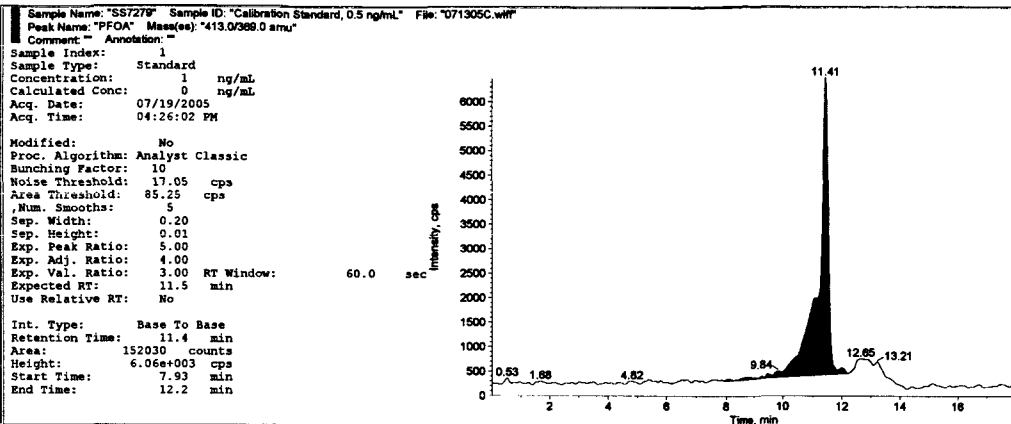
① Dilution required, see data set 071305CR. BAK 08/15/05

AMB 07/22/05

■ Untitled 3 (PFOA): "Linear" Regression ("1 / x" weighting): $y = 2.23e+005 x + 1.17e+005$ ($r = 0.9944$)

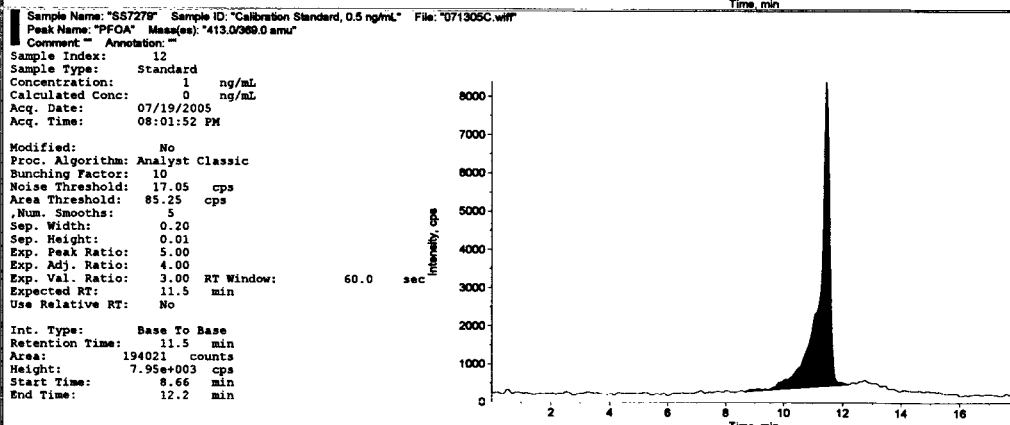
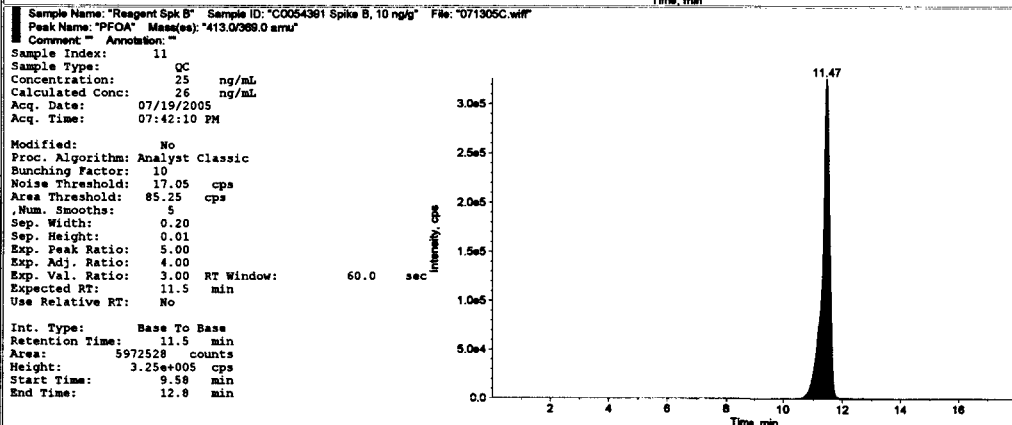
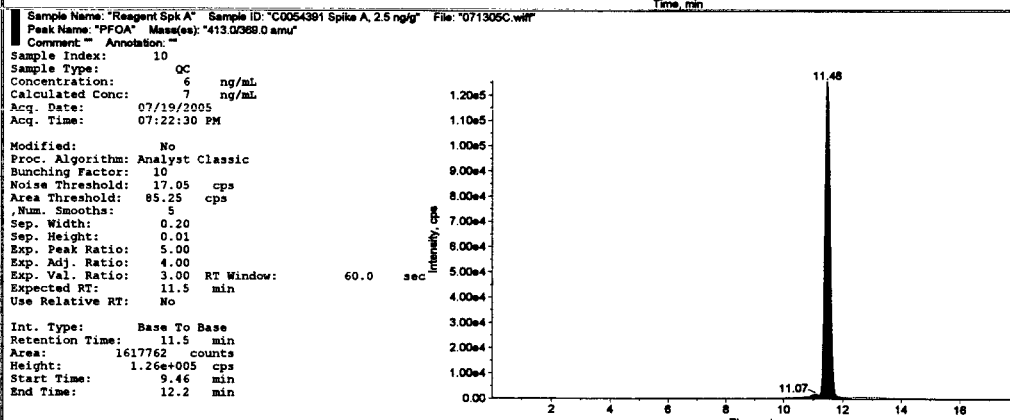
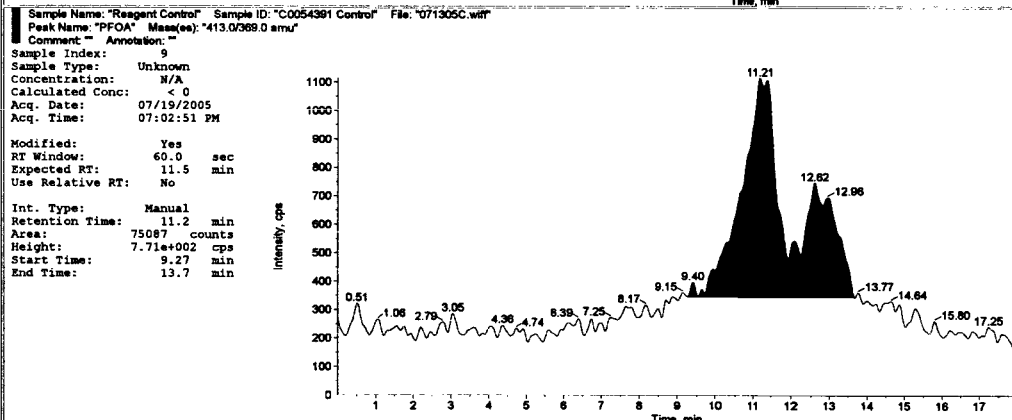
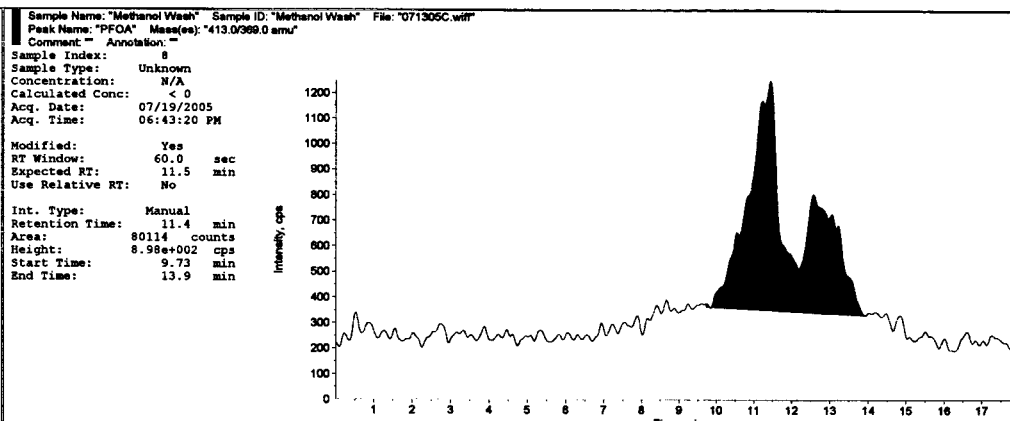
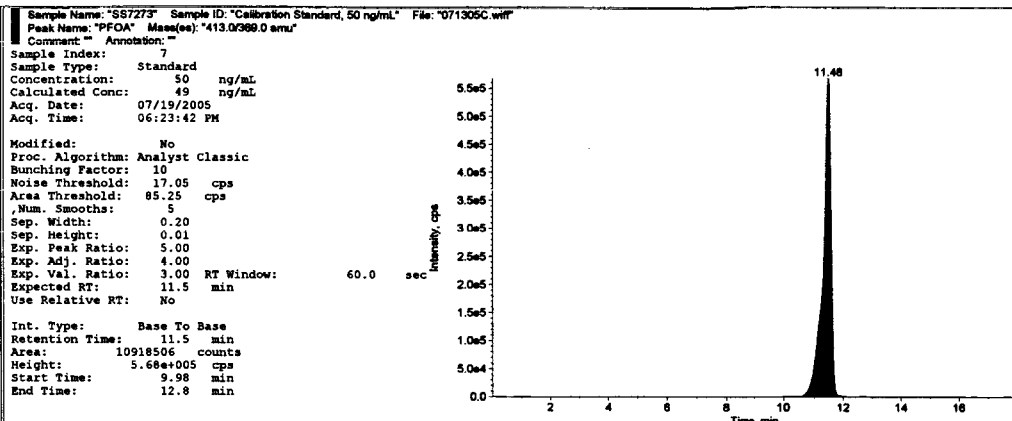


0415

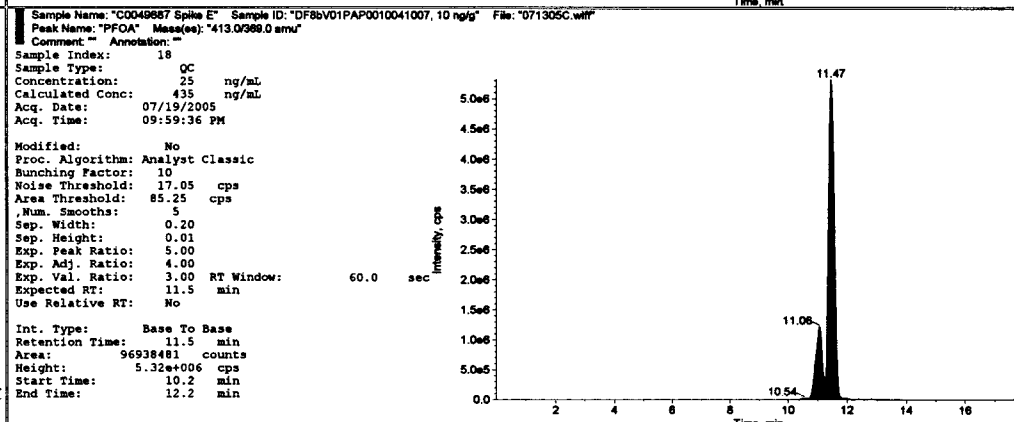
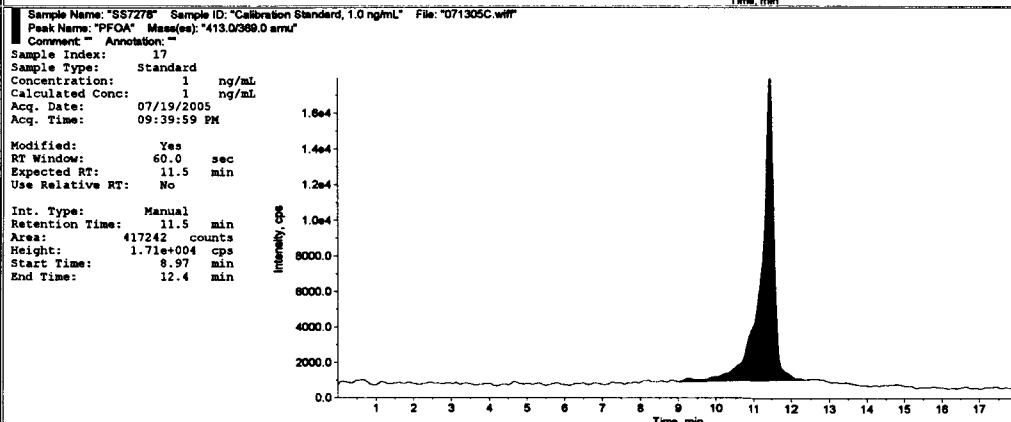
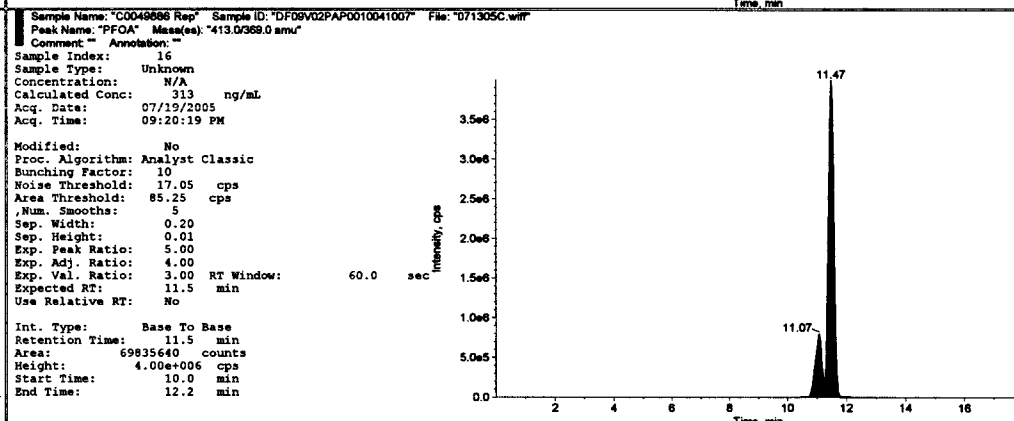
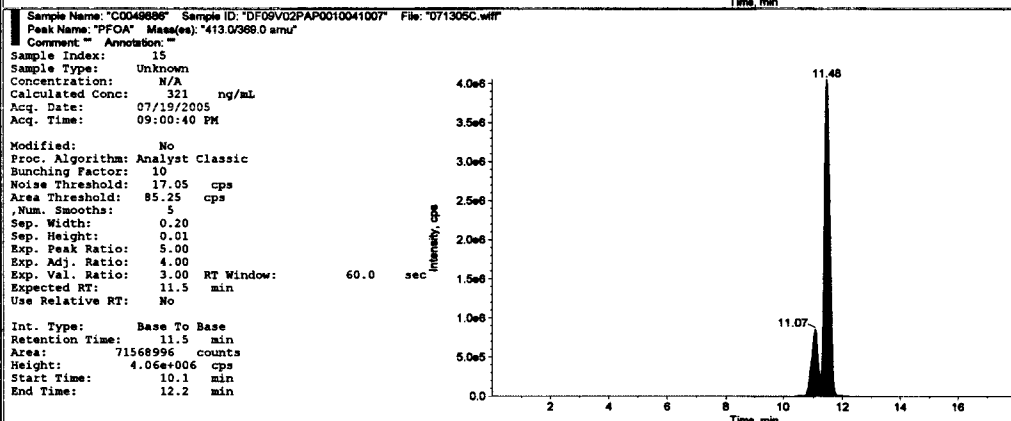
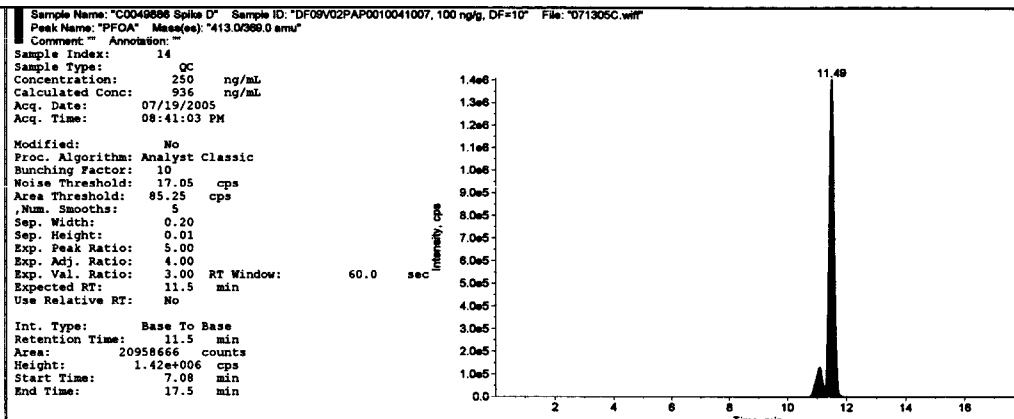
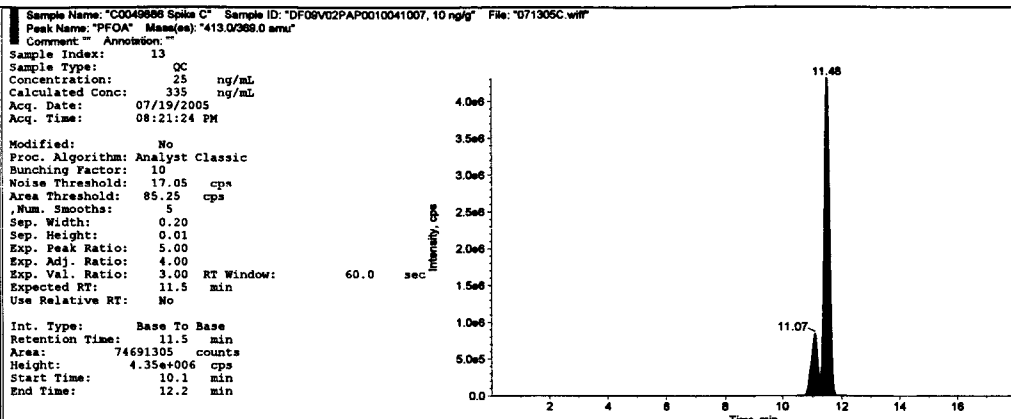


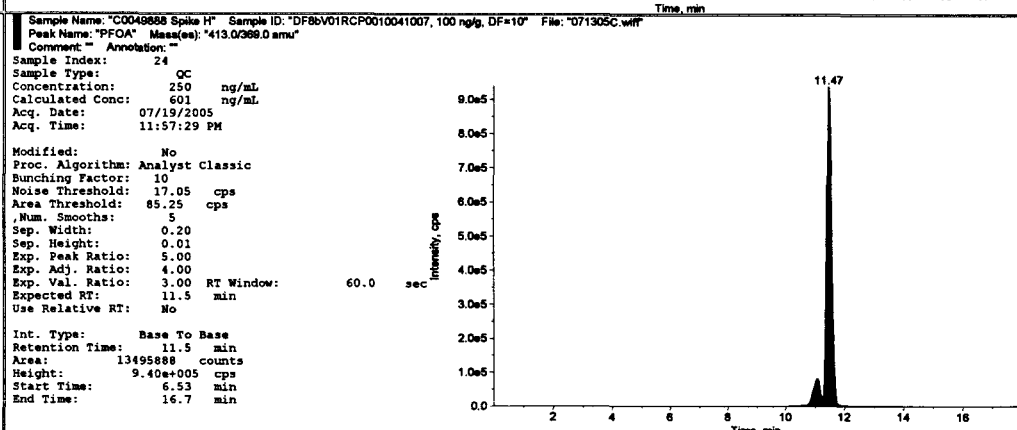
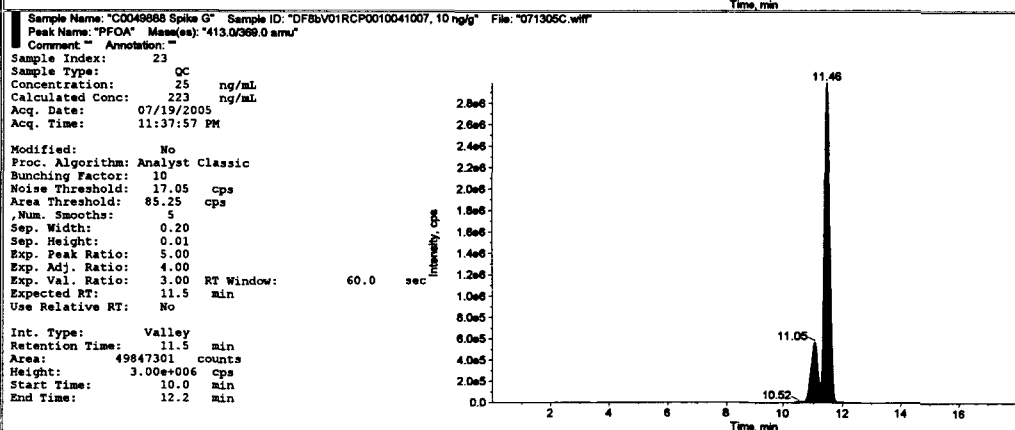
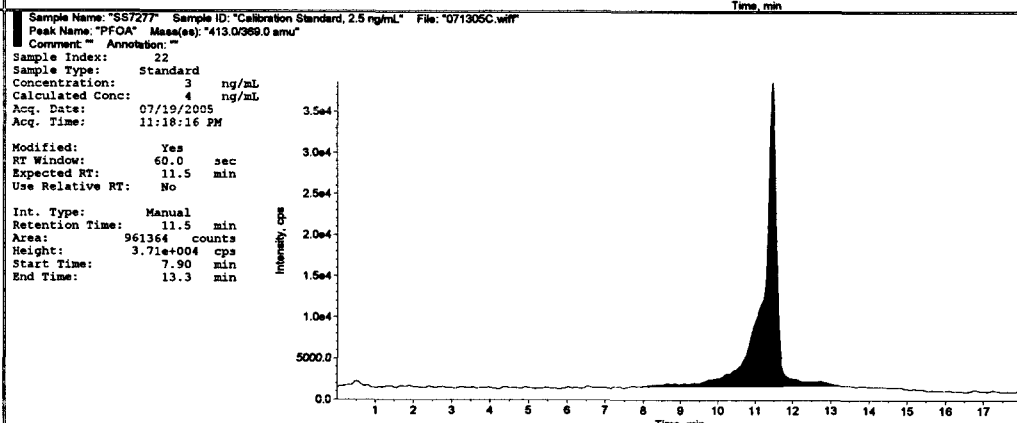
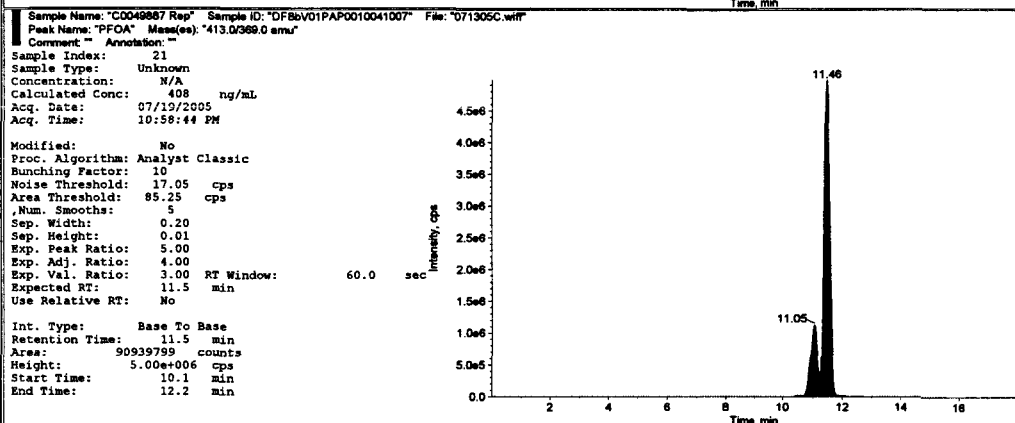
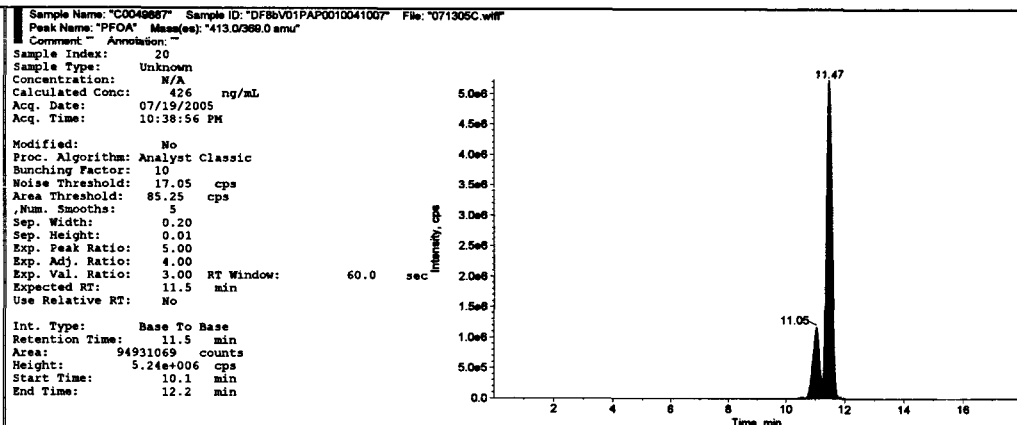
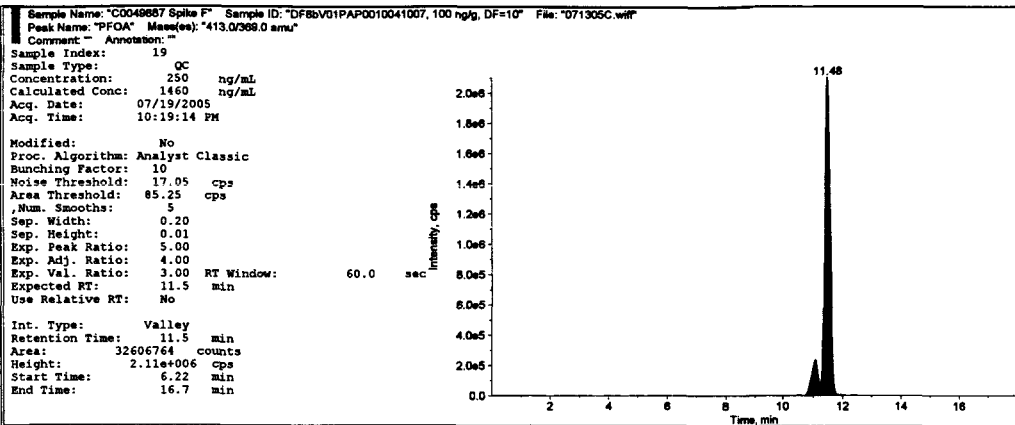
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 Date: 07/22/05
 Sample Index: 1 To 38
 @ AMB 07/22/05

