

## SUMMARY OF ANALYTICAL RESULTS: MULTI-CITY FOOD STUDY

The Multi-City Study was originally designed by Battelle Memorial Institute (Columbus, OH), to obtain preliminary data about the presence of fluorochemicals in foods and in drinking water to understand the potential sources of human exposure. The Multi-City Study paired each of three cities having manufacturing or commercial use of fluorochemical products (test cities) with three cities that do not (control cities).

Information on residues in selected foods was obtained from analyses of food samples collected in a market basket study. The market basket sampling of the original Multi-City Study design was implemented by Pace Analytical Services, Inc., Minneapolis, MN. The samples were analyzed for PFOS, PFOA, and FOSA by Centre Analytical Laboratories, Inc., State College, PA.

The distributions of the PFOS, PFOA, and FOSA residue data by food and city category reveal similar patterns of residue concentrations in the control and test cities for each type of food. A total of 12 samples were found to contain levels of fluorochemical residues above the limit of quantification. Of the 12 samples with measurable fluorochemical residue levels, eight were samples collected in test cities.

Measurable quantities of PFOS were found in five samples: four whole milk samples (three from test cities) and a ground beef sample (test city). PFOS residues found in the foods ranged from non-quantifiable levels to 0.852 ng/g.

Measurable quantities of PFOA were found in seven samples: two ground beef samples (neither from test cities); two bread samples (one from a test city); two apple samples (both from test cities); and one green bean sample (from a test city). PFOA residue levels ranged from non-quantifiable levels to 2.35 ng/g. A value of 14.7 ng/g was found for PFOA in a bread sample from a control city, but was considered “suspect” by Centre Analytical.

# **ANALYTICAL REPORT**

## **STUDY TITLE**

Analysis of PFOS, FOSA and PFOA From Various Food Matrices Using HPLC  
Electrospray/Mass Spectrometry

## **DATA REQUIREMENTS**

EPA TSCA Good Laboratory Practice Standards 40 CFR 792

## **STUDY DIRECTOR**

Karen Smith, Centre

## **SPONSOR REPRESENTATIVE**

Susan A. Beach, 3M

## **ANALYTICAL REPORT COMPLETION DATE**

June 21, 2001

## **PERFORMING LABORATORY / TESTING FACILITY**

Centre Analytical Laboratories, Inc. (Centre)  
3048 Research Drive  
State College, PA 16801  
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## **STUDY SPONSOR**

3M Environmental Technology and Safety Services  
Building 2-3E-09  
PO Box 33331  
St. Paul, MN 55133-3331

## **PROJECT IDENTIFICATION**

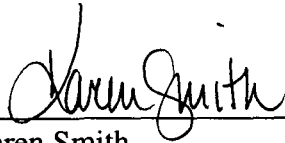
Centre Protocol Number: 00P-023-057  
Centre Study Number: 023-057

Total Pages: 151

**GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT**

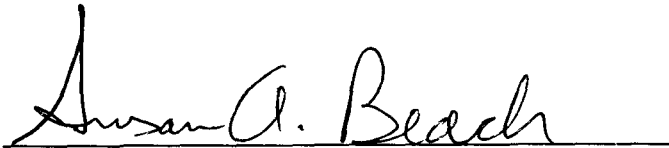
Centre Study Number 023-057, entitled "Analysis of PFOS, FOSA and PFOA From Various Food Matrices Using HPLC Electrospray/Mass Spectrometry," conducted for 3M Environmental Technology and Safety Services, was performed in compliance with EPA TSCA Good Laboratory Practice Standards (40 CFR, Part 792) by Centre Analytical Laboratories, Inc. with the following exceptions:

1. The FOSA reference substance (analytical standard) used in this study has not been characterized under GLP's. §792.105(a).
2. Specimen (egg sample) identification for three samples was not according to 792.130(c) because the labels became removed from the containers.



Karen Smith  
Study Director  
Centre Analytical Laboratories, Inc.

06/21/01  
Date



Susan A. Beach  
Sponsor Representative  
3M Environmental Technology and Safety Services

6/22/01  
Date

**QUALITY ASSURANCE STATEMENT**

Centre Analytical Laboratories' Quality Assurance Unit reviewed Centre Study Number 023-057, entitled, "Analysis of PFOS, FOSA and PFOA From Various Food Matrices Using HPLC Electrospray/Mass Spectrometry". All phases were reviewed for conduct according to Centre Analytical Laboratories' Standard Operating Procedures, the Study Protocol, and all applicable Good Laboratory Practice Standards. All findings were reported to the Study Director and to management.

<u>Phase</u>	<u>Date Inspected</u>	<u>Date Reported to Study Director*</u>	<u>Date Reported to Centre Management</u>	<u>Date Reported to Sponsor Management</u>
1. Protocol Review	02/14/01	03/23/01	02/26/01	03/23/01
2. Extraction and Fortification	02/14/01	03/23/01	02/26/01	03/23/01
3. Raw Data and Draft Report Review	04/10-13/01	05/21/01	06/04/01	06/15/01
4. Raw Data and Draft Report Review	05/30/01	06/01/01	06/04/01	06/15/01
5. Final Report Review	06/19/01	06/20/01	06/21/01	06/21/01

\*The Study Director resided at the sponsor until 05/01/01 at which time a protocol amendment transferred Study Director duties to Centre Analytical Laboratories.

Naomi Lovallo  
 Senior Quality Assurance Auditor

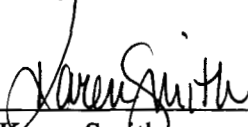
6/21/01  
 Date

**CERTIFICATION OF AUTHENTICITY**

This report, for Centre Study Number 023-057, is a true and complete representation of the raw data for the study.

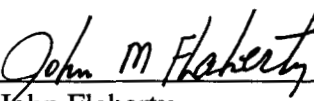
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Study Director, Centre:

  
\_\_\_\_\_  
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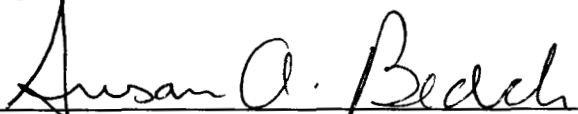
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Date

Sponsor Representative, 3M

  
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Date

**STUDY IDENTIFICATION**

Analysis of PFOS, FOSA and PFOA From Various Food Matrices Using HPLC  
Electrospray/Mass Spectrometry

CENTRE PROTOCOL NUMBER: 00P-023-057

TYPE OF STUDY: Residue

TEST SYSTEM: Green beans, apples, pork muscle, cow milk,  
chicken muscle, chicken eggs, bread, hot dogs,  
catfish and ground beef

TEST MATERIAL: PFOS, FOSA and PFOA

SPONSOR: 3M Environmental Technology and Safety Services  
Building 2-3E-09  
PO Box 33331  
St. Paul, MN 55133-3331

STUDY DIRECTOR: Karen Smith  
Centre Analytical Laboratories, Inc.  
Phone: (814) 231-8