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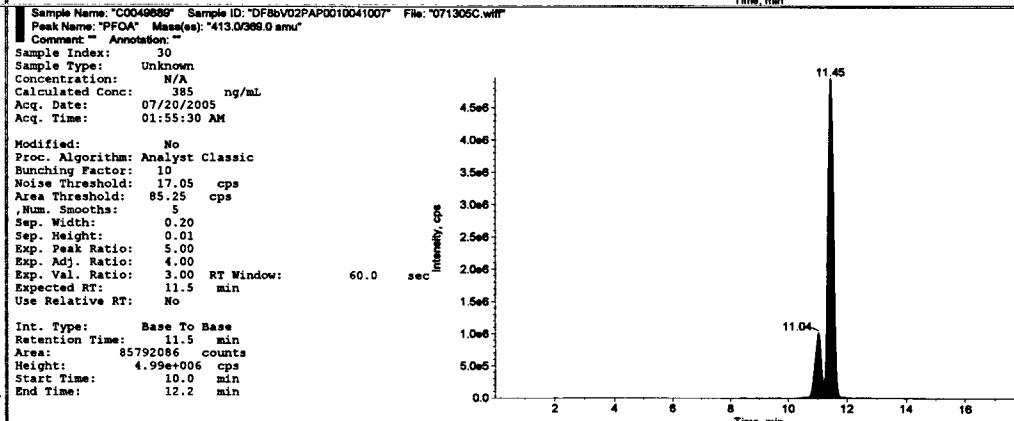
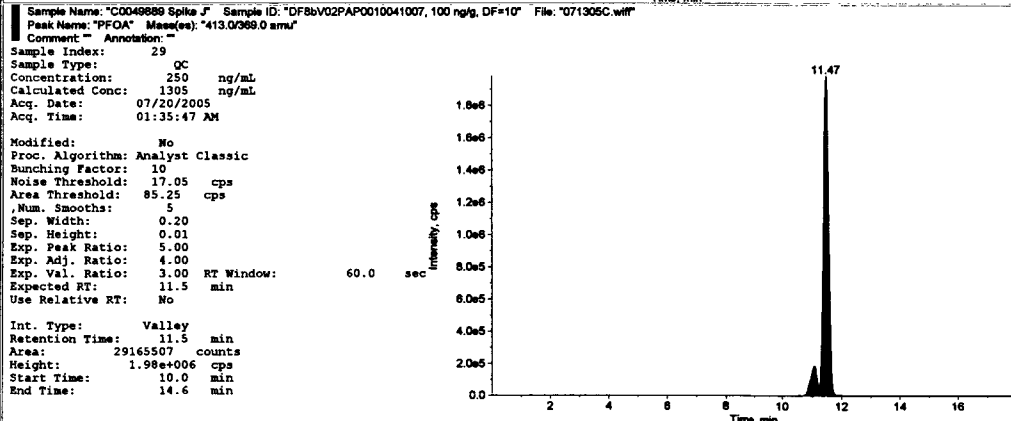
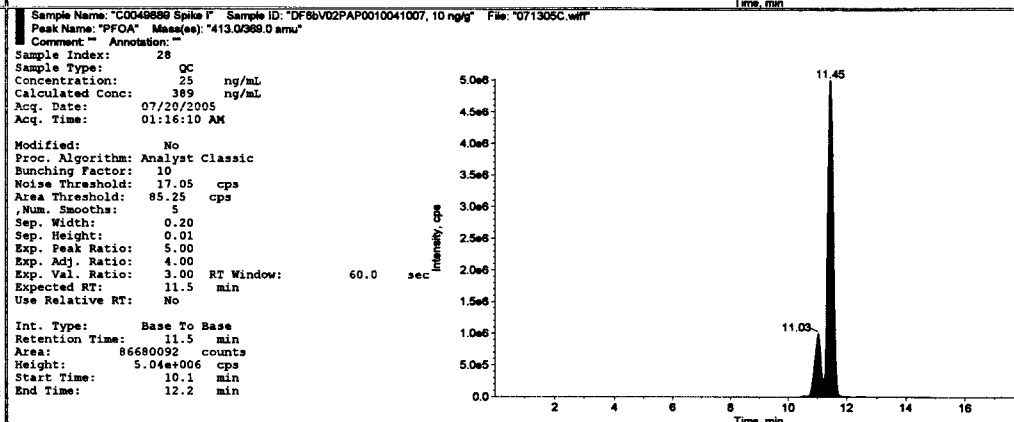
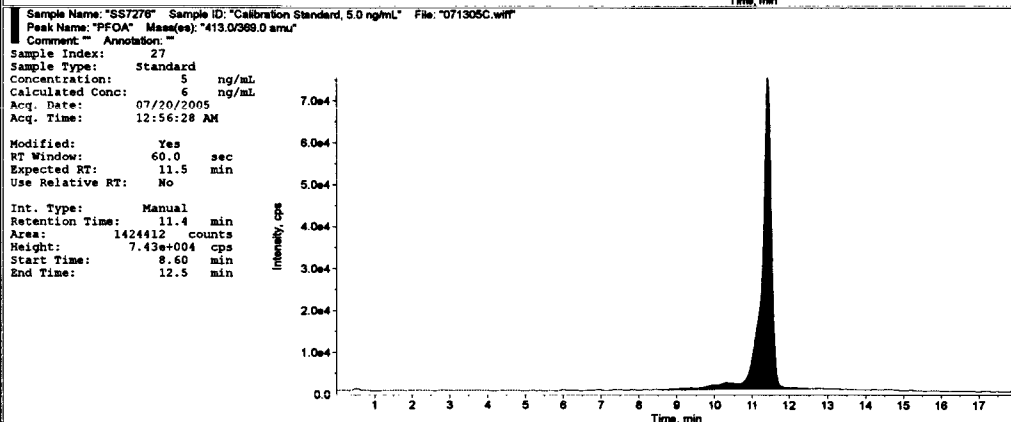
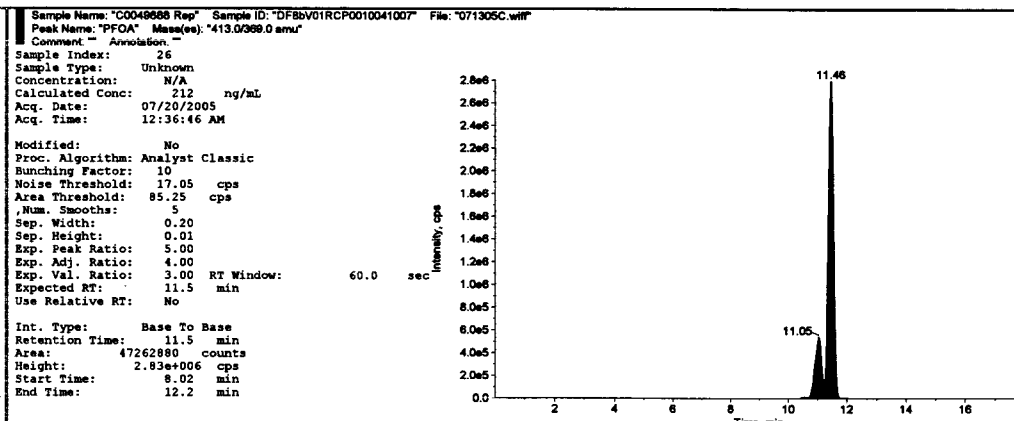
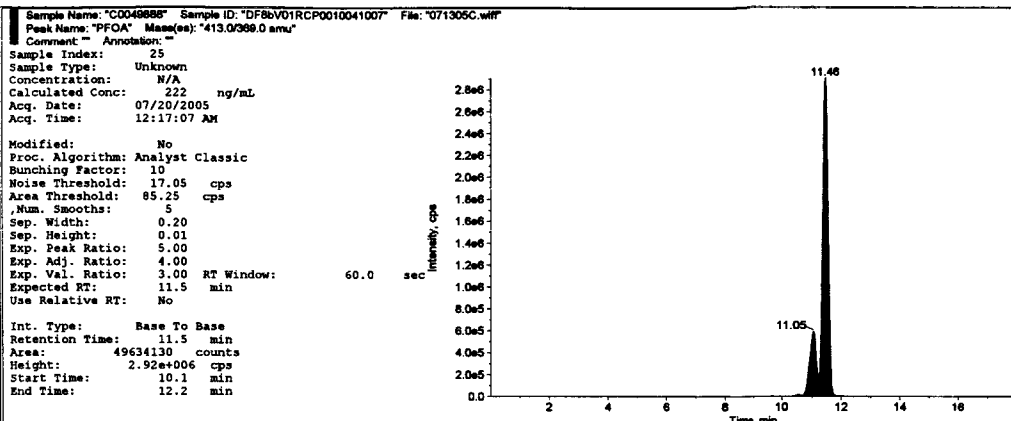


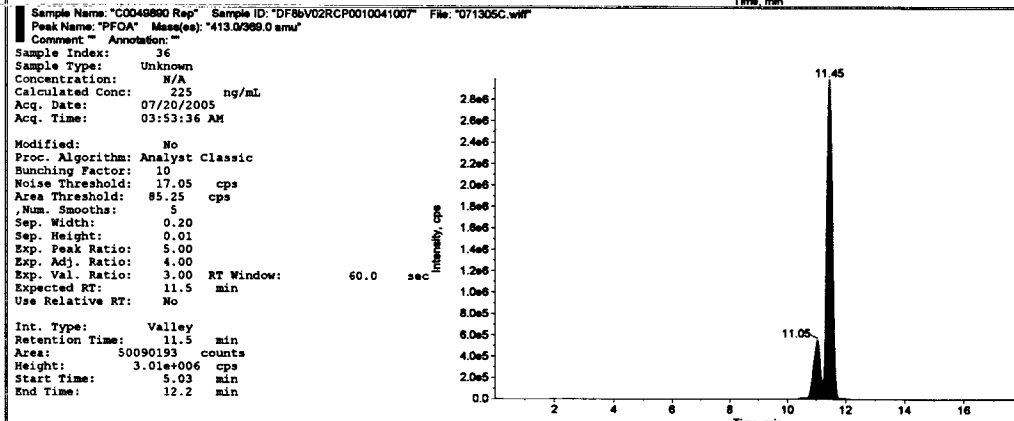
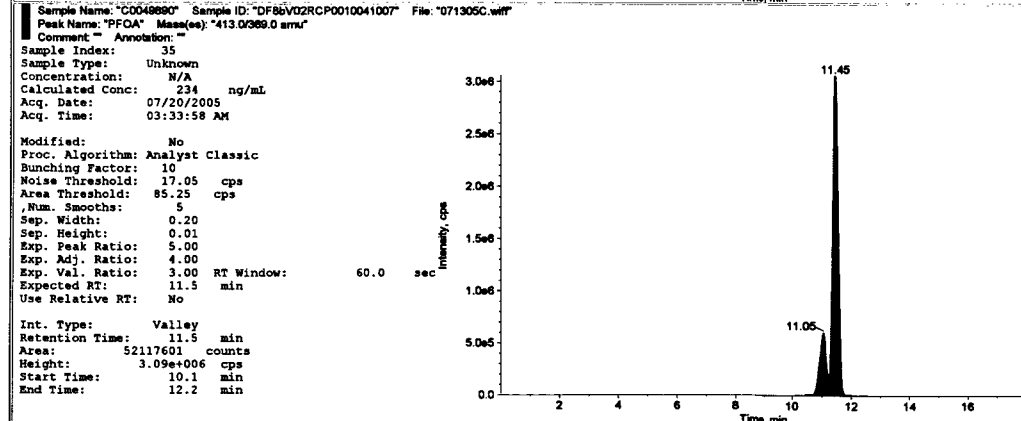
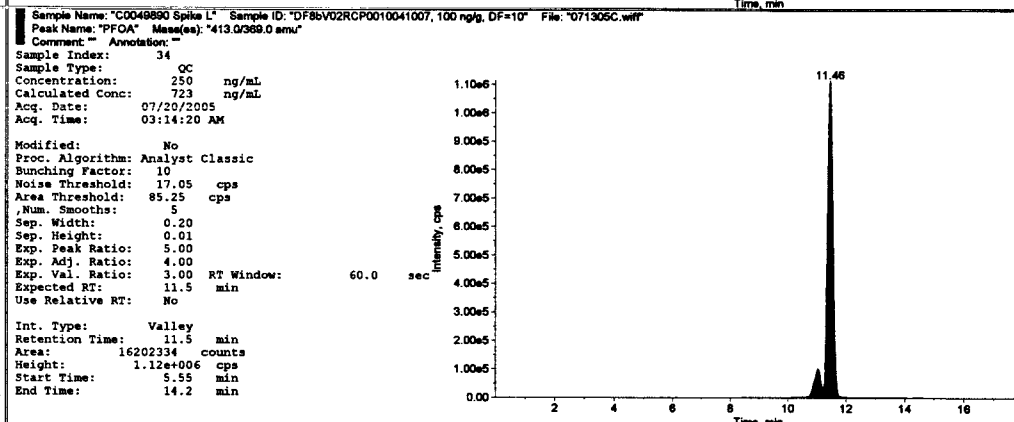
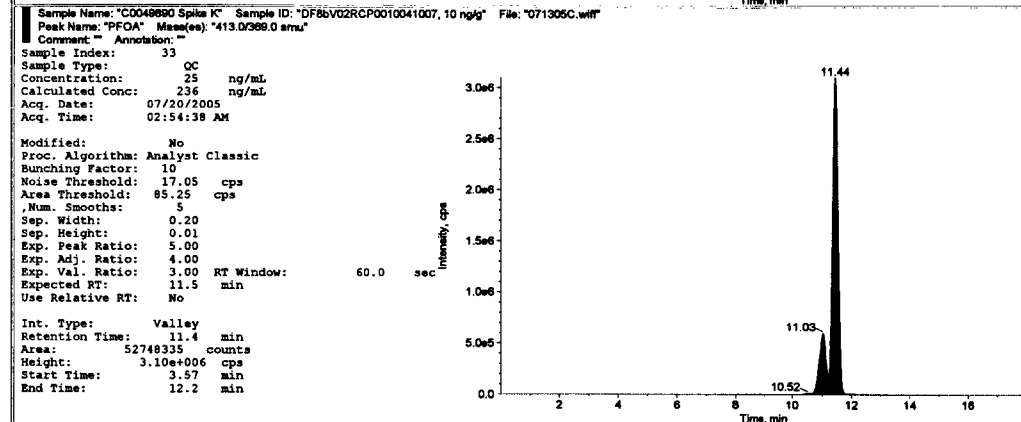
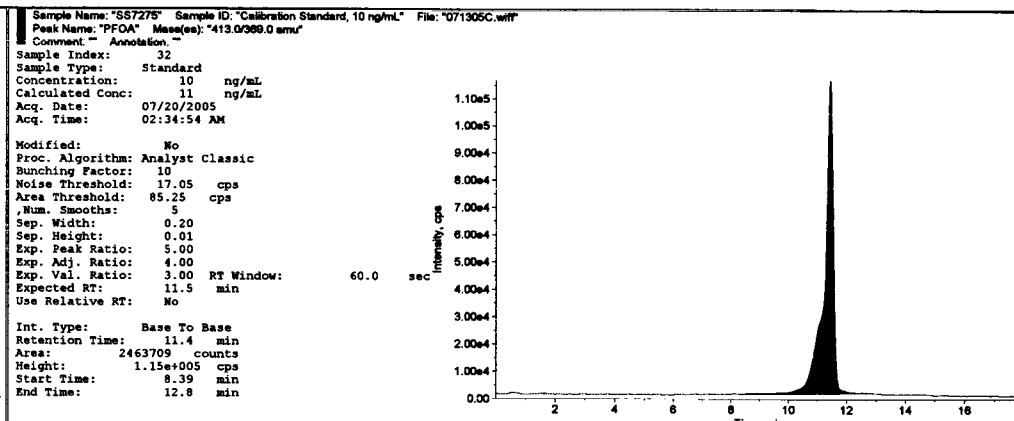
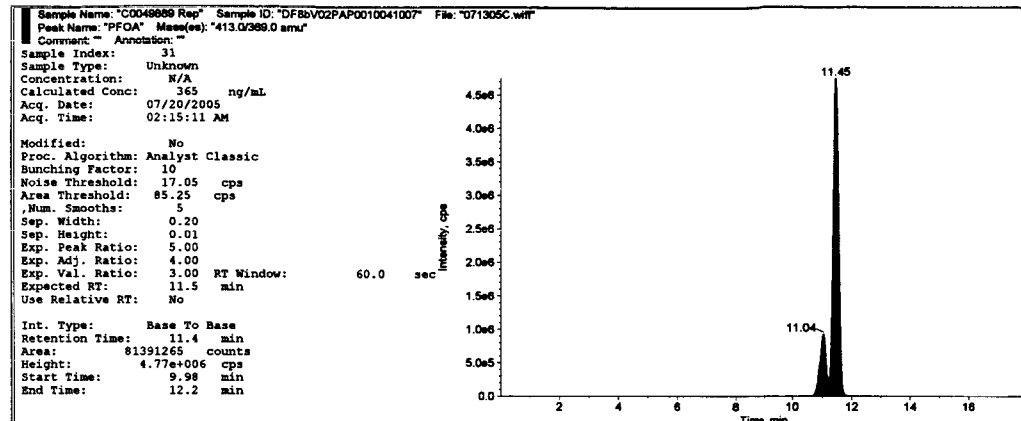
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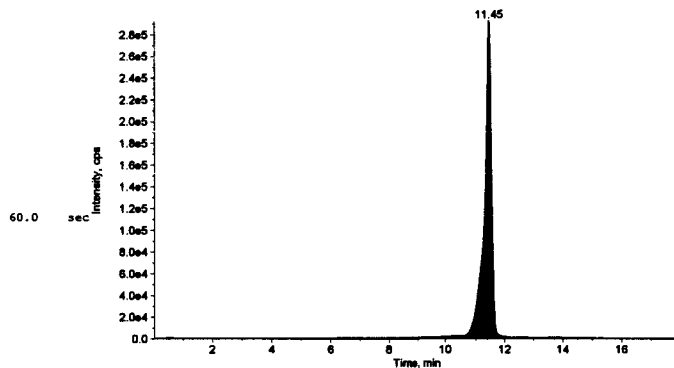
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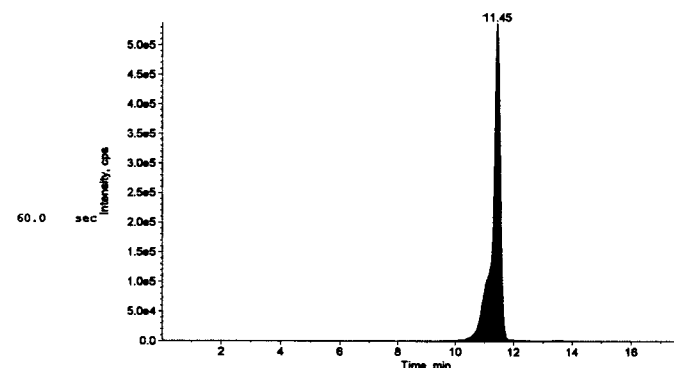


0421

Sample Name: "SS7274" Sample ID: "Calibration Standard, 25 ng/mL" File: "071305C.wiff"
Peak Name: "PFOA" Mass(es): "413.0/389.0 amu"
Comment: "Annotation: "
Sample Index: 37
Sample Type: Standard
Concentration: 25 ng/mL
Calculated Conc: 24 ng/mL
Acq. Date: 07/20/2005
Acq. Time: 04:13:19 AM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
.Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 5527250 counts
Height: 2.93e+005 cps
Start Time: 6.19 min
End Time: 14.0 min



Sample Name: "SS7273" Sample ID: "Calibration Standard, 50 ng/mL" File: "071305C.wiff"
Peak Name: "PFOA" Mass(es): "413.0/389.0 amu"
Comment: "Annotation: "
Sample Index: 38
Sample Type: Standard
Concentration: 50 ng/mL
Calculated Conc: 47 ng/mL
Acq. Date: 07/20/2005
Acq. Time: 04:32:57 AM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 17.05 cps
Area Threshold: 85.25 cps
.Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.4 min
Area: 10584455 counts
Height: 5.40e+005 cps
Start Time: 6.44 min
End Time: 13.7 min



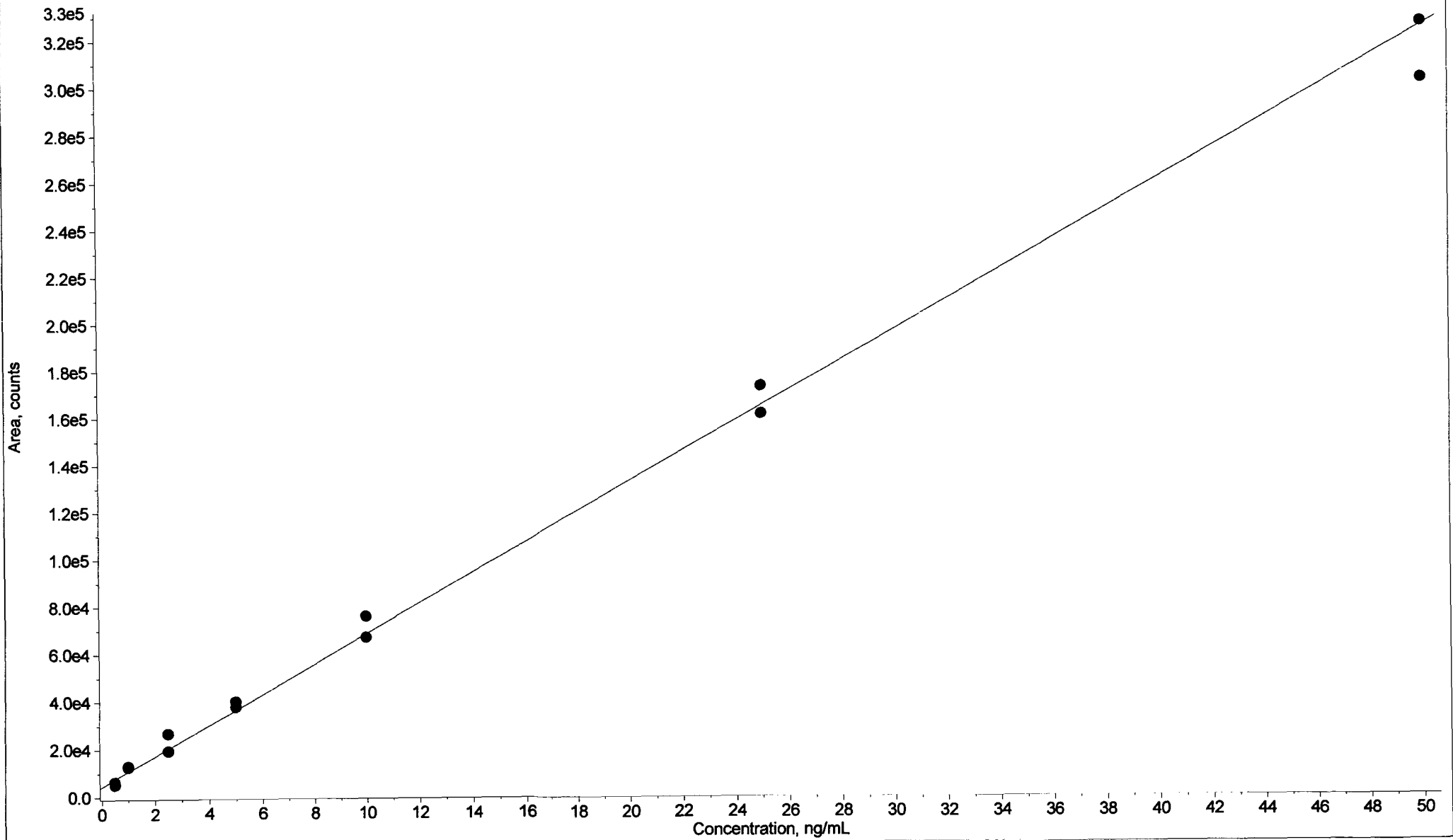
BAK 08/15/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	6352	0.500	0.300	071305C-301
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	12624	1.00	1.28	071305C-302
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	19373	2.50	2.33	071305C-303
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	40132	5.00	5.57	071305C-304
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	75879	10.0	11.2	071305C-305
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	172774	25.0	26.3	071305C-306
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	326301	50.0	50.2	071305C-307
Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	071305C-308
Reagent Control	C0054391 Control	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	071305C-309
Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1	PFOA Confirm Ion	46671	6.25	6.59	071305C-310
Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1	PFOA Confirm Ion	170414	25.0	25.9	071305C-311
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5222	0.500	0.124	071305C-312
C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	6913423	25.0	1080.	071305C-313
C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	1226161	25.0	1910.	071305C-314
C0049886	DF09V02PAP0010041007	Unknown	1	PFOA Confirm Ion	6737133	N/A	1050.	071305C-315
C0049886 Rep	DF09V02PAP0010041007	Unknown	1	PFOA Confirm Ion	6525592	N/A	1020.	071305C-316
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	12985	1.00	1.34	071305C-317
C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	10067174	25.0	1570.	071305C-318
C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	2051419	25.0	3190.	071305C-319
C0049887	DF8bV01PAP0010041007	Unknown	1	PFOA Confirm Ion	9737628	N/A	1520.	071305C-320
C0049887 Rep	DF8bV01PAP0010041007	Unknown	1	PFOA Confirm Ion	9406124	N/A	1470.	071305C-321
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	26726	2.50	3.48	071305C-322
C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	4362183	25.0	680.	071305C-323
C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	717773	25.0	1110.	071305C-324
C0049888	DF8bV01RCP0010041007	Unknown	1	PFOA Confirm Ion	4449633	N/A	694.	071305C-325
C0049888 Rep	DF8bV01RCP0010041007	Unknown	1	PFOA Confirm Ion	4227080	N/A	659.	071305C-326
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	37928	5.00	5.23	071305C-327
C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	7951599	25.0	1240.	071305C-328
C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	1623128	25.0	2530.	071305C-329
C0049889	DF8bV02PAP0010041007	Unknown	1	PFOA Confirm Ion	7874176	N/A	1230.	071305C-330
C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	PFOA Confirm Ion	7262598	N/A	1130.	071305C-331
SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	67020	10.0	9.77	071305C-332
C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	4476412	25.0	698.	071305C-333
C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFOA Confirm Ion	852860	25.0	1320.	071305C-334
C0049890	DF8bV02RCP0010041007	Unknown	1	PFOA Confirm Ion	4402801	N/A	686.	071305C-335
C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	PFOA Confirm Ion	4335966	N/A	676.	071305C-336
SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	160961	25.0	24.4	071305C-337
SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	302383	50.0	46.5	071305C-338

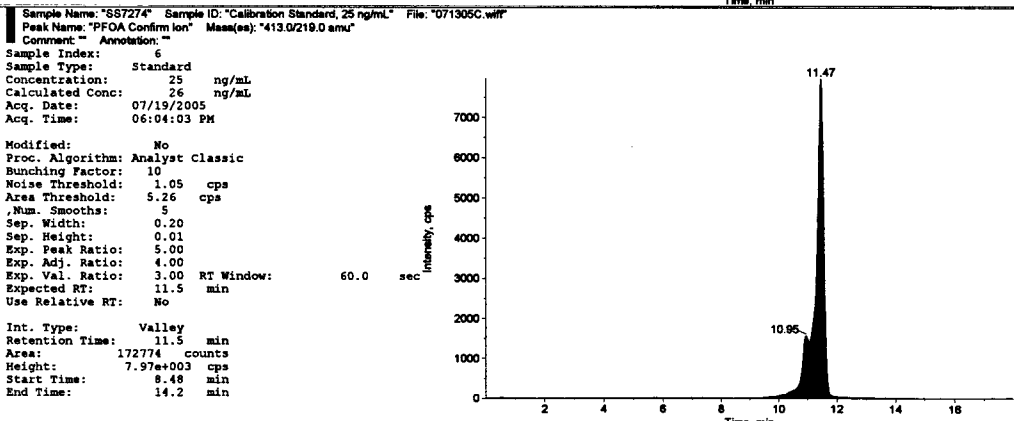
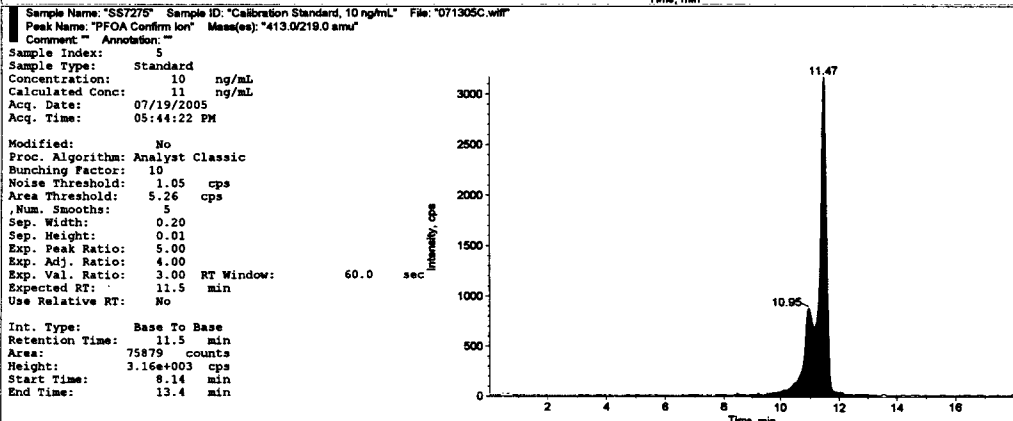
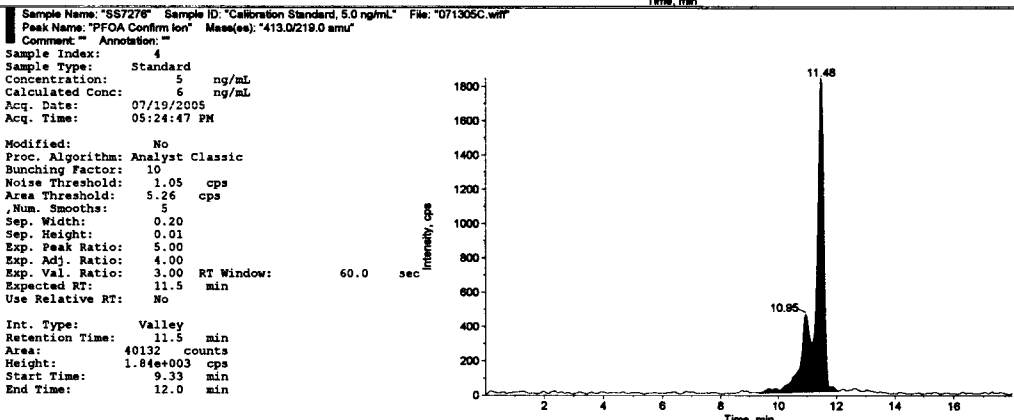
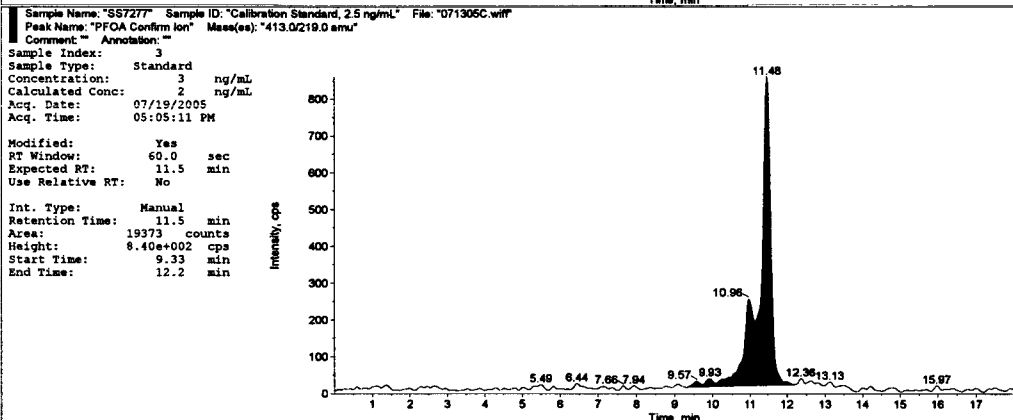
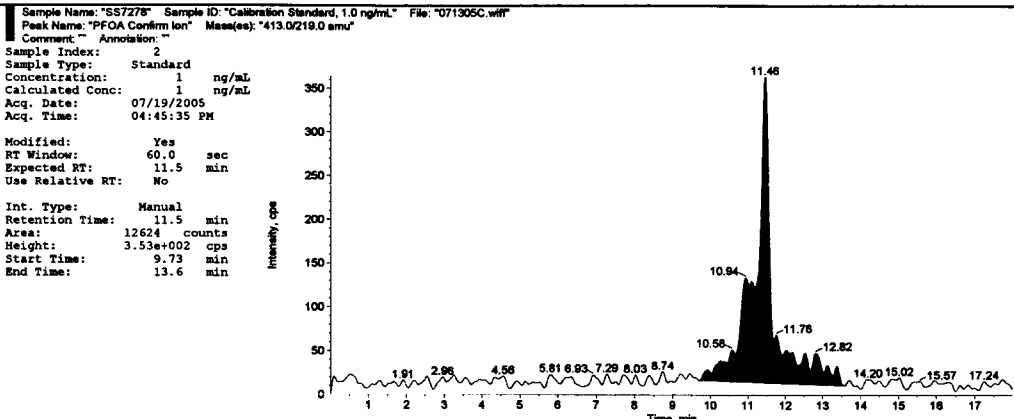
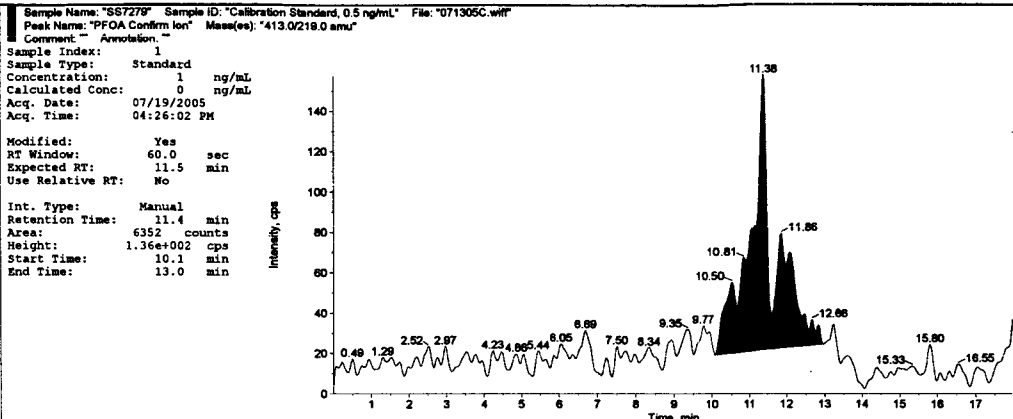
① Dilution required, see data set 071305CR. BAK 08/15/05

AMB 07/22/05

■ Untitled 3 (PFOA Confirm Ion): "Linear" Regression ("1/x" weighting): $y = 6.41e+003 x + 4.43e+003$ ($r = 0.9954$)

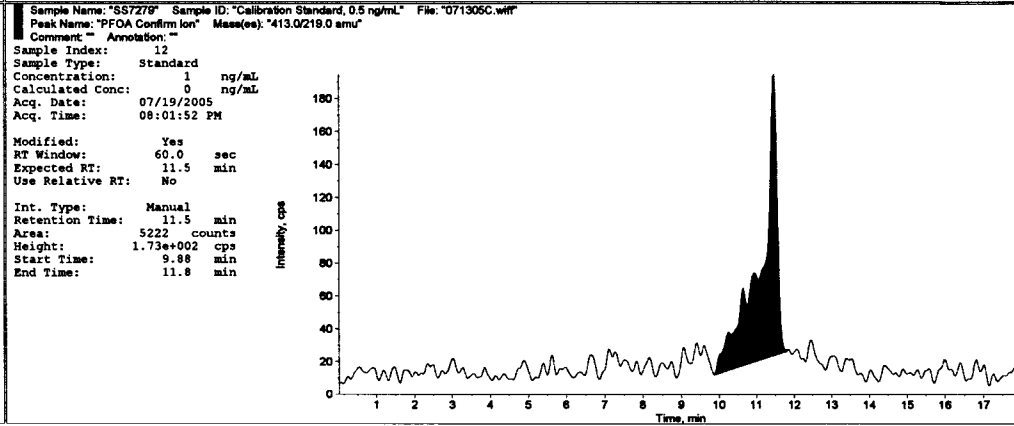
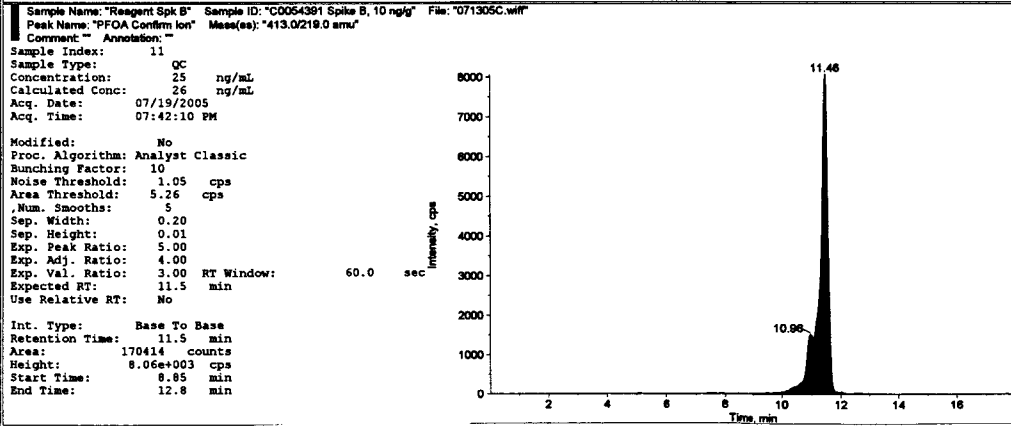
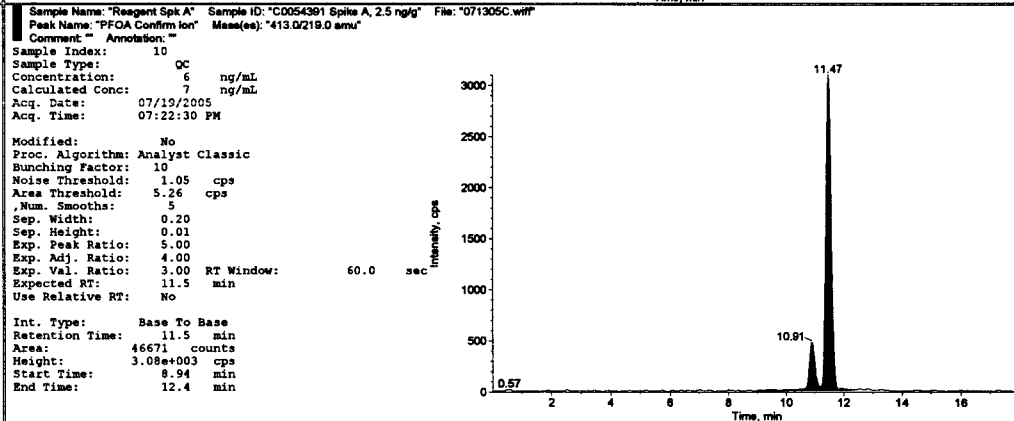
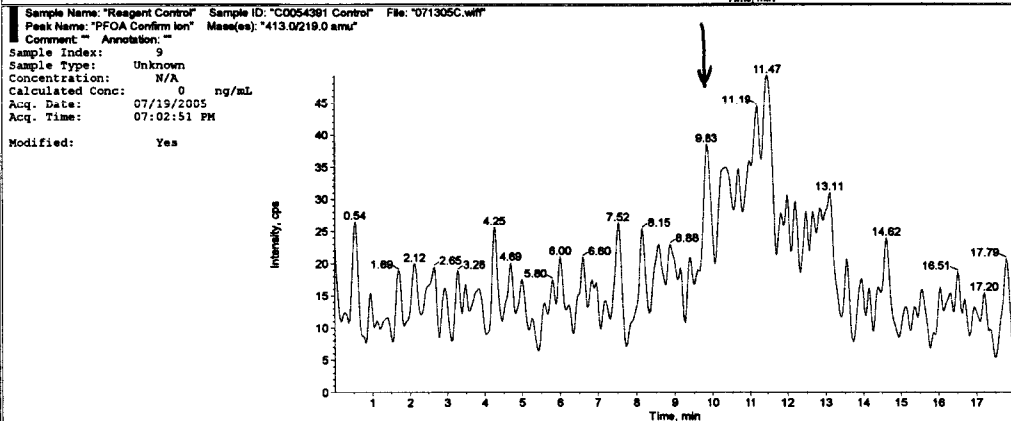
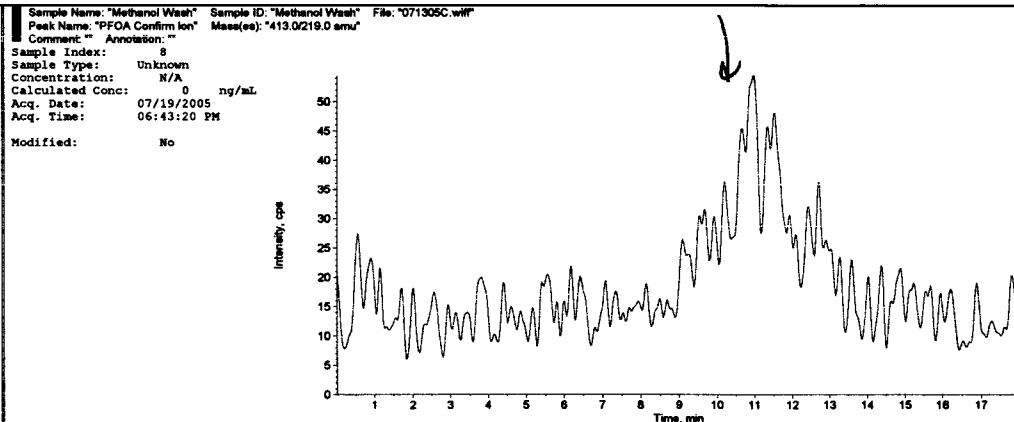
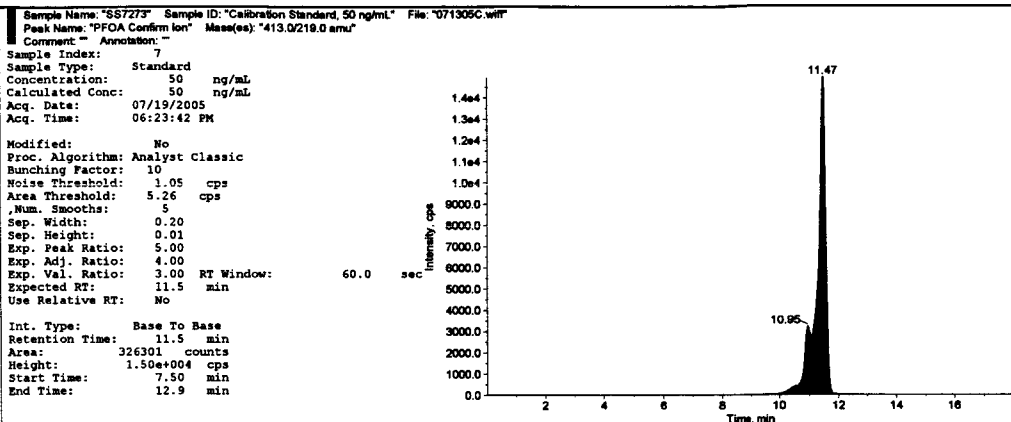


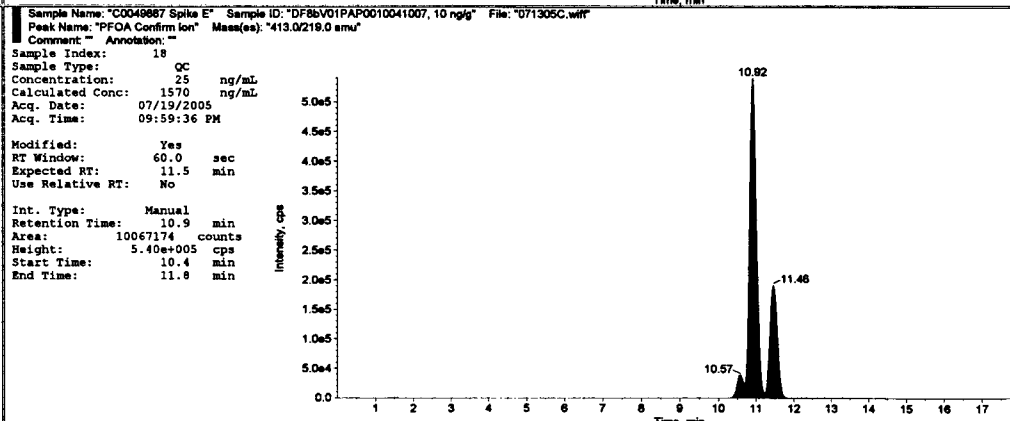
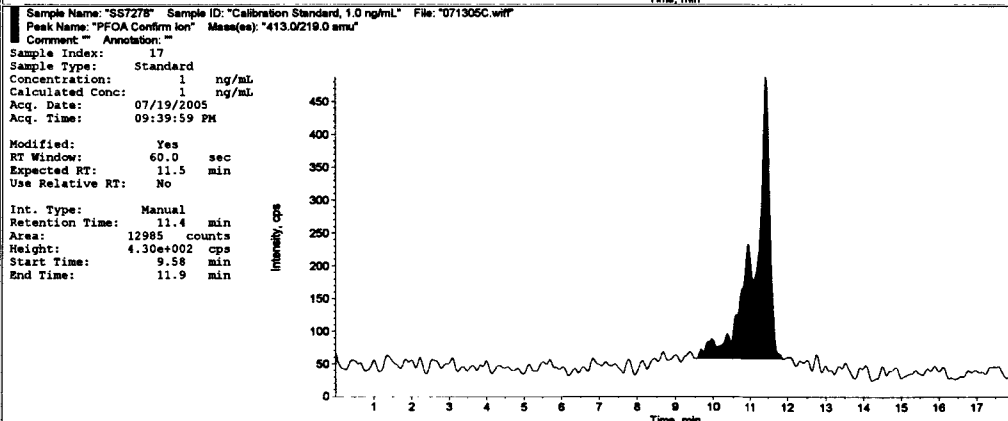
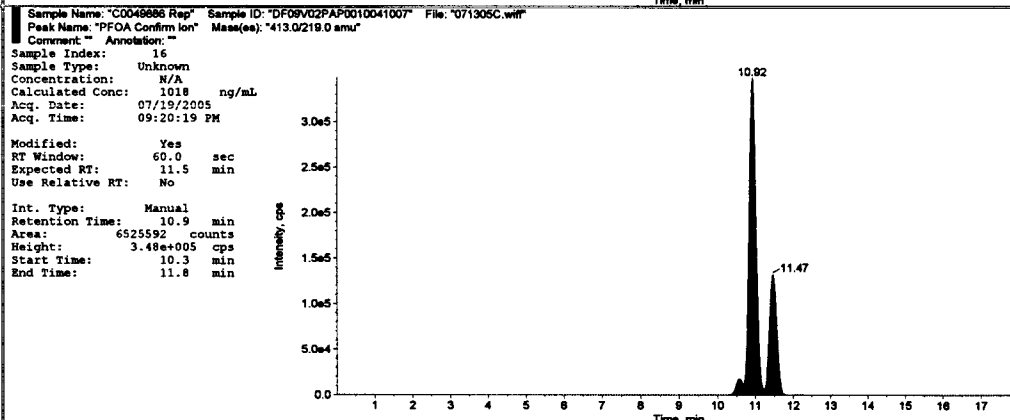
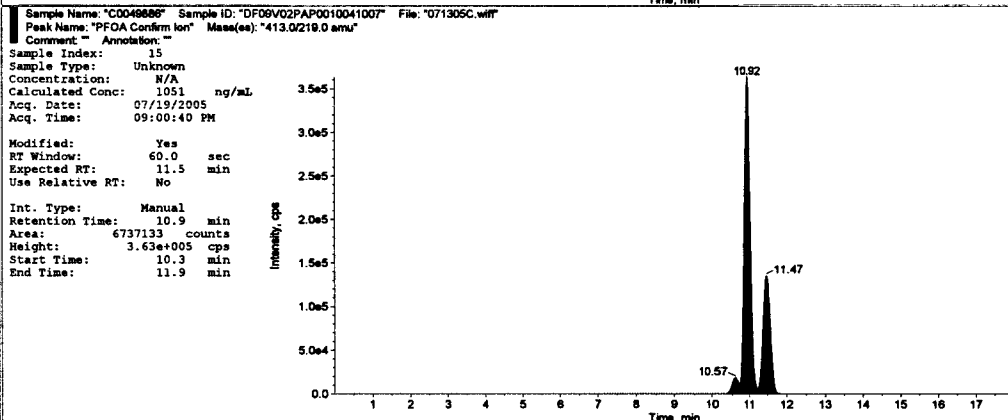
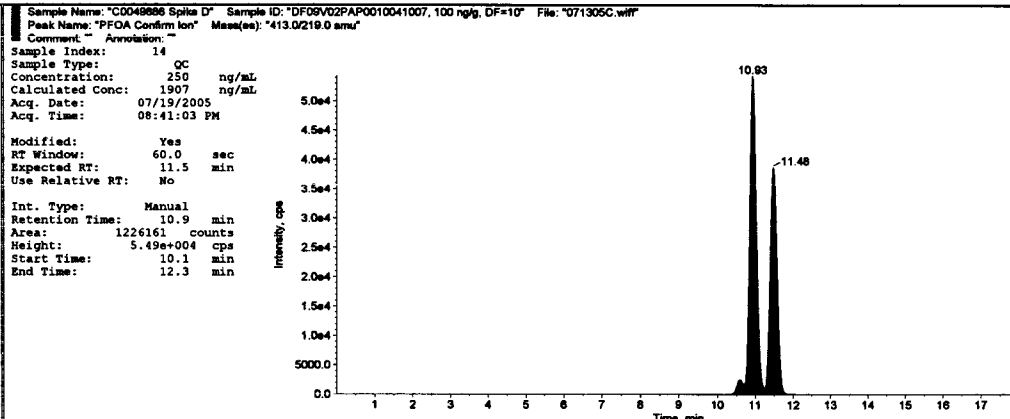
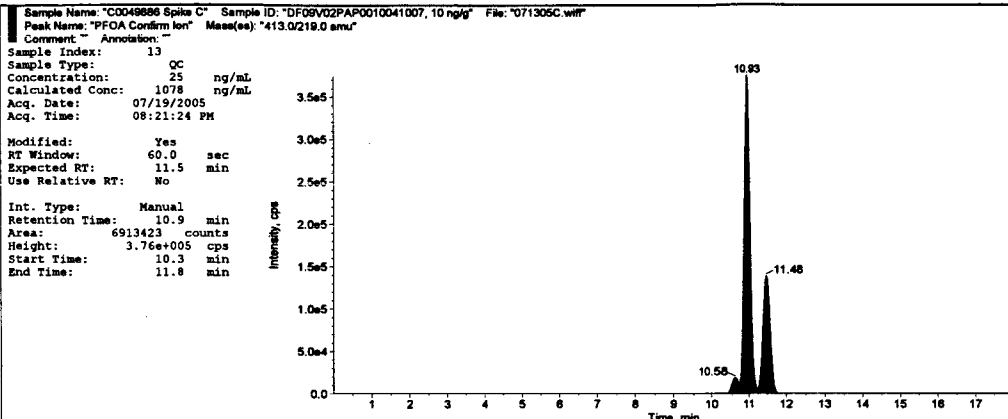
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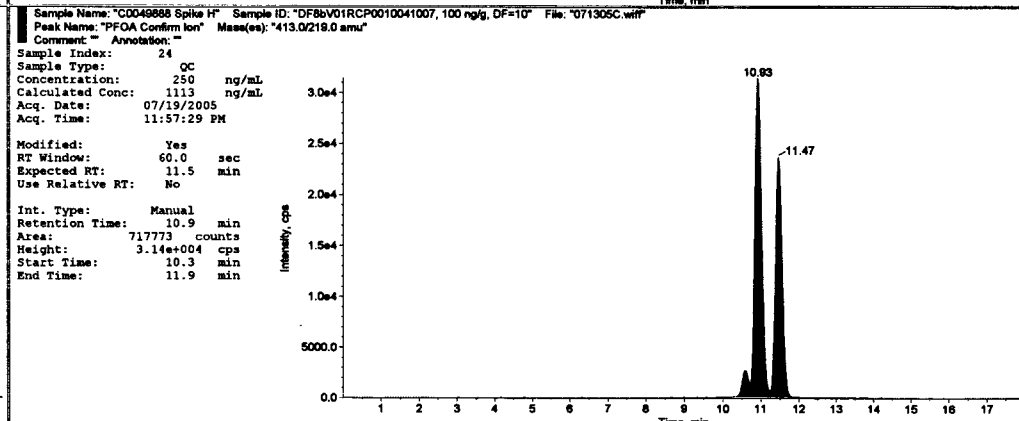
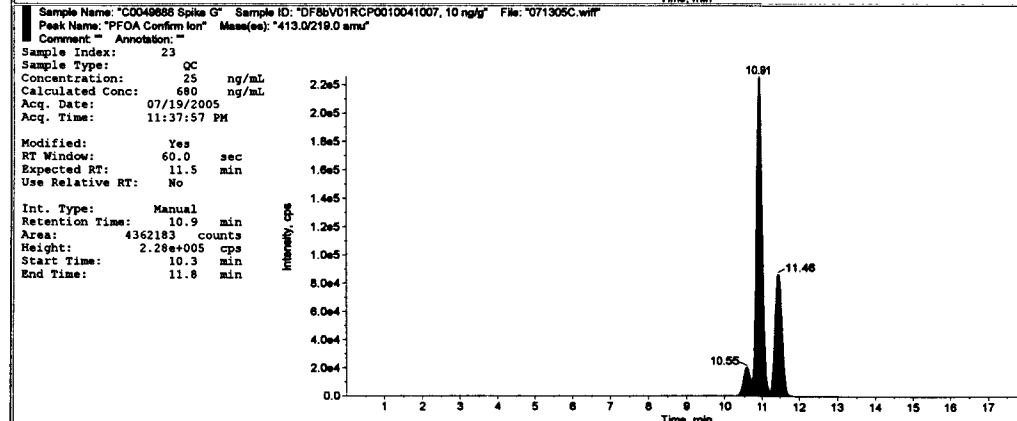
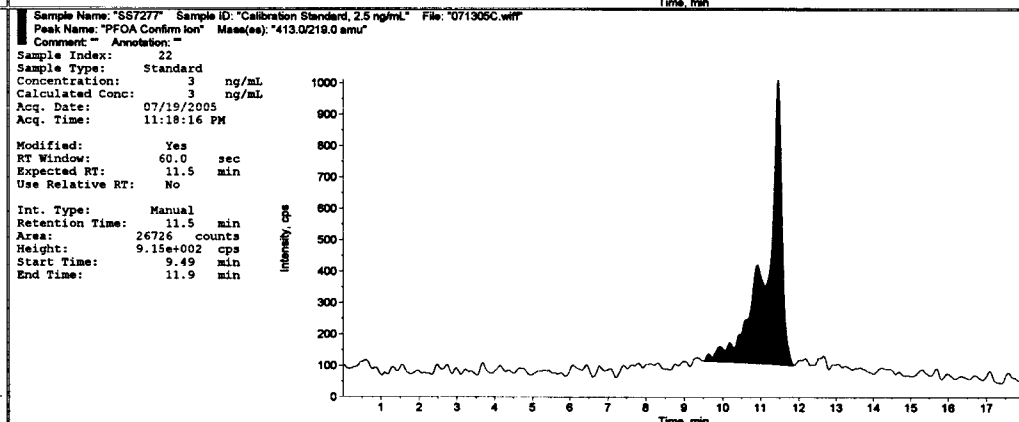
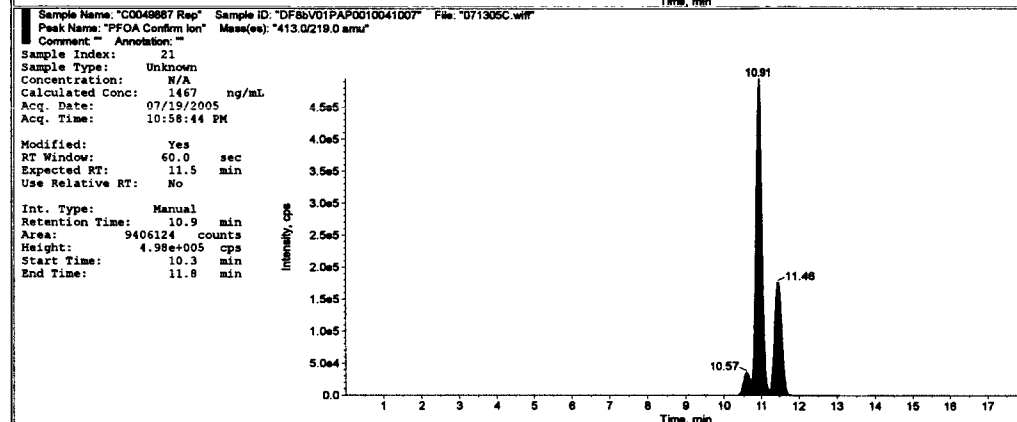
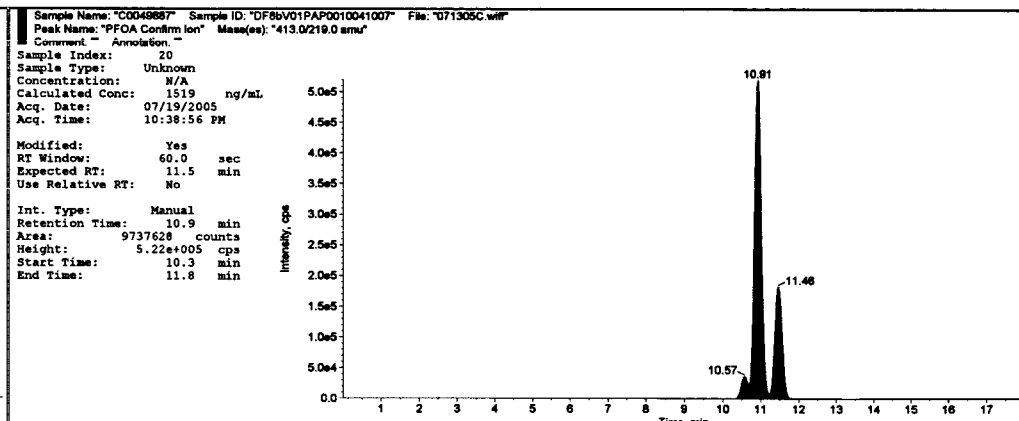
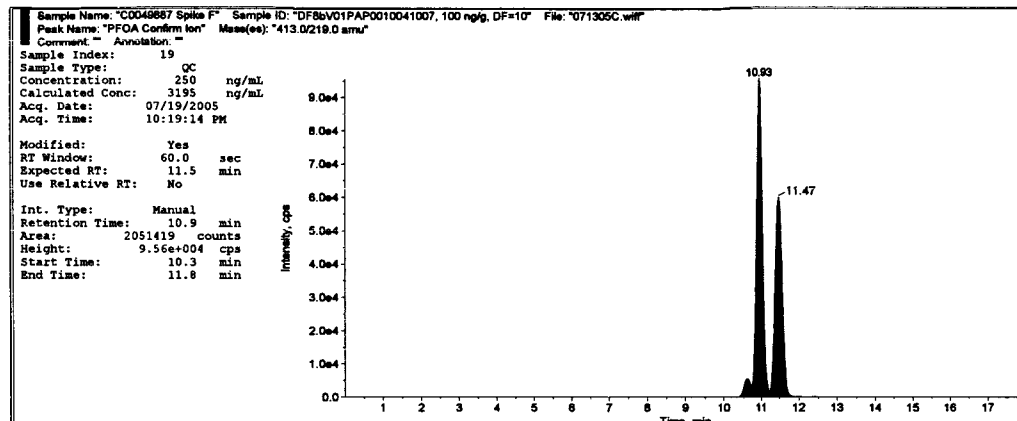
0425
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 Date 07/22/05
 Run# 1 To 38

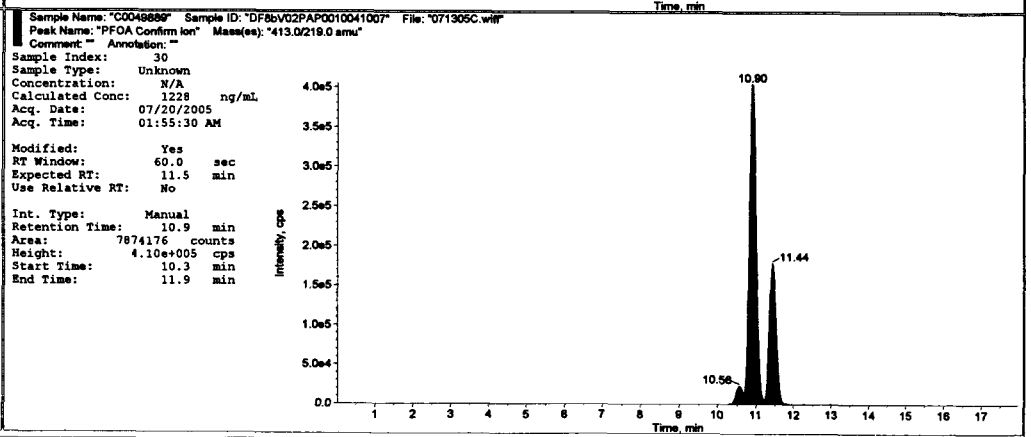
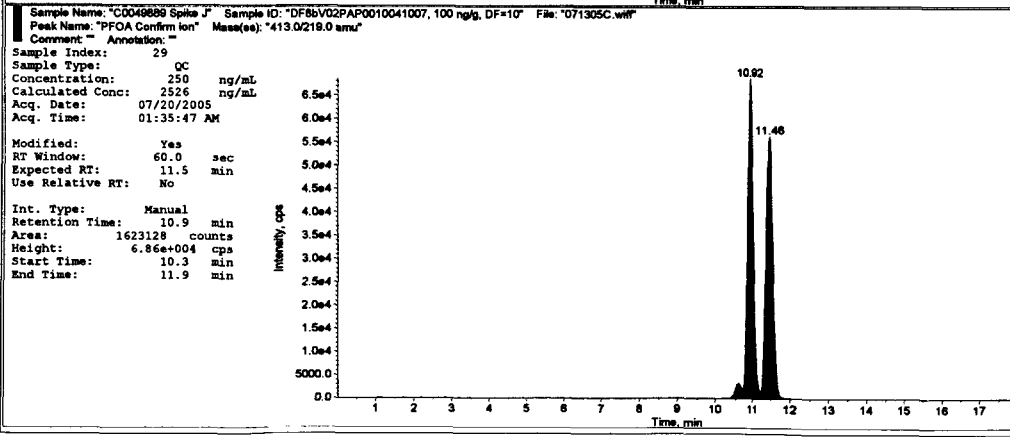
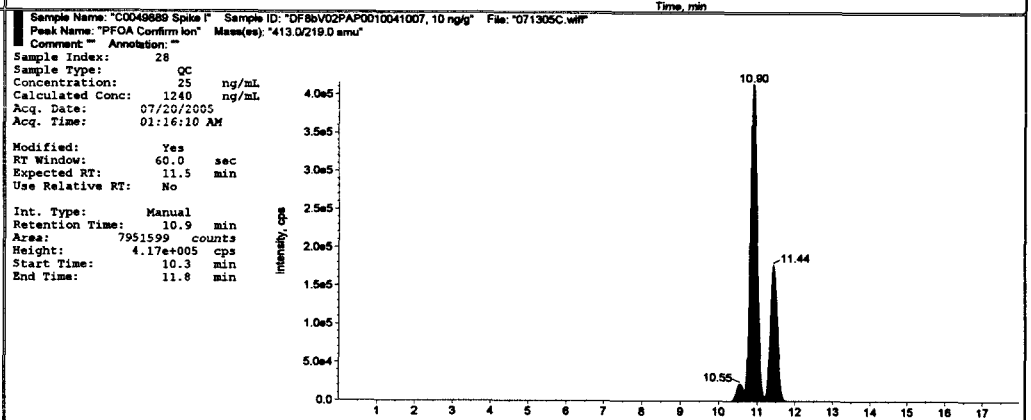
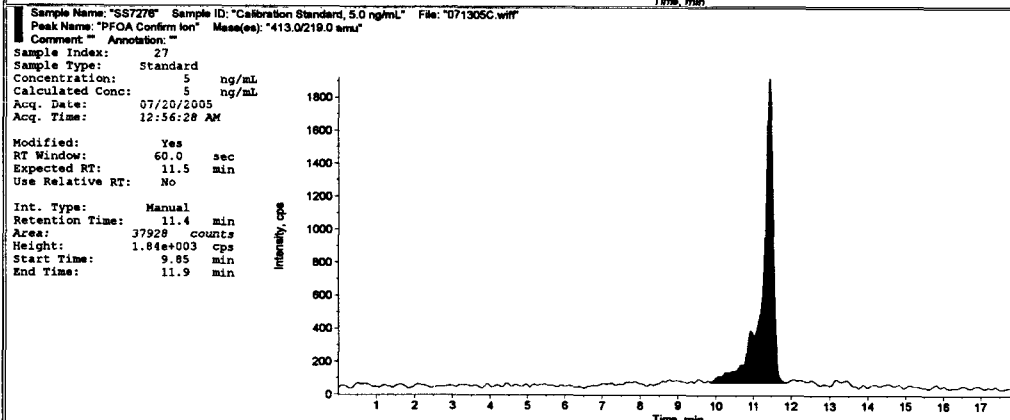
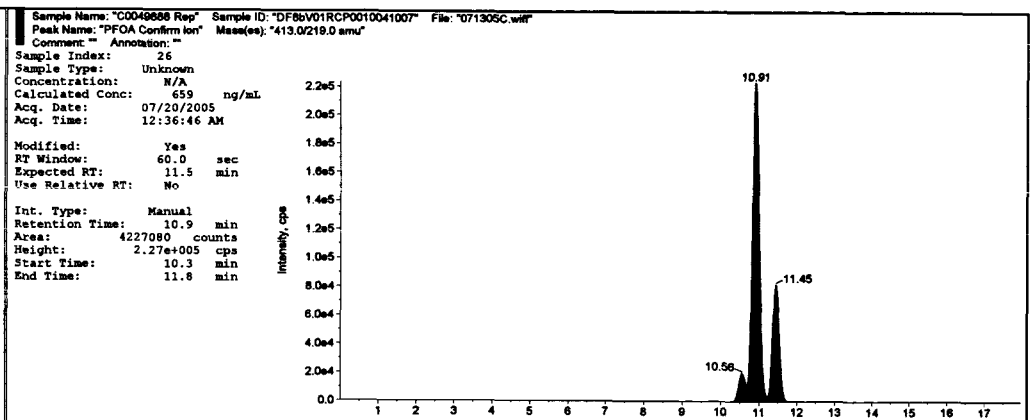
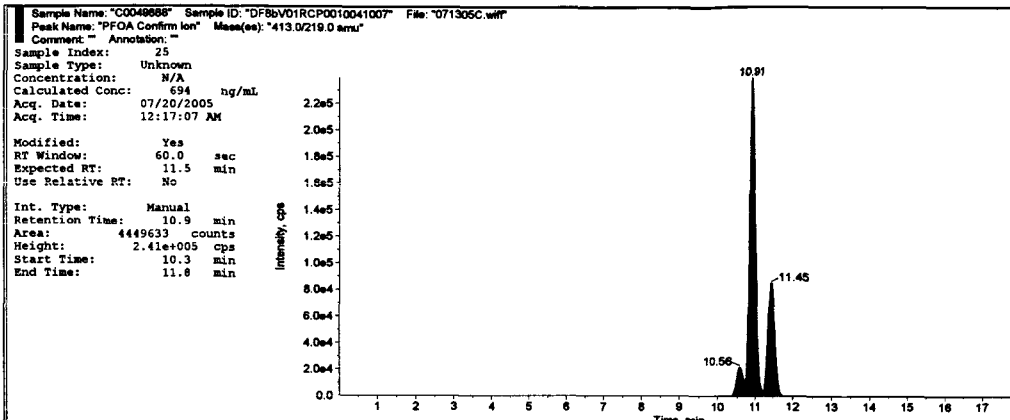
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 Sample Index: 1 To: 38



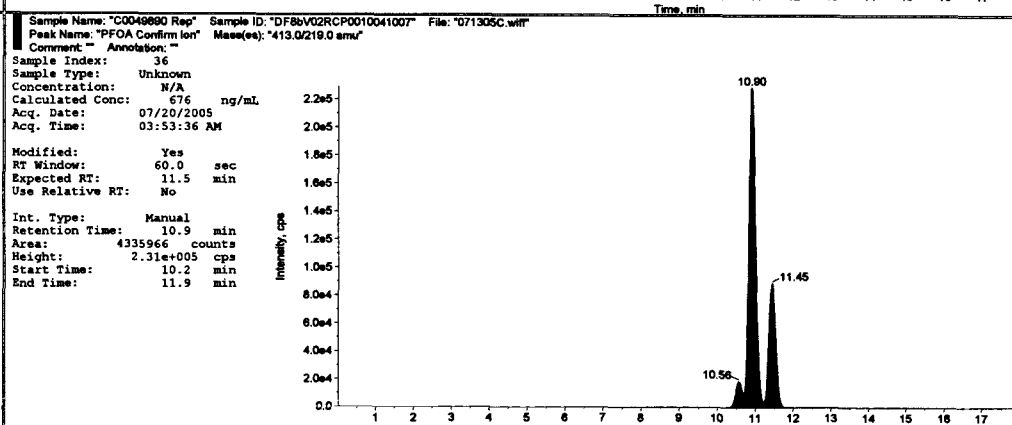
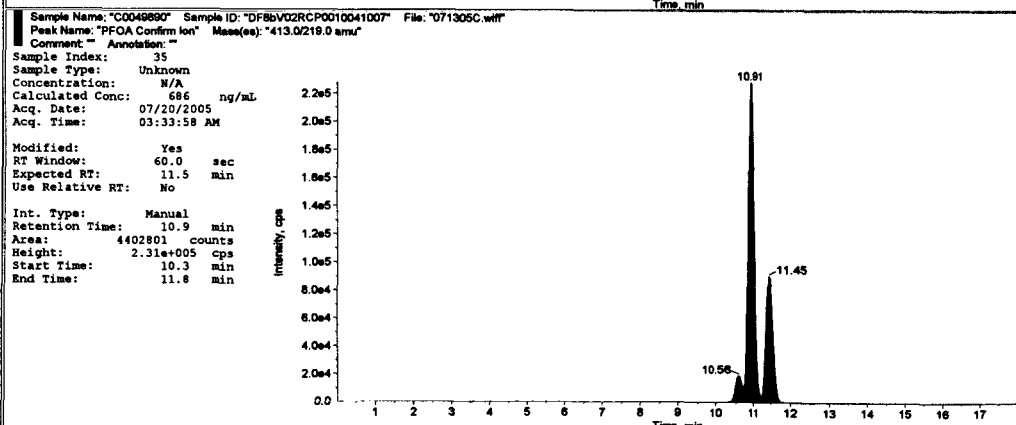
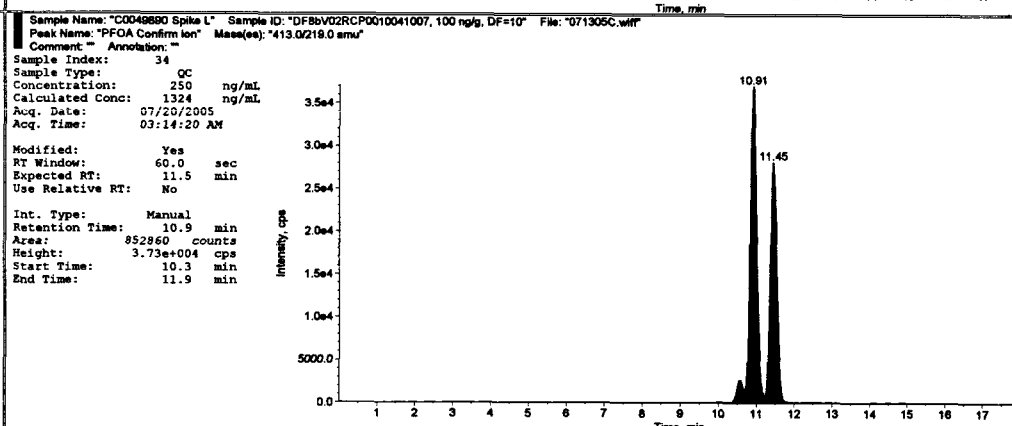
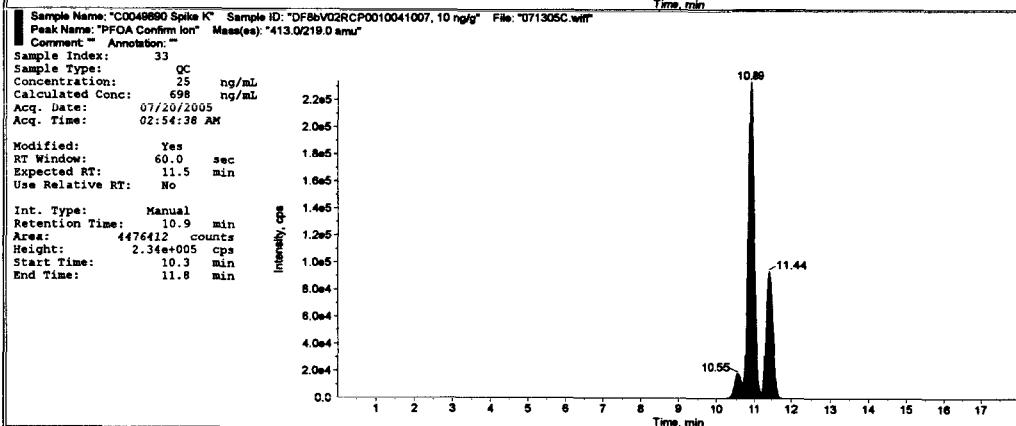
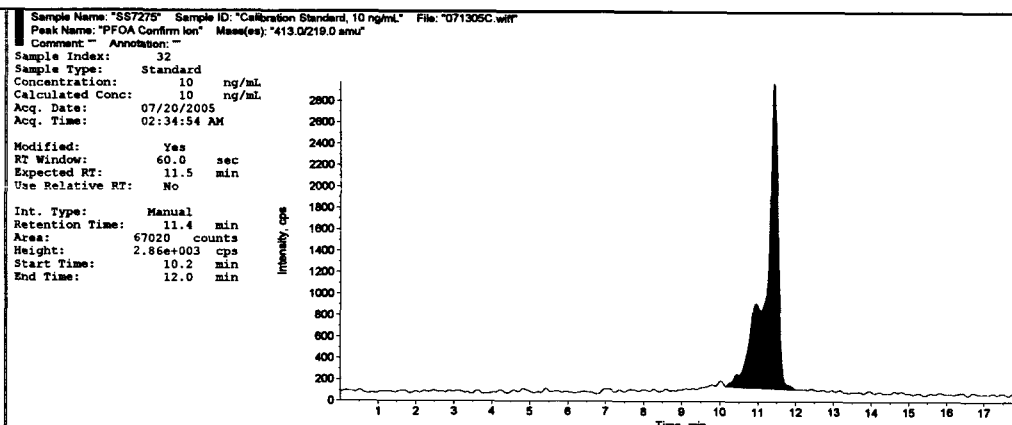
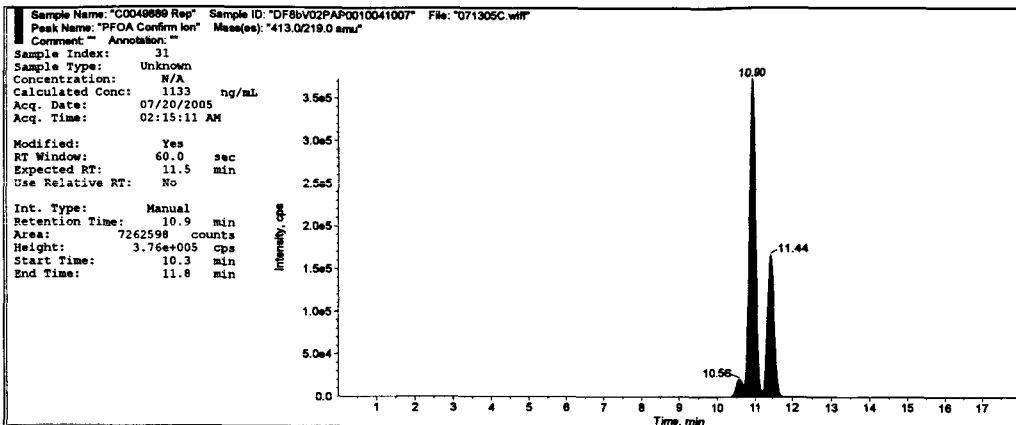


0427





0429

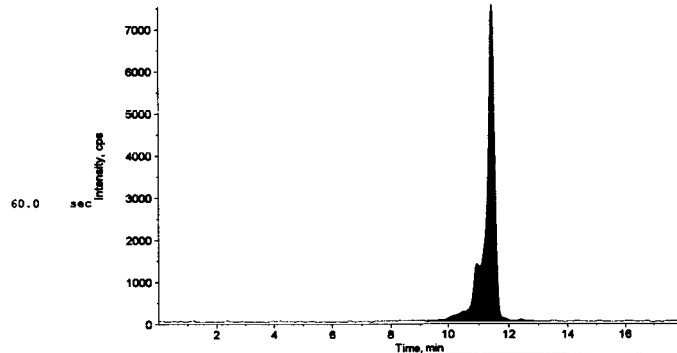


0430

Sample Name: "S67274" Sample ID: "Calibration Standard, 25 ng/mL" File: "071305C.wiff"
Peak Name: "PFOA Confirm Ion" Mass(es): "413.0219.0 amu"
Comment: " Annotation: "
Sample Index: 37
Sample Type: Standard
Concentration: 25 ng/mL
Calculated Conc: 24 ng/mL
Acq. Date: 07/20/2005
Acq. Time: 04:13:19 AM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 1.05 cps
Area Threshold: 5.26 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No

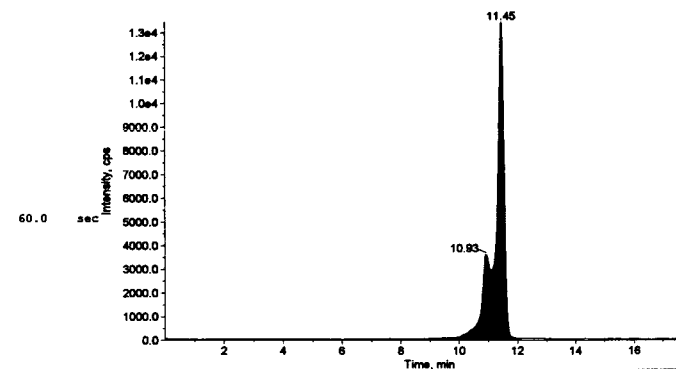
Int. Type: Valley
Retention Time: 11.4 min
Area: 160961 counts
Height: 7.50e+003 cps
Start Time: 9.21 min
End Time: 13.1 min



Sample Name: "S67273" Sample ID: "Calibration Standard, 50 ng/mL" File: "071305C.wiff"
Peak Name: "PFOA Confirm Ion" Mass(es): "413.0219.0 amu"
Comment: " Annotation: "
Sample Index: 38
Sample Type: Standard
Concentration: 50 ng/mL
Calculated Conc: 46 ng/mL
Acq. Date: 07/20/2005
Acq. Time: 04:32:57 AM

Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 1.05 cps
Area Threshold: 5.26 cps
Num. Smoother: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window:
Expected RT: 11.5 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 11.4 min
Area: 302383 counts
Height: 1.34e+004 cps
Start Time: 7.87 min
End Time: 12.1 min



BRL 10/15/05

Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ng/mL)	Calculated Concentration (ng/mL)	Run #
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	177700	0.500	0.428	071305C-301
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	282623	1.00	0.985	071305C-302
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	615685	2.50	2.75	071305C-303
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1243615	5.00	6.08	071305C-304
SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2212688	10.0	11.2	071305C-305
SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	5103737	25.0	26.6	071305C-306
SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9692598	50.0	50.9	071305C-307
Methanol Wash	Methanol Wash	Unknown	1	13C PFOA	83863	N/A	< 0	071305C-308
Reagent Control	C0054391 Control	Unknown	1	13C PFOA	81272	N/A	< 0	071305C-309
Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1	13C PFOA	1505693	6.25	7.47	071305C-310
Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1	13C PFOA	5232865	25.0	27.2	071305C-311
SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	206221	0.500	0.580	071305C-312
C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1641852	25.0	8.19	071305C-313
C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3166590	250.	163.	071305C-314
C0049886	DF09V02PAP0010041007	Unknown	1	13C PFOA	1396805	N/A	6.90	071305C-315
C0049886 Rep	DF09V02PAP0010041007	Unknown	1	13C PFOA	1324428	N/A	6.51	071305C-316
SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	228890	1.00	0.700	071305C-317
C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1003830	25.0	4.81	071305C-318
C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2365176	250.	120.	071305C-319
C0049887	DF8bV01PAP0010041007	Unknown	1	13C PFOA	882809	N/A	4.17	071305C-320
C0049887 Rep	DF8bV01PAP0010041007	Unknown	1	13C PFOA	861894	N/A	4.06	071305C-321
SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	524237	2.50	2.27	071305C-322
C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1967525	25.0	9.92	071305C-323
C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3472130	250.	179.	071305C-324
C0049888	DF8bV01RCP0010041007	Unknown	1	13C PFOA	1766290	N/A	8.86	071305C-325
C0049888 Rep	DF8bV01RCP0010041007	Unknown	1	13C PFOA	1786476	N/A	8.96	071305C-326
SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1084785	5.00	5.24	071305C-327
C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	925018	25.0	4.39	071305C-328
C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2309827	250.	117.	071305C-329
C0049889	DF8bV02PAP0010041007	Unknown	1	13C PFOA	795105	N/A	3.70	071305C-330
C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	13C PFOA	770235	N/A	3.57	071305C-331
SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	1855254	10.0	9.33	071305C-332
C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1906062	25.0	9.60	071305C-333
C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3555452	250.	183.	071305C-334
C0049890	DF8bV02RCP0010041007	Unknown	1	13C PFOA	1781528	N/A	8.94	071305C-335
C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	13C PFOA	1538615	N/A	7.65	071305C-336
SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	4546418	25.0	23.6	071305C-337
SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9024710	50.0	47.4	071305C-338

Vegetation Conversion

Compound: 13C PFOA

Exygen Study No: P760

Exygen ID	Sponsor ID	Analyte Found (ng/mL)	Analyte Found (ppb)
C0049886	DF09-V02-PAP001-0-041007	6.90	2.76
C0049886 Rep	DF09-V02-PAP001-0-041007	6.51	2.60
C0049886 Spk C	DF09-V02-PAP001-0-041007	8.19	3.28
C0049886 Spk D	DF09-V02-PAP001-0-041007	163	65.2
C0049887	DF8b-V01-PAP001-041007	4.17	1.67
C0049887 Rep	DF8b-V01-PAP001-041007	4.06	1.62
C0049887 Spk E	DF8b-V01-PAP001-041007	4.81	1.92
C0049887 Spk F	DF8b-V01-PAP001-041007	120	48.0
C0049888	DF8b-V01-RCP001-041007	8.86	3.54
C0049888 Rep	DF8b-V01-RCP001-041007	8.96	3.58
C0049888 Spk G	DF8b-V01-RCP001-041007	9.92	3.97
C0049888 Spk H	DF8b-V01-RCP001-041007	179	71.6
C0049889	DF8b-V02-PAP001-041007	3.70	1.48
C0049889 Rep	DF8b-V02-PAP001-041007	3.57	1.43
C0049889 Spk I	DF8b-V02-PAP001-041007	4.39	1.76
C0049889 Spk J	DF8b-V02-PAP001-041007	117	46.8
C0049890	DF8b-V02-RCP001-041007	8.94	3.58
C0049890 Rep	DF8b-V02-RCP001-041007	7.65	3.06
C0049890 Spk K	DF8b-V02-RCP001-041007	9.60	3.84
C0049890 Spk L	DF8b-V02-RCP001-041007	183	73.2

Analyte Found (ppb) = [analyte found (ng/mL) x final volume (2 mL)] / sample weight (5 g)

ND = Not detected = ~~Peak Area~~ *Ar or ABOVE 0.2ng/g* ①

NQ = Not quantifiable = Measured concentration ^{below} Limit of Quantitation (LOQ) which is 0.5 ng/g.

^
BETWEEN 0.2ng/g AND ①

4①

① RE MLC 9/20/05

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BK 08/04/05

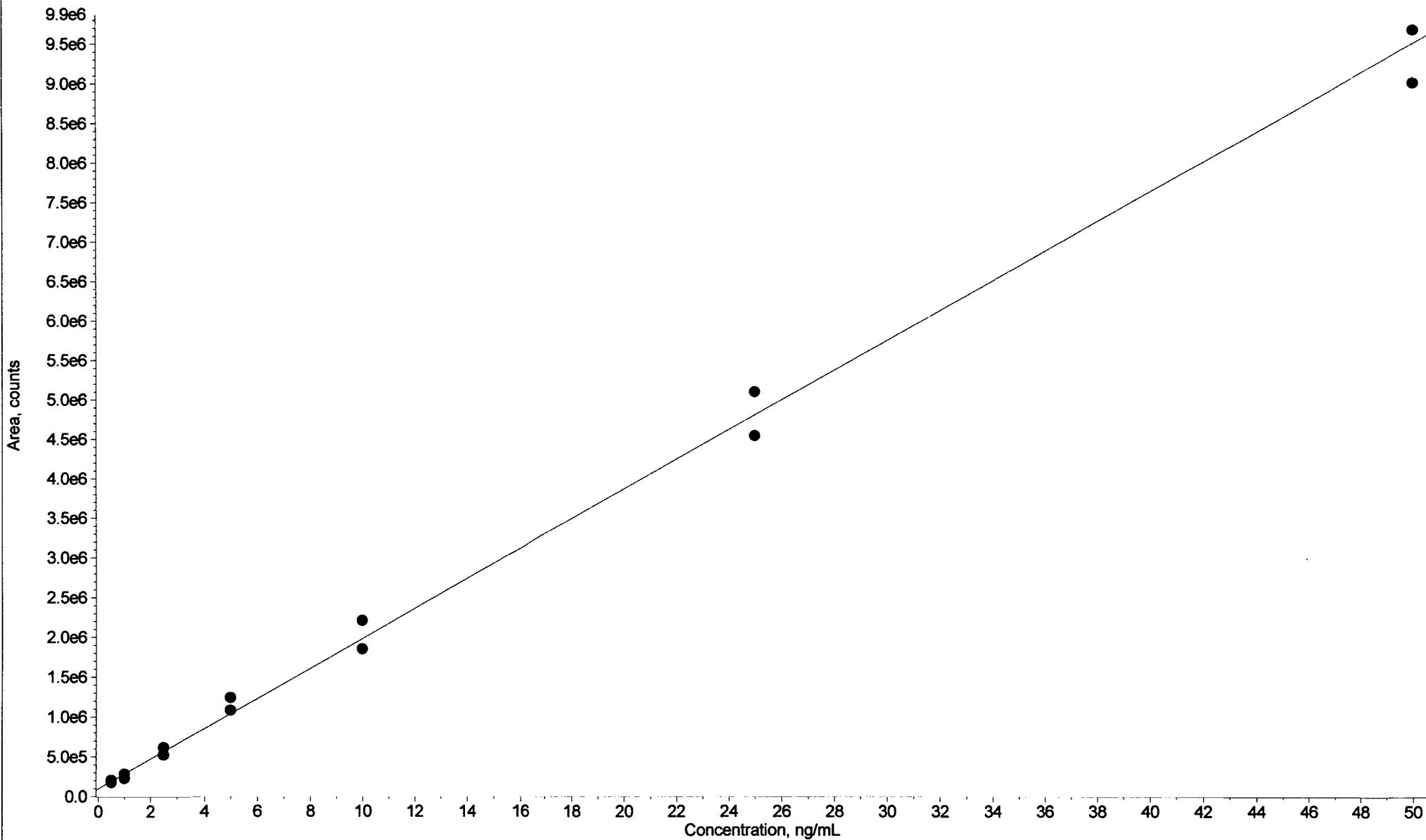
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1	Vegetation Conversion (FORMULAS)			
2				
3	Compound:	13C PFOA	Exygen Study No:	P760
4				
5			Analyte	Analyte
6	Exygen	Sponsor	Found	Found
7	ID	ID	(ng/mL)	(ppb)
8				
9	C0049886	DF09-V02-PAP001-0-041007	=Raw Data!H142	=(C9*2)/5
10	C0049886 Rep	DF09-V02-PAP001-0-041007	=Raw Data!H143	=(C10*2)/5
11	C0049886 Spk C	DF09-V02-PAP001-0-041007	=Raw Data!H140	=(C11*2)/5
12	C0049886 Spk D	DF09-V02-PAP001-0-041007	=Raw Data!H141	=(C12*2)/5
13				
14	C0049887	DF8b-V01-PAP001-041007	=Raw Data!H147	=(C14*2)/5
15	C0049887 Rep	DF8b-V01-PAP001-041007	=Raw Data!H148	=(C15*2)/5
16	C0049887 Spk E	DF8b-V01-PAP001-041007	=Raw Data!H145	=(C16*2)/5
17	C0049887 Spk F	DF8b-V01-PAP001-041007	=Raw Data!H146	=(C17*2)/5
18				
19	C0049888	DF8b-V01-RCP001-041007	=Raw Data!H152	=(C19*2)/5
20	C0049888 Rep	DF8b-V01-RCP001-041007	=Raw Data!H153	=(C20*2)/5
21	C0049888 Spk G	DF8b-V01-RCP001-041007	=Raw Data!H150	=(C21*2)/5
22	C0049888 Spk H	DF8b-V01-RCP001-041007	=Raw Data!H151	=(C22*2)/5
23				
24	C0049889	DF8b-V02-PAP001-041007	=Raw Data!H157	=(C24*2)/5
25	C0049889 Rep	DF8b-V02-PAP001-041007	=Raw Data!H158	=(C25*2)/5
26	C0049889 Spk I	DF8b-V02-PAP001-041007	=Raw Data!H155	=(C26*2)/5
27	C0049889 Spk J	DF8b-V02-PAP001-041007	=Raw Data!H156	=(C27*2)/5
28				
29	C0049890	DF8b-V02-RCP001-041007	=Raw Data!H162	=(C29*2)/5
30	C0049890 Rep	DF8b-V02-RCP001-041007	=Raw Data!H163	=(C30*2)/5
31	C0049890 Spk K	DF8b-V02-RCP001-041007	=Raw Data!H160	=(C31*2)/5
32	C0049890 Spk L	DF8b-V02-RCP001-041007	=Raw Data!H161	=(C32*2)/5
33				

0434

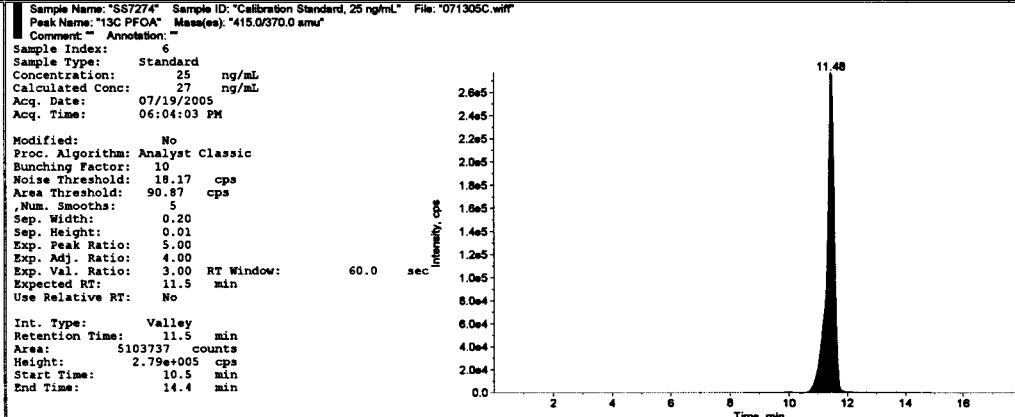
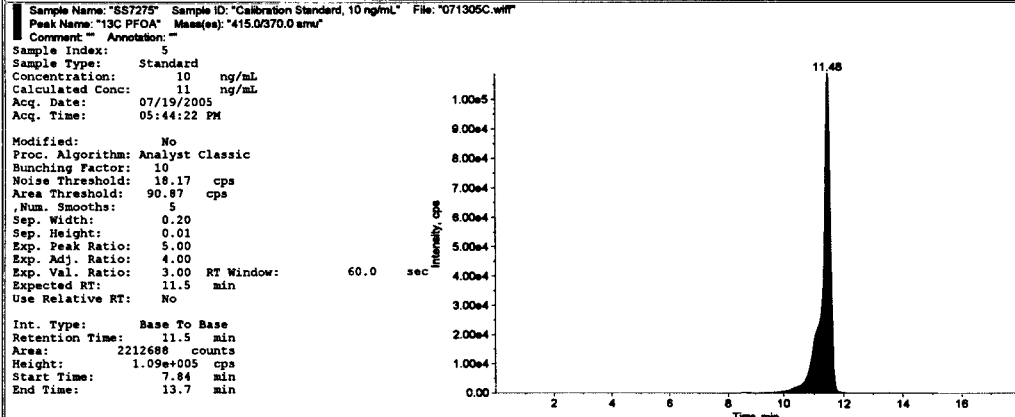
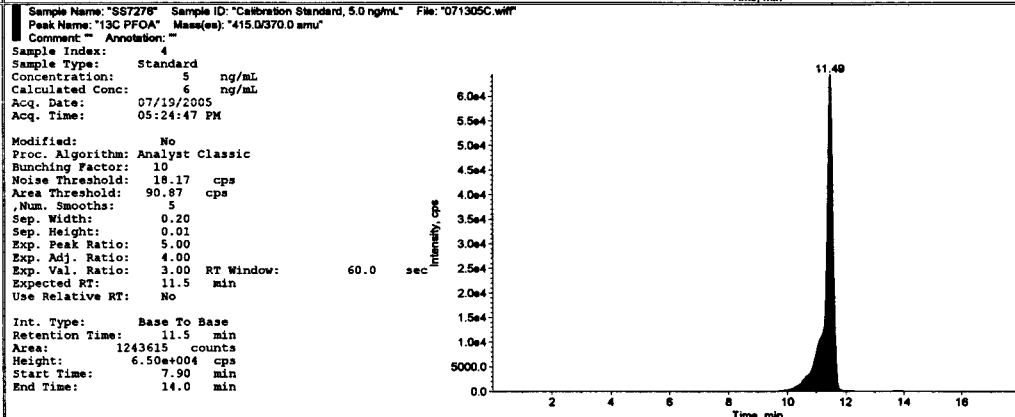
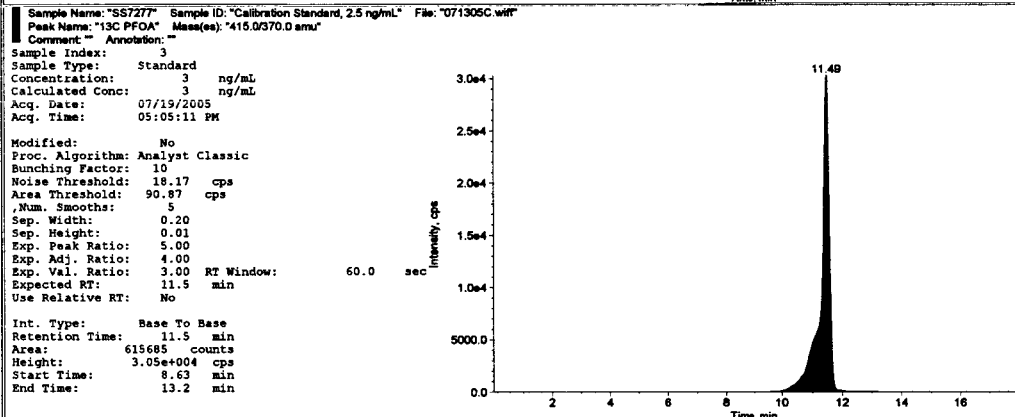
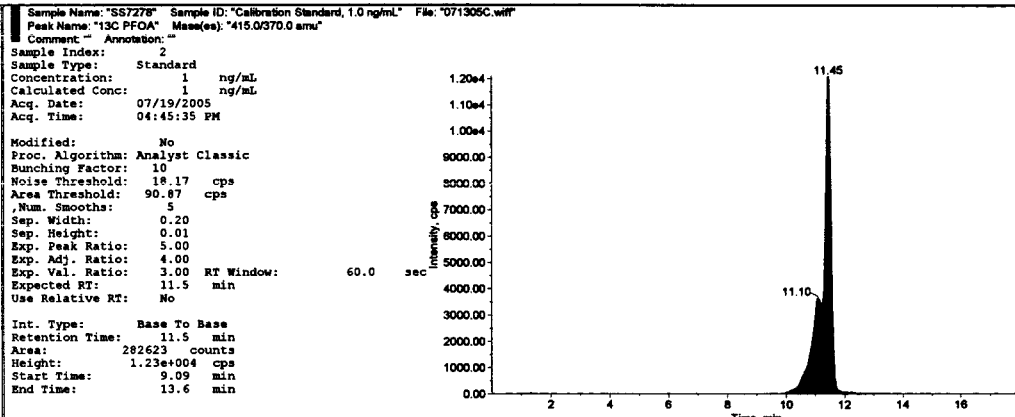
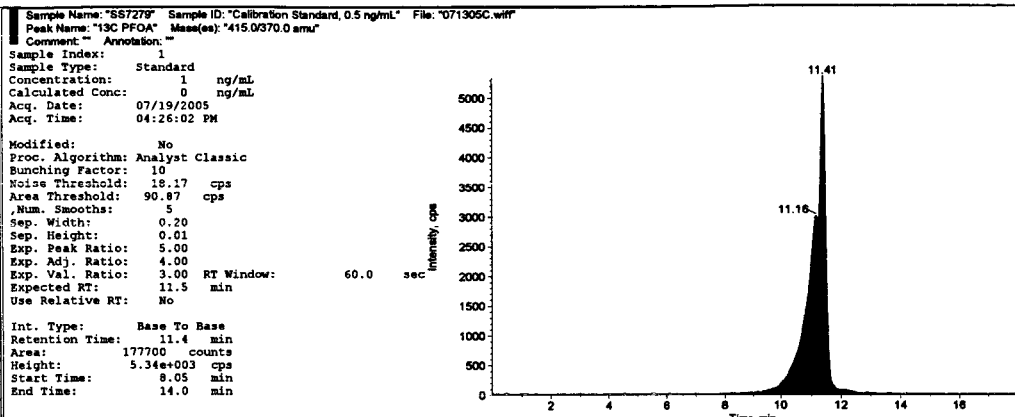
BSM 08/04/05

AMB 07/22/05

■ Untitled 3 (13C PFOA): "Linear" Regression ("1/x" weighting): $y = 1.89e+005 x + 9.69e+004$ ($r = 0.9971$)

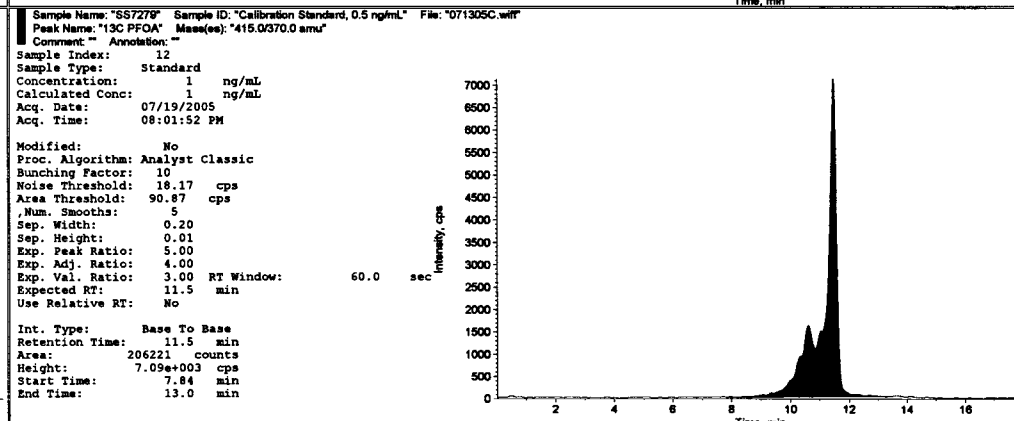
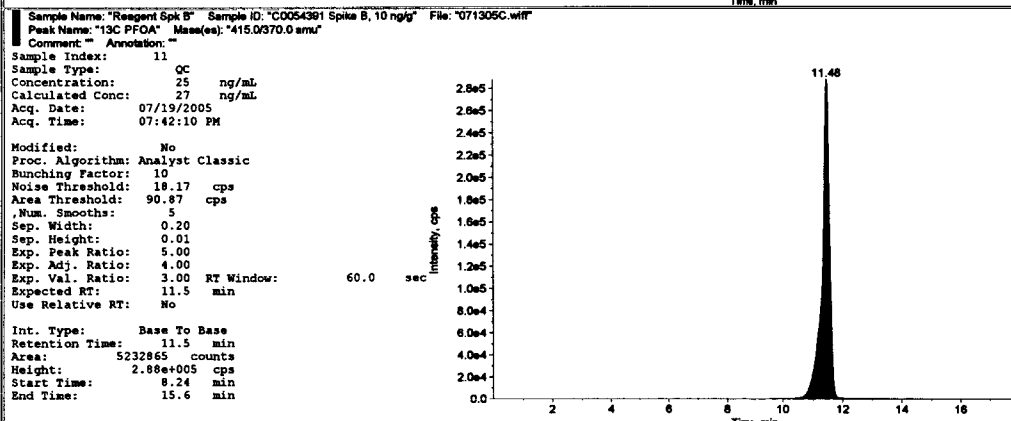
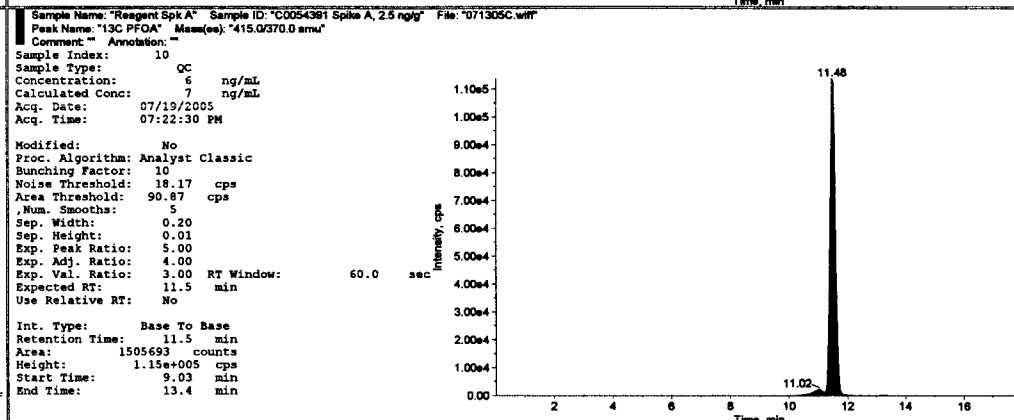
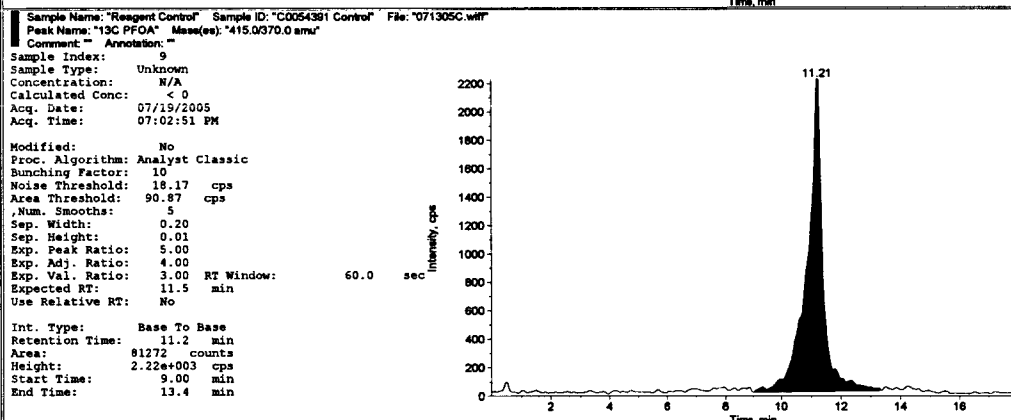
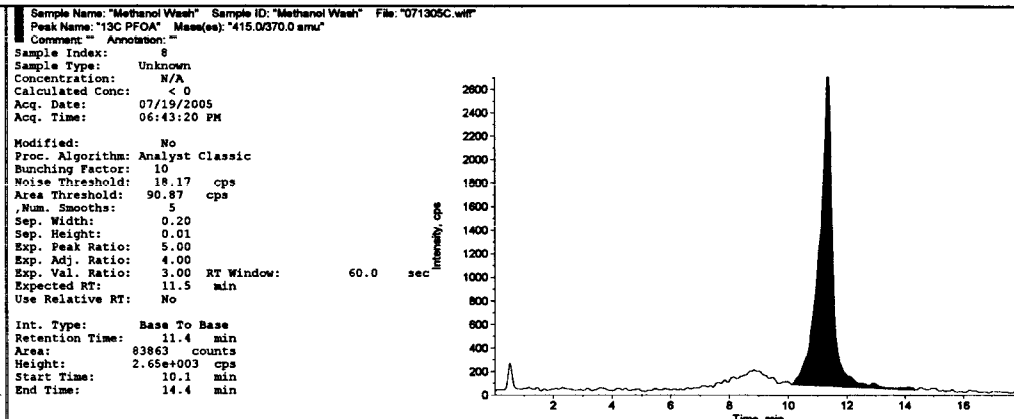
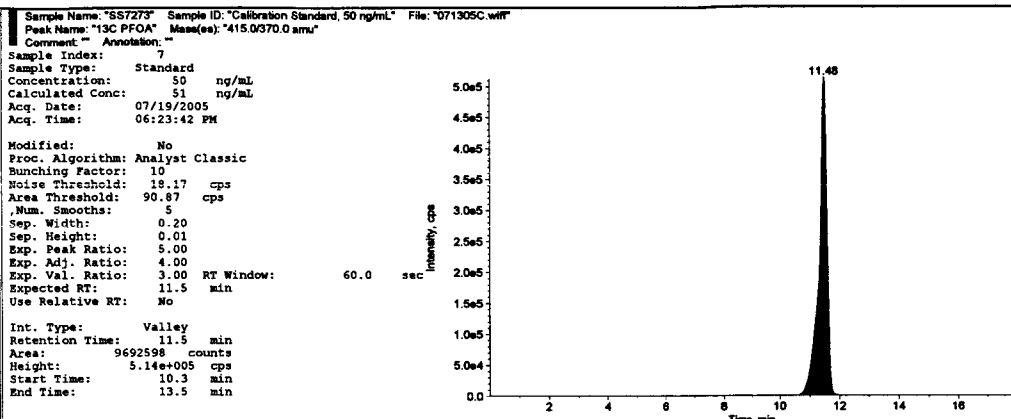


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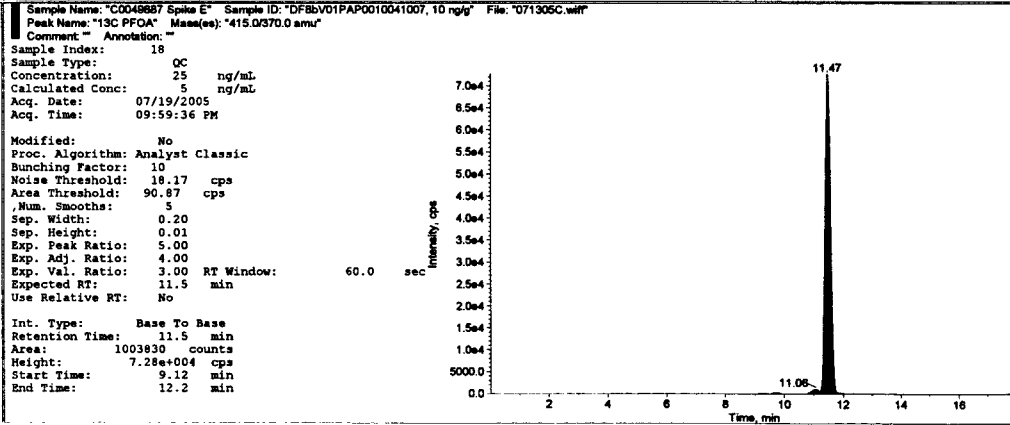
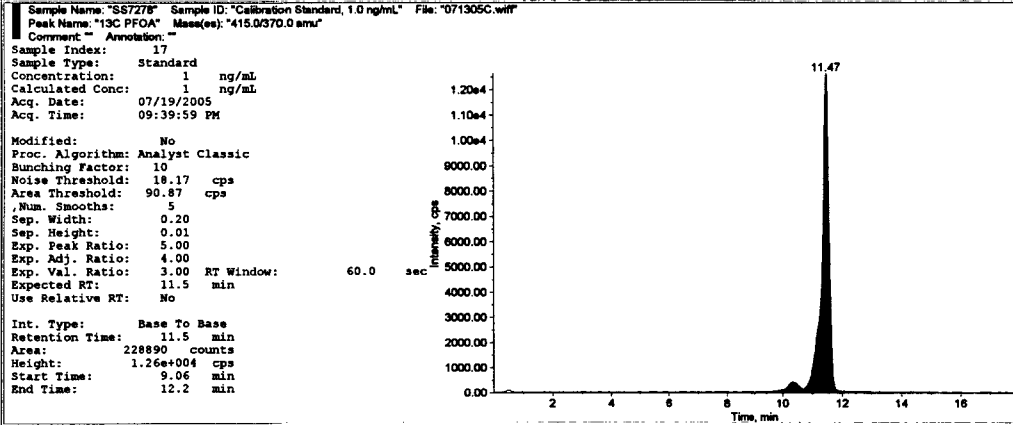
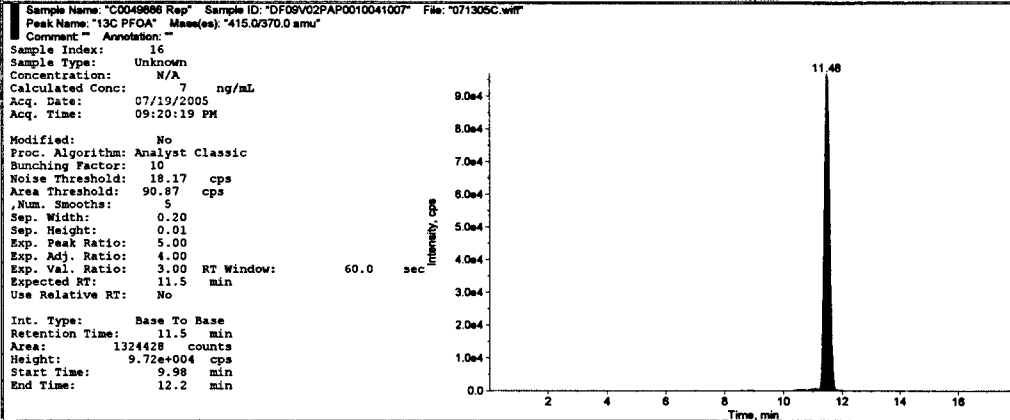
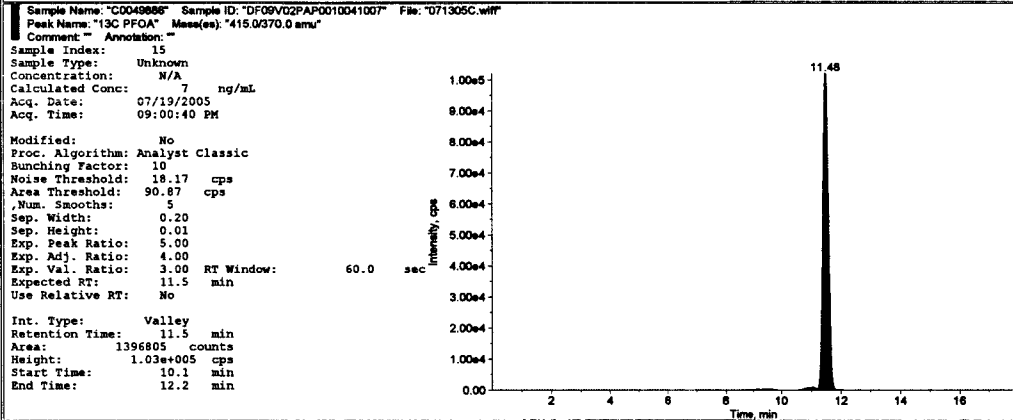
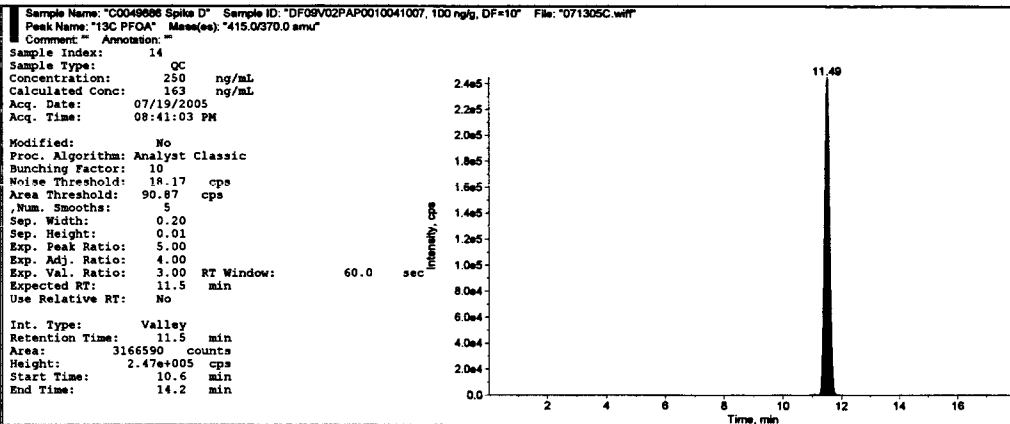
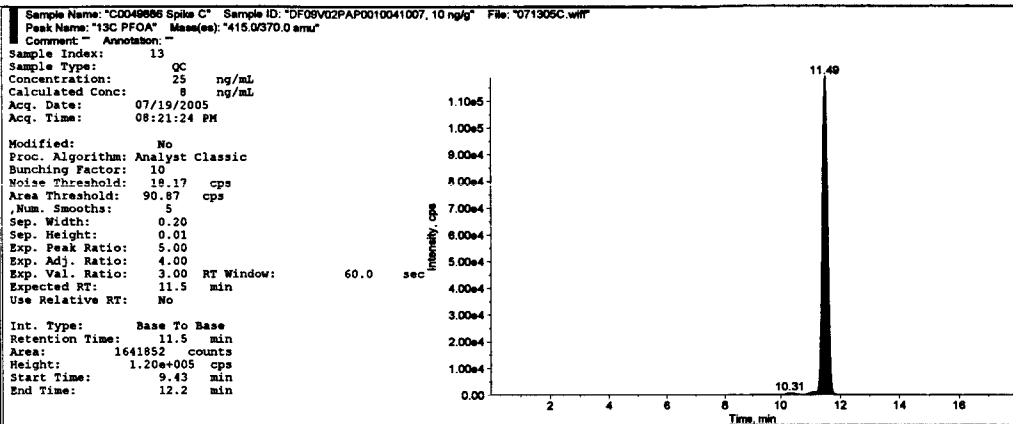


0436 Initials AMB
 Date 07/22/05
 Run# 1 To 38
 Sample Index 1 To 38
 © AMB 07/22/05

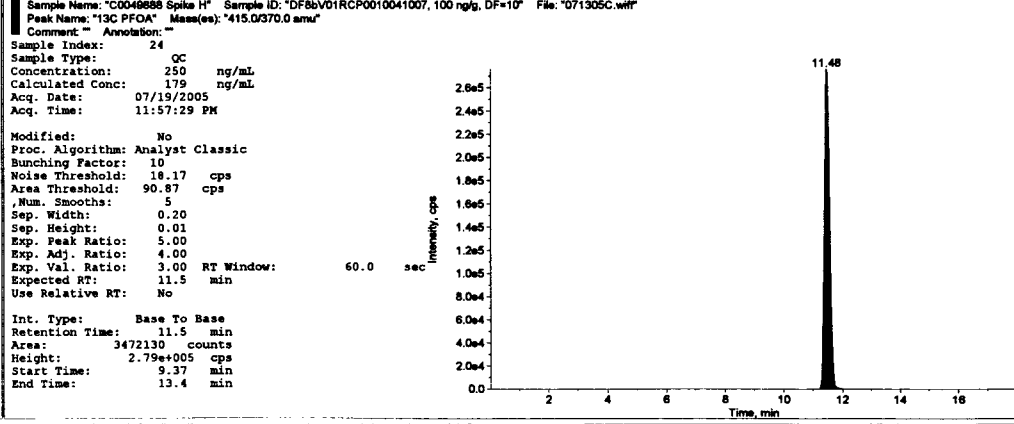
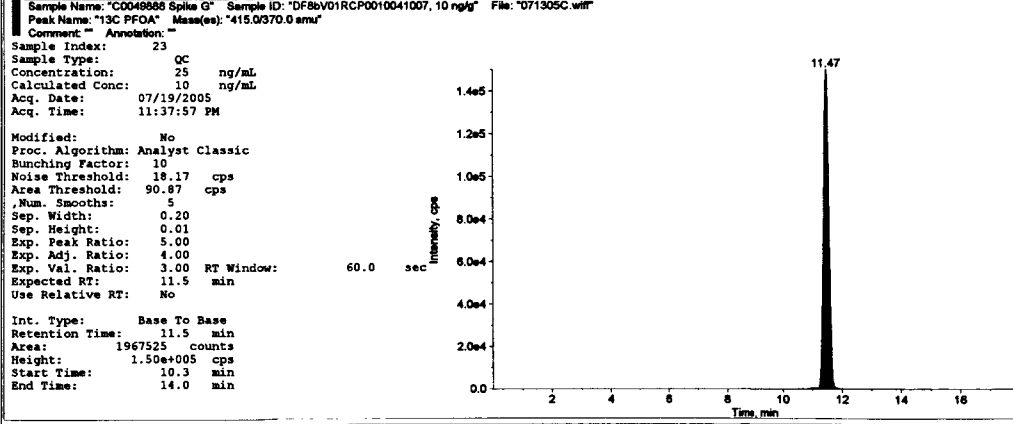
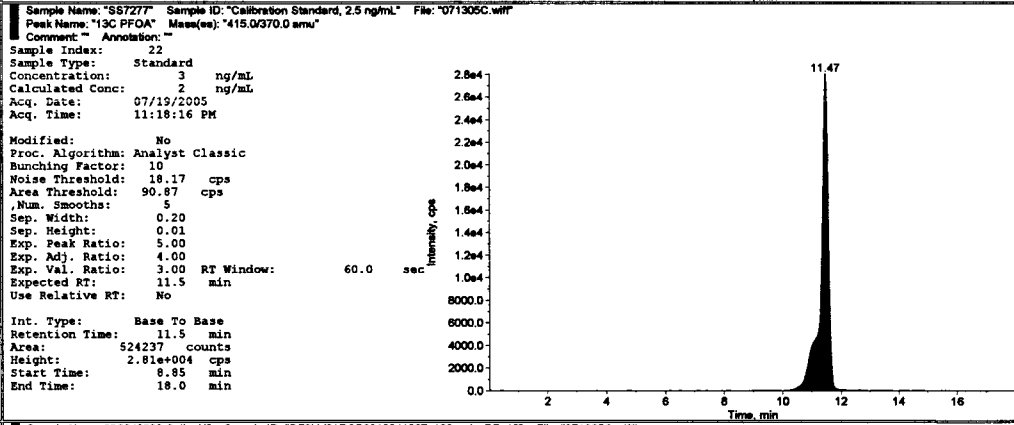
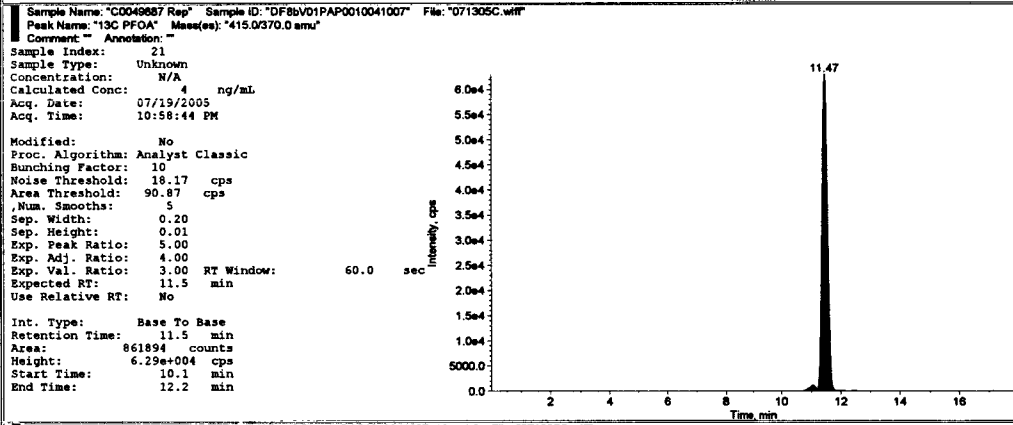
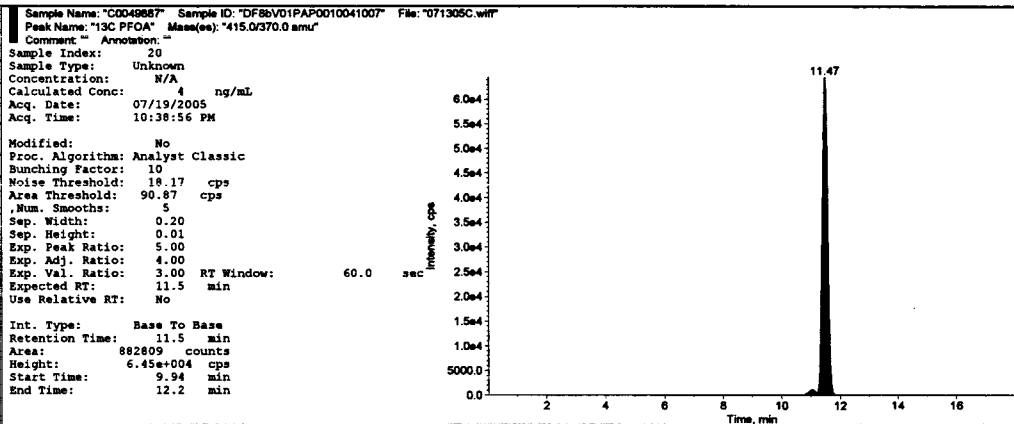
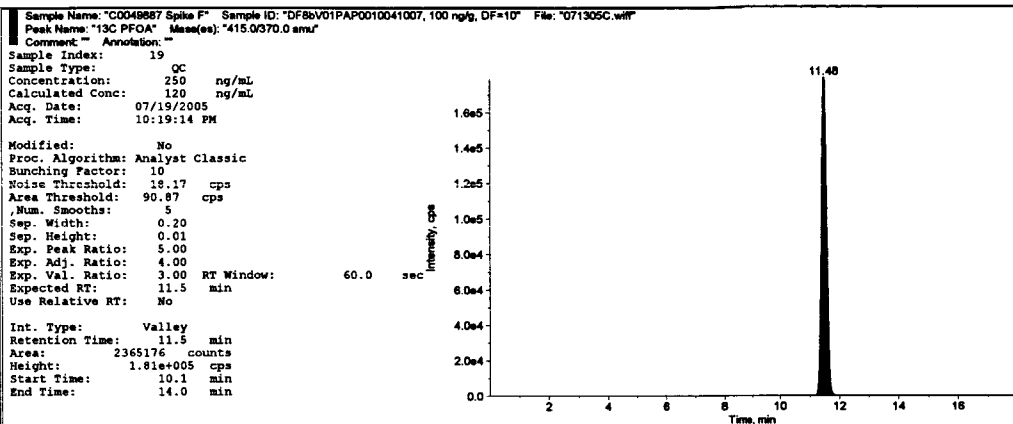
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 Sample Index: 1 To: 38



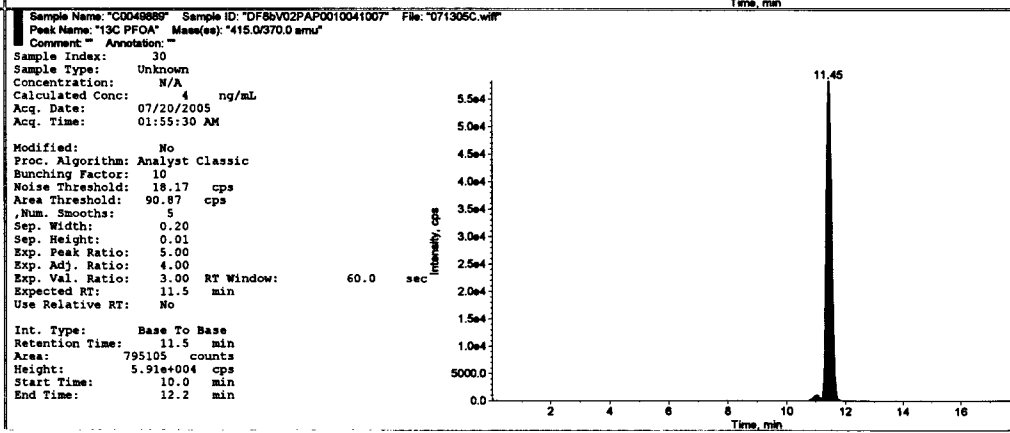
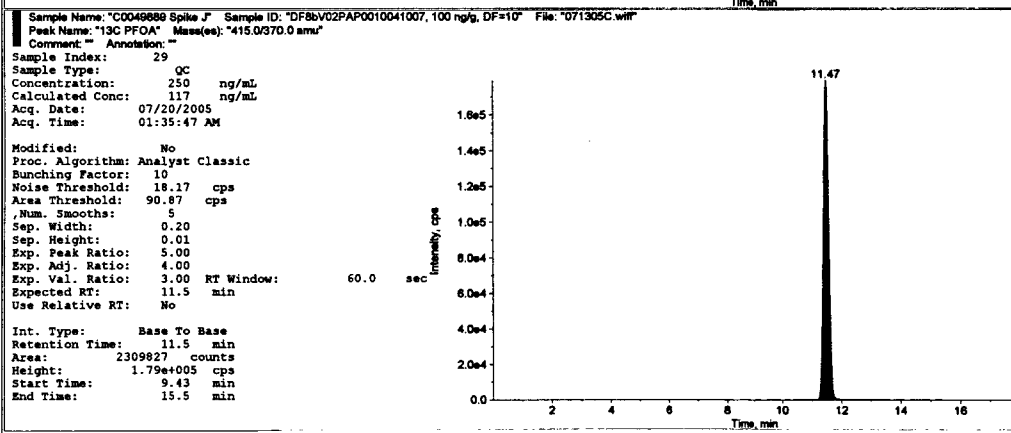
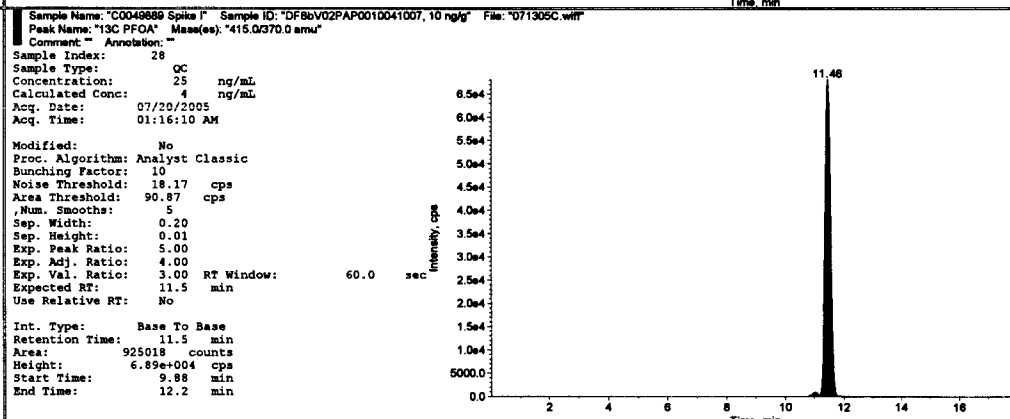
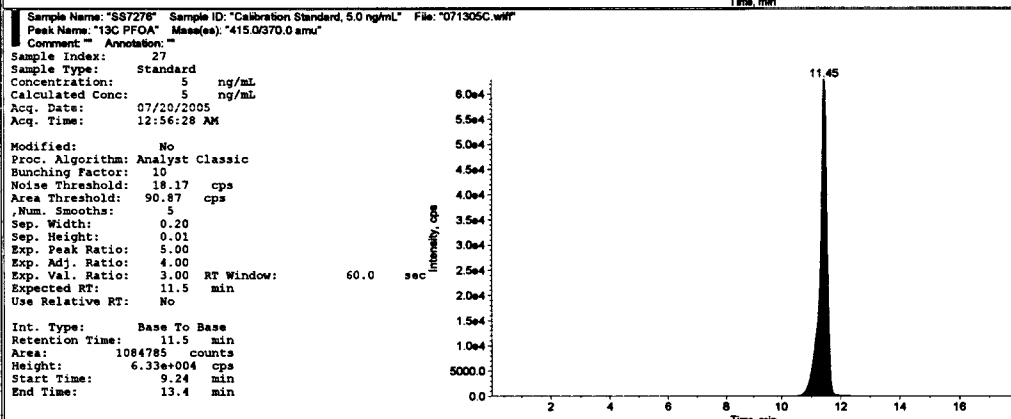
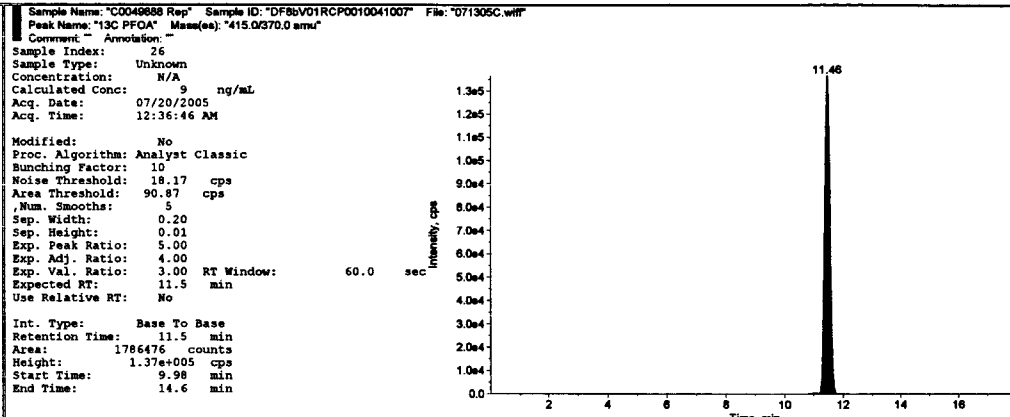
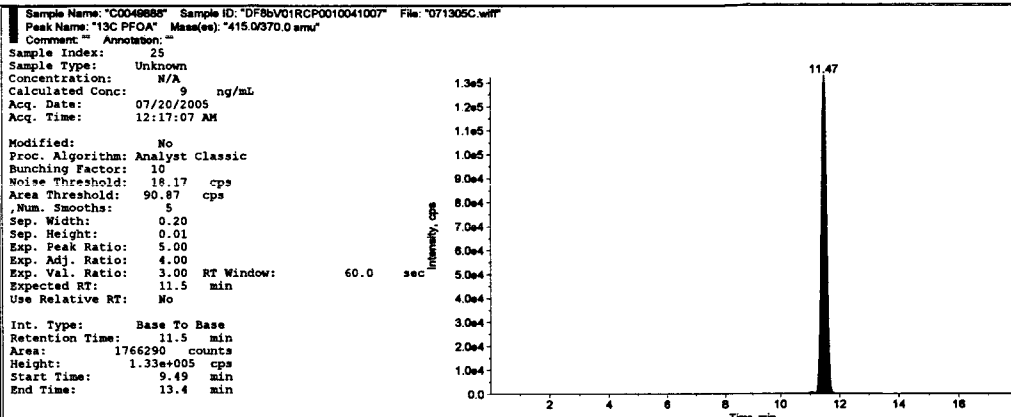
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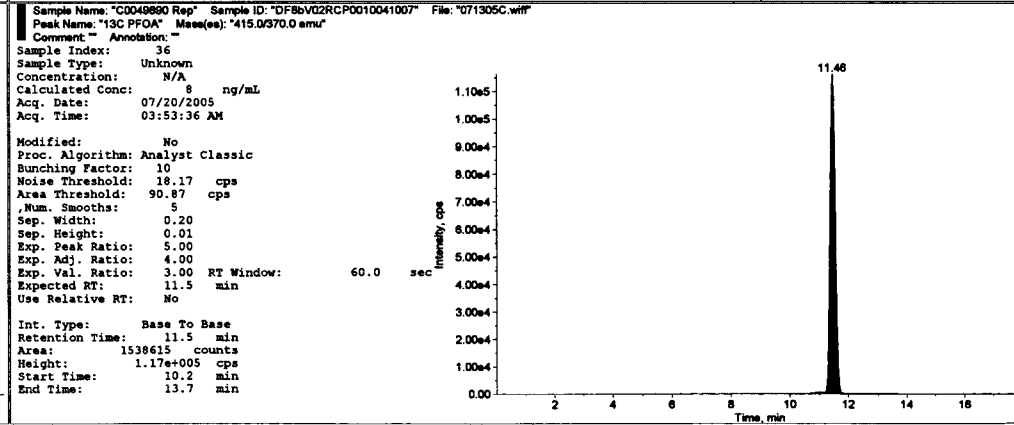
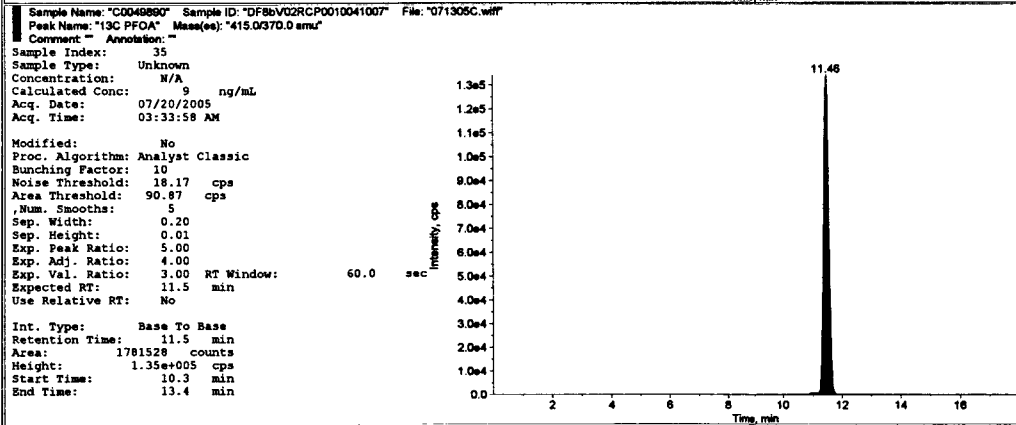
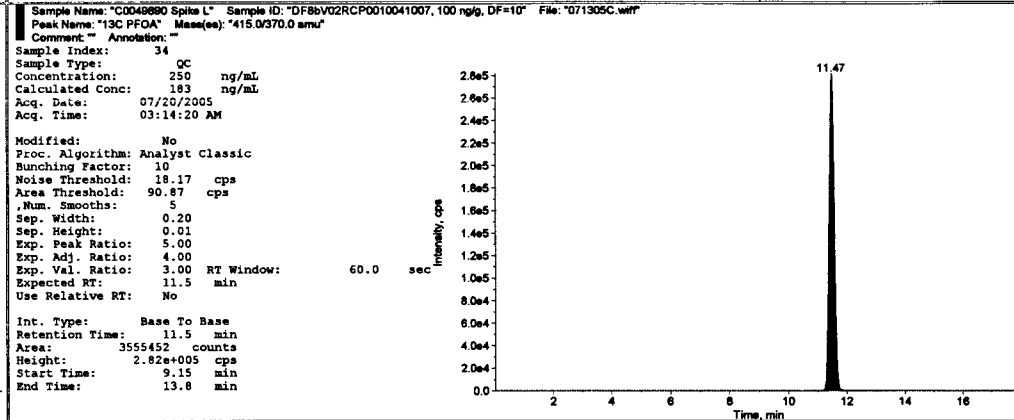
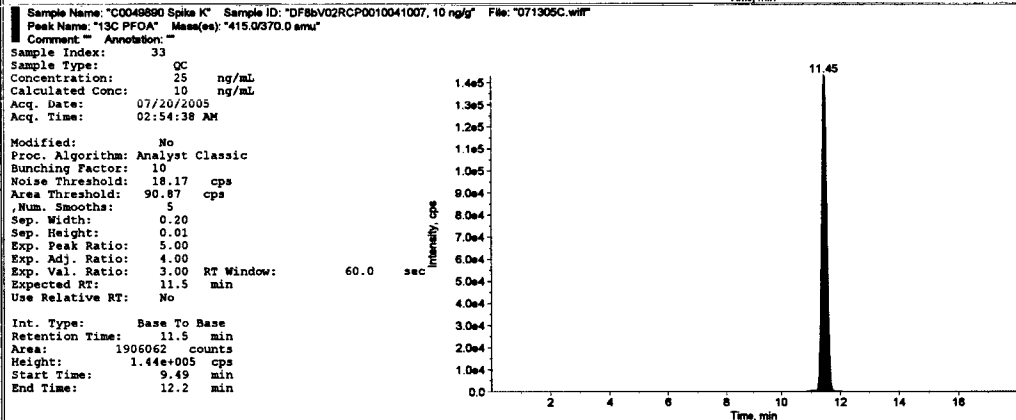
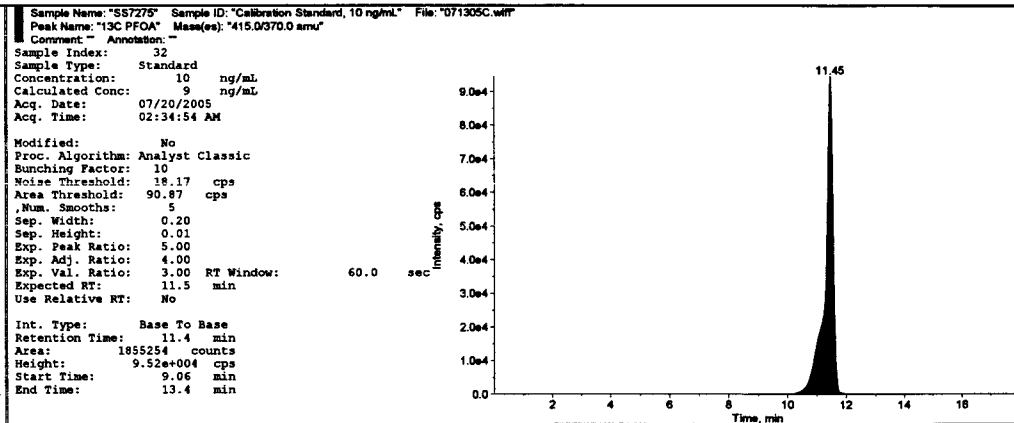
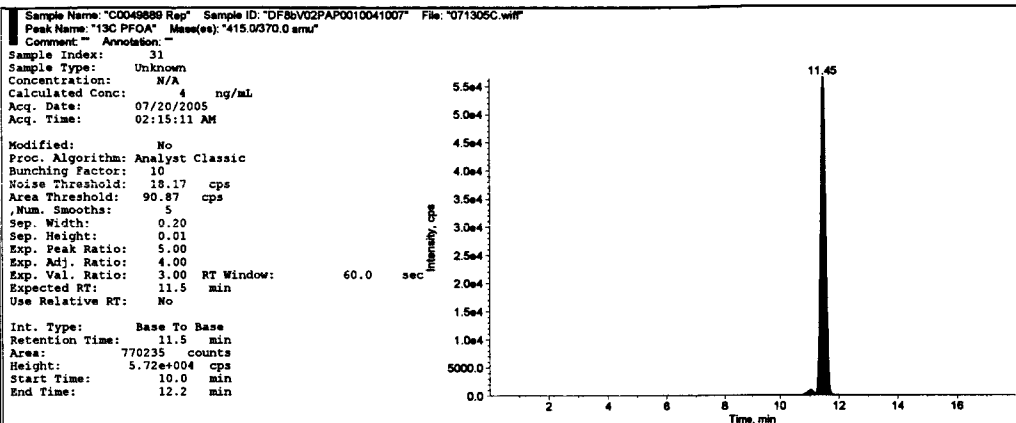


0438



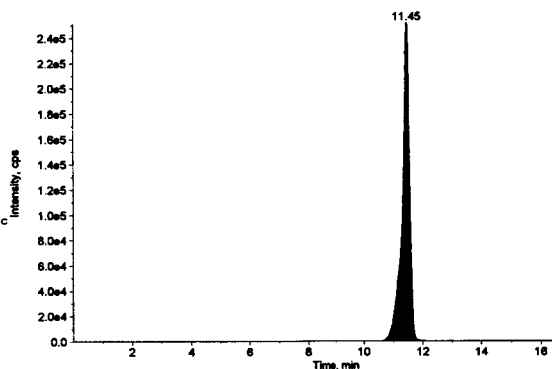
0439



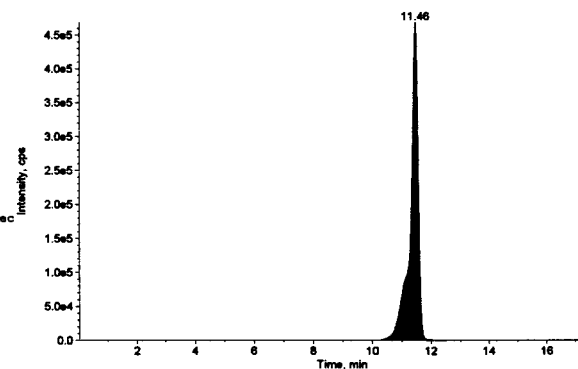


0441

Sample Name: "S67274" Sample ID: "Calibration Standard, 25 ng/mL" File: "071305C.wiff"
Peak Name: "13C PFOA" Mass(es): "415.0370.0 amu"
Comment: "" Annotation: ""
Sample Index: 37
Sample Type: Standard
Concentration: 25 ng/mL
Calculated Conc: 24 ng/mL
Acq. Date: 07/20/2005
Acq. Time: 04:13:19 AM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 18.17 cps
Area Threshold: 90.87 cps
Num. Smooths: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window: 60.0 sec
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 4546418 counts
Height: 2.54e+005 cps
Start Time: 9.58 min
End Time: 14.6 min



Sample Name: "S67273" Sample ID: "Calibration Standard, 50 ng/mL" File: "071305C.wiff"
Peak Name: "13C PFOA" Mass(es): "415.0370.0 amu"
Comment: "" Annotation: ""
Sample Index: 38
Sample Type: Standard
Concentration: 50 ng/mL
Calculated Conc: 47 ng/mL
Acq. Date: 07/20/2005
Acq. Time: 04:32:57 AM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 10
Noise Threshold: 18.17 cps
Area Threshold: 90.87 cps
Num. Smooths: 5
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 RT Window: 60.0 sec
Expected RT: 11.5 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 11.5 min
Area: 9024710 counts
Height: 4.72e+005 cps
Start Time: 8.45 min
End Time: 14.5 min



Raw Data

A	B	C	D	E	F	G	H	I	J	K
1	PFBS									
2	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Net Concentration	Recalculated Concentration (ng)	Accuracy (%)	Run #
3	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFBS	15755	0.5	0.46	92	71305C-301
4	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFBS	31671	1	1.04	104	71305C-302
5	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFBS	74740	2.5	2.61	104	71305C-303
6	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFBS	152640	5	5.44	109	71305C-304
7	SS7275	Calibration Standard, 10 ng/mL	Standard	1.0	PFBS	290389	10	10.5	105	71305C-305
8	SS7274	Calibration Standard, 25 ng/mL	Standard	1.0	PFBS	705057	25	25.5	102	71305C-306
9	SS7273	Calibration Standard, 50 ng/mL	Standard	1.0	PFBS	1373165	50	49.9	99.7	71305C-307
10	Methanol Wash	Methanol Wash	Unknown	1.0	PFBS	1257	N/A	< 0	N/A	71305C-308
11	Reagent Control	C0054391 Control	Unknown	1.0	PFBS	0	N/A	No Peak	N/A	71305C-309
12	Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1.0	PFBS	164948	6.25	5.89	94.2	71305C-310
13	Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1.0	PFBS	708966	25	25.7	103	71305C-311
14	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1.0	PFBS	16906	0.5	0.502	100	71305C-312
15	C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1.0	PFBS	1940717	25	70.5	282	71305C-313
16	C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	755999	250	274	110	71305C-314
17	C0049886	DF09V02PAP0010041007	Unknown	1.0	PFBS	1385792	N/A	50.3	N/A	71305C-315
18	C0049886 Rep	DF09V02PAP0010041007	Unknown	1.0	PFBS	1371807	N/A	49.8	N/A	71305C-316
19	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1.0	PFBS	28426	1	0.921	92.1	71305C-317
20	C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1.0	PFBS	1983943	25	72.1	288	71305C-318
21	C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	722933	250	262	105	71305C-319
22	C0049887	DF8bV01PAP0010041007	Unknown	1.0	PFBS	1566607	N/A	56.9	N/A	71305C-320
23	C0049887 Rep	DF8bV01PAP0010041007	Unknown	1.0	PFBS	1478165	N/A	53.7	N/A	71305C-321
24	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1.0	PFBS	67450	2.5	2.34	93.7	71305C-322
25	C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1.0	PFBS	670117	25	24.3	97.1	71305C-323
26	C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	572938	250	207	83	71305C-324
27	C0049888	DF8bV01RCP0010041007	Unknown	1.0	PFBS	151514	N/A	5.4	N/A	71305C-325
28	C0049888 Rep	DF8bV01RCP0010041007	Unknown	1.0	PFBS	147800	N/A	5.27	N/A	71305C-326
29	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1.0	PFBS	149337	5	5.32	106	71305C-327
30	C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1.0	PFBS	3305236	25	120	481	71305C-328
31	C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=10	Quality Control	10.0	PFBS	890030	250	323	129	71305C-329
32	C0049889	DF8bV02PAP0010041007	Unknown	1.0	PFBS	2871236	N/A	104	N/A	71305C-330
33	C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	PFBS	2.69E+06	N/A	98	N/A	71305C-331
34	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFBS	2.66E+05	10.0	9.57	95.7	71305C-332
35	C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	PFBS	7.05E+05	25.0	25.6	102	71305C-333
36	C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFBS	6.06E+05	250	219	87.8	71305C-334
37	C0049890	DF8bV02RCP0010041007	Unknown	1	PFBS	1.77E+05	N/A	6.32	N/A	71305C-335
38	C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	PFBS	1.57E+05	N/A	5.61	N/A	71305C-336
39	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFBS	6.69E+05	25.0	24.2	97	71305C-337
40	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFBS	1.37E+06	50.0	49.7	99.4	71305C-338
41										
42										
43	PFHS									
44	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Net Concentration	Recalculated Concentration (ng)	Accuracy (%)	Run #
45	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFHS	25081	0.500	0.437	87.5	71305C-301
46	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFHS	54186	1.00	1.02	102	71305C-302
47	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFHS	131654	2.50	2.58	103	71305C-303
48	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFHS	284582	5.00	5.65	113	71305C-304
49	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFHS	491306	10.0	9.8	98	71305C-305
50	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFHS	1278573	25.0	25.6	102	71305C-306
51	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFHS	2495835	50.0	50.1	100	71305C-307
52	Methanol Wash	Methanol Wash	Unknown	1	PFHS	0	N/A	No Peak	N/A	71305C-308
53	Reagent Control	C0054391 Control	Unknown	1	PFHS	0	N/A	No Peak	N/A	71305C-309
54	Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1	PFHS	340405	6.25	6.77	108	71305C-310
55	Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1	PFHS	1257070	25.0	25.2	101	71305C-311
56	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFHS	25783	0.500	0.452	90.3	71305C-312
57	C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1	PFHS	21367826	25.0	429	1720	71305C-313
58	C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	5188657	250	1040	417	71305C-314
59	C0049886	DF09V02PAP0010041007	Unknown	1	PFHS	20759953	N/A	417	N/A	71305C-315
60	C0049886 Rep	DF09V02PAP0010041007	Unknown	1	PFHS	21364120	N/A	429	N/A	71305C-316
61	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFHS	57314	1.00	1.08	108	71305C-317
62	C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1	PFHS	13942093	25.0	280	1120	71305C-318
63	C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	3294002	250	661	264	71305C-319
64	C0049887	DF8bV01PAP0010041007	Unknown	1	PFHS	13775398	N/A	277	N/A	71305C-320
65	C0049887 Rep	DF8bV01PAP0010041007	Unknown	1	PFHS	13267946	N/A	266	N/A	71305C-321
66	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFHS	128624	2.50	2.52	101	71305C-322
67	C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1	PFHS	4894294	25.0	98.2	393	71305C-323
68	C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1660051	250	333	133	71305C-324
69	C0049888	DF8bV01RCP0010041007	Unknown	1	PFHS	3862872	N/A	77.5	N/A	71305C-325
70	C0049888 Rep	DF8bV01RCP0010041007	Unknown	1	PFHS	3703155	N/A	74.3	N/A	71305C-326
71	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFHS	261226	5.00	5.18	104	71305C-327
72	C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1	PFHS	28098317	25.0	564	2260	71305C-328
73	C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	6746494	250	1350	542	71305C-329
74	C0049889	DF8bV02PAP0010041007	Unknown	1	PFHS	27631107	N/A	555	N/A	71305C-330
75	C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	PFHS	26021886	N/A	523	N/A	71305C-331
76	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFHS	468387	10.0	9.34	93.4	71305C-332
77	C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	PFHS	5201686	25.0	104	418	71305C-333
78	C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PFHS	1822199	250	365	146	71305C-334
79	C0049890	DF8bV02RCP0010041007	Unknown	1	PFHS	4073514	N/A	81.8	N/A	71305C-335
80	C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	PFHS	4176035	N/A	83.8	N/A	71305C-336
81	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFHS	1211103	25.0	24.3	97	71305C-337
82	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFHS	2491646	50.0	50	100	71305C-338
83										
84	PFOS									
85	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Net Concentration	Recalculated Concentration (ng)	Accuracy (%)	Run #

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Raw Data

	A	B	C	D	E	F	G	H	I	J	K
86	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PPOS	21820	0.5	< 0	N/A	71305C-301	
87	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PPOS	47267	1	< 0	N/A	71305C-302	
88	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PPOS	122976	2.5	1.27	50.8	71305C-303	
89	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PPOS	255175	5	3.94	78.7	71305C-304	
90	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PPOS	477207	10	8.41	84.1	71305C-305	
91	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PPOS	1162335	25	22.2	88.9	71305C-306	
92	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PPOS	2314198	50	45.5	90.9	71305C-307	
93	Methanol Wash	Methanol Wash	Unknown	1	PPOS	2487	N/A	< 0	N/A	71305C-308	
94	Reagent Control	C0054391 Control	Unknown	1	PPOS	3692	N/A	< 0	N/A	71305C-309	
95	Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1	PPOS	299213	6.25	4.83	77.2	71305C-310	
96	Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1	PPOS	1180630	25	22.6	90.4	71305C-311	
97	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PPOS	28165	0.5	< 0	N/A	71305C-312	
98	C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1	PPOS	97596657	25	1970	7870	71305C-313	
99	C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PPOS	36648997	250	7380	2950	71305C-314	
100	C0049886 Rep	DF09V02PAP0010041007	Unknown	1	PPOS	96720824	N/A	1950	N/A	71305C-315	
101	C0049886 Rep	DF09V02PAP0010041007	Unknown	1	PPOS	98123666	N/A	1980	N/A	71305C-316	
102	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PPOS	319687	1	5.24	524	71305C-317	
103	C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1	PPOS	105505816	25	2130	8510	71305C-318	
104	C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PPOS	40807276	250	8220	3290	71305C-319	
105	C0049887	DF8bV01PAP0010041007	Unknown	1	PPOS	105545656	N/A	2130	N/A	71305C-320	
106	C0049887 Rep	DF8bV01PAP0010041007	Unknown	1	PPOS	104606650	N/A	2110	N/A	71305C-321	
107	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PPOS	403794	2.5	6.93	277	71305C-322	
108	C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1	PPOS	85551418	25	1720	6900	71305C-323	
109	C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PPOS	26167433	250	5270	2110	71305C-324	
110	C0049888	DF8bV01RCP0010041007	Unknown	1	PPOS	89034612	N/A	1790	N/A	71305C-325	
111	C0049888 Rep	DF8bV01RCP0010041007	Unknown	1	PPOS	90133845	N/A	1820	N/A	71305C-326	
112	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PPOS	407625	5	7.01	140	71305C-327	
113	C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1	PPOS	124720771	25	2510	10100	71305C-328	
114	C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	PPOS	54414391	250	11000	4380	71305C-329	
115	C0049889	DF8bV02PAP0010041007	Unknown	1	PPOS	124601079	N/A	2510	N/A	71305C-330	
116	C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	PPOS	121368229	N/A	2450	N/A	71305C-331	
117	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PPOS	953650	10	18	180	71305C-332	
118	C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	PPOS	83417886	25	1680	6720	71305C-333	
119	C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	PPOS	32440695	250	6530	2610	71305C-334	
120	C0049890	DF8bV02RCP0010041007	Unknown	1	PPOS	84199422	N/A	1700	N/A	71305C-335	
121	C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	PPOS	88092828	N/A	1780	N/A	71305C-336	
122	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PPOS	1304161	25	25.1	100	71305C-337	
123	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PPOS	2343297	50	46	92.1	71305C-338	
124											
125											
126											

13C PFOA

	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng)	Calculated Concentration (ng)	Accuracy (%)	Run #	
127	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	177700	0.5	0.428	85.7	71305C-301	
128	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	282623	1	0.985	98.5	71305C-302	
129	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	615685	2.5	2.75	110	71305C-303	
130	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1243615	5	6.08	122	71305C-304	
131	SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	2212688	10	11.2	112	71305C-305	
132	SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	5103737	25	26.6	106	71305C-306	
133	SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9692598	50	50.9	102	71305C-307	
134	Methanol Wash	Methanol Wash	Unknown	1	13C PFOA	83863	N/A	< 0	N/A	71305C-308	
135	Reagent Control	C0054391 Control	Unknown	1	13C PFOA	81272	N/A	< 0	N/A	71305C-309	
136	Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1	13C PFOA	1505693	6.25	7.47	120	71305C-310	
137	Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1	13C PFOA	5232865	25	27.2	109	71305C-311	
138	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	13C PFOA	206221	0.5	0.58	116	71305C-312	
139	C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1641852	25	8.19	32.8	71305C-313	
140	C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3166590	250	163	65.1	71305C-314	
141	C0049886	DF09V02PAP0010041007	Unknown	1	13C PFOA	1396805	N/A	6.9	N/A	71305C-315	
142	C0049886 Rep	DF09V02PAP0010041007	Unknown	1	13C PFOA	1324428	N/A	6.51	N/A	71305C-316	
143	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	13C PFOA	228890	1	0.7	70	71305C-317	
144	C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1003830	25	4.81	19.2	71305C-318	
145	C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2365176	250	120	48.1	71305C-319	
146	C0049887	DF8bV01PAP0010041007	Unknown	1	13C PFOA	882809	N/A	4.17	N/A	71305C-320	
147	C0049887 Rep	DF8bV01PAP0010041007	Unknown	1	13C PFOA	861894	N/A	4.06	N/A	71305C-321	
148	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	13C PFOA	524237	2.5	2.27	90.7	71305C-322	
149	C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1967525	25	9.92	39.7	71305C-323	
150	C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3472130	250	179	71.6	71305C-324	
151	C0049888	DF8bV01RCP0010041007	Unknown	1	13C PFOA	1766290	N/A	8.86	N/A	71305C-325	
152	C0049888 Rep	DF8bV01RCP0010041007	Unknown	1	13C PFOA	1786476	N/A	8.96	N/A	71305C-326	
153	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	13C PFOA	1084785	5	5.24	105	71305C-327	
154	C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1	13C PFOA	925018	25	4.39	17.6	71305C-328	
155	C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	2309827	250	117	47	71305C-329	
156	C0049889	DF8bV02PAP0010041007	Unknown	1	13C PFOA	795105	N/A	3.7	N/A	71305C-330	
157	C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	13C PFOA	770235	N/A	3.57	N/A	71305C-331	
158	SS7275	Calibration Standard, 10 ng/mL	Standard	1	13C PFOA	1855254	10	9.33	93.3	71305C-332	
159	C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	13C PFOA	1906062	25	9.6	38.4	71305C-333	
160	C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=10	Quality Control	10	13C PFOA	3555452	250	183	73.4	71305C-334	
161	C0049890	DF8bV02RCP0010041007	Unknown	1	13C PFOA	1781528	N/A	8.94	N/A	71305C-335	
162	C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	13C PFOA	1538615	N/A	7.65	N/A	71305C-336	
163	SS7274	Calibration Standard, 25 ng/mL	Standard	1	13C PFOA	4546418	25	23.6	94.4	71305C-337	
164	SS7273	Calibration Standard, 50 ng/mL	Standard	1	13C PFOA	9024710	50	47.4	94.7	71305C-338	
165											
166											
167											
168											

PFOA

	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Concentration (ng)	Calculated Concentration (ng)	Accuracy (%)	Run #
169	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	152030	0.5	0.158	31.6	71305C-301
170	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	276132	1	0.716	71.6	71305C-302
171	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	676478	2.5	2.51	101	71305C-303
172	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1386295	5	5.7	114	71305C-304

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Raw Data

A	B	C	D	E	F	G	H	I	J	K
174	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2572205	10	11	110	71305C-305
175	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5922677	25	26.1	104	71305C-306
176	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10918506	50	48.5	97.1	71305C-307
177	Methanol Wash	Methanol Wash	Unknown	1	PFOA	80114	N/A	<0	N/A	71305C-308
178	Reagent Control	C0054391 Control	Unknown	1	PFOA	75087	N/A	<0	N/A	71305C-309
179	Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1	PFOA	1617762	6.25	6.74	108	71305C-310
180	Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1	PFOA	5972528	25	26.3	105	71305C-311
181	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA	194021	0.5	0.347	69.4	71305C-312
182	C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1	PFOA	74691305	25	335	1340	71305C-313
183	C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA	20958666	250	936	375	71305C-314
184	C0049886	DF09V02PAP0010041007	Unknown	1	PFOA	71568996	N/A	321	N/A	71305C-315
185	C0049886 Rep	DF09V02PAP0010041007	Unknown	1	PFOA	69835640	N/A	313	N/A	71305C-316
186	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA	417242	1	1.35	135	71305C-317
187	C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1	PFOA	96938481	25	435	1740	71305C-318
188	C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA	32606764	250	1460	584	71305C-319
189	C0049887	DF8bV01PAP0010041007	Unknown	1	PFOA	94931069	N/A	426	N/A	71305C-320
190	C0049887 Rep	DF8bV01PAP0010041007	Unknown	1	PFOA	90939799	N/A	408	N/A	71305C-321
191	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA	961364	2.5	3.79	152	71305C-322
192	C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1	PFOA	49847301	25	223	894	71305C-323
193	C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA	13495888	250	601	240	71305C-324
194	C0049888	DF8bV01RCP0010041007	Unknown	1	PFOA	49634130	N/A	222	N/A	71305C-325
195	C0049888 Rep	DF8bV01RCP0010041007	Unknown	1	PFOA	47262880	N/A	212	N/A	71305C-326
196	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA	1424412	5	5.88	118	71305C-327
197	C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1	PFOA	86680092	25	389	1560	71305C-328
198	C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA	29165507	250	1310	522	71305C-329
199	C0049889	DF8bV02PAP0010041007	Unknown	1	PFOA	85792086	N/A	365	N/A	71305C-330
200	C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	PFOA	81391265	N/A	385	N/A	71305C-331
201	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA	2463709	10	10.5	105	71305C-332
202	C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	PFOA	52748335	25	236	946	71305C-333
203	C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA	16202334	250	723	289	71305C-334
204	C0049890	DF8bV02RCP0010041007	Unknown	1	PFOA	52117601	N/A	234	N/A	71305C-335
205	C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	PFOA	50090193	N/A	225	N/A	71305C-336
206	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA	5527250	25	24.3	97.2	71305C-337
207	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA	10584455	50	47	94.1	71305C-338
208										
209										
210										
PFOA Confirm Ion										
211	Sample Name	Sample ID	Sample Type	Dilution Factor	Analyte Peak Name	Analyte Peak Area (counts)	Peak Concentration (ng/mL)	Calculated Concentration (ng/mL)	Accuracy (%)	Run #
212	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	6352	0.5	0.3	60	71305C-301
213	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	12624	1	1.28	128	71305C-302
214	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	19373	2.5	2.33	93.3	71305C-303
215	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	40132	5	5.57	111	71305C-304
216	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	75879	10	11.2	112	71305C-305
217	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	172774	25	26.3	105	71305C-306
218	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	326301	50	50.2	100	71305C-307
219	Methanol Wash	Methanol Wash	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	N/A	71305C-308
220	Reagent Control	C0054391 Control	Unknown	1	PFOA Confirm Ion	0	N/A	No Peak	N/A	71305C-309
221	Reagent Spk A	C0054391 Spike A, 2.5 ng/g	Quality Control	1	PFOA Confirm Ion	46671	6.25	6.59	105	71305C-310
222	Reagent Spk B	C0054391 Spike B, 10 ng/g	Quality Control	1	PFOA Confirm Ion	170414	25	25.9	104	71305C-311
223	SS7279	Calibration Standard, 0.5 ng/mL	Standard	1	PFOA Confirm Ion	5222	0.5	0.124	24.7	71305C-312
224	C0049886 Spike C	DF09V02PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	6913423	25	1080	4310	71305C-313
225	C0049886 Spike D	DF09V02PAP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA Confirm Ion	1226161	250	1910	763	71305C-314
226	C0049886	DF09V02PAP0010041007	Unknown	1	PFOA Confirm Ion	6737133	N/A	1050	N/A	71305C-315
227	C0049886 Rep	DF09V02PAP0010041007	Unknown	1	PFOA Confirm Ion	6525592	N/A	1020	N/A	71305C-316
228	SS7278	Calibration Standard, 1.0 ng/mL	Standard	1	PFOA Confirm Ion	12985	1	1.34	134	71305C-317
229	C0049887 Spike E	DF8bV01PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	10067174	25	1570	6280	71305C-318
230	C0049887 Spike F	DF8bV01PAP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA Confirm Ion	2051419	250	3190	1280	71305C-319
231	C0049887	DF8bV01PAP0010041007	Unknown	1	PFOA Confirm Ion	9737628	N/A	1520	N/A	71305C-320
232	C0049887 Rep	DF8bV01PAP0010041007	Unknown	1	PFOA Confirm Ion	9406124	N/A	1470	N/A	71305C-321
233	SS7277	Calibration Standard, 2.5 ng/mL	Standard	1	PFOA Confirm Ion	26726	2.5	3.48	139	71305C-322
234	C0049888 Spike G	DF8bV01RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	4362183	25	680	2720	71305C-323
235	C0049888 Spike H	DF8bV01RCP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA Confirm Ion	1717773	250	1110	445	71305C-324
236	C0049888	DF8bV01RCP0010041007	Unknown	1	PFOA Confirm Ion	4449633	N/A	694	N/A	71305C-325
237	C0049888 Rep	DF8bV01RCP0010041007	Unknown	1	PFOA Confirm Ion	4227080	N/A	659	N/A	71305C-326
238	SS7276	Calibration Standard, 5.0 ng/mL	Standard	1	PFOA Confirm Ion	37928	5	5.23	105	71305C-327
239	C0049889 Spike I	DF8bV02PAP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	7951599	25	1240	4960	71305C-328
240	C0049889 Spike J	DF8bV02PAP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA Confirm Ion	1623128	250	2530	1010	71305C-329
241	C0049889	DF8bV02PAP0010041007	Unknown	1	PFOA Confirm Ion	7874176	N/A	1230	N/A	71305C-330
242	C0049889 Rep	DF8bV02PAP0010041007	Unknown	1	PFOA Confirm Ion	7262598	N/A	1130	N/A	71305C-331
243	SS7275	Calibration Standard, 10 ng/mL	Standard	1	PFOA Confirm Ion	67020	10	9.77	97.7	71305C-332
244	C0049890 Spike K	DF8bV02RCP0010041007, 10 ng/g	Quality Control	1	PFOA Confirm Ion	4476412	25	698	2790	71305C-333
245	C0049890 Spike L	DF8bV02RCP0010041007, 100 ng/g, DF=1C	Quality Control	10	PFOA Confirm Ion	852860	250	1320	530	71305C-334
246	C0049890	DF8bV02RCP0010041007	Unknown	1	PFOA Confirm Ion	4402801	N/A	686	N/A	71305C-335
247	C0049890 Rep	DF8bV02RCP0010041007	Unknown	1	PFOA Confirm Ion	4335966	N/A	676	N/A	71305C-336
248	SS7274	Calibration Standard, 25 ng/mL	Standard	1	PFOA Confirm Ion	160961	25	24.4	97.7	71305C-337
249	SS7273	Calibration Standard, 50 ng/mL	Standard	1	PFOA Confirm Ion	302383	50	46.5	93	71305C-338

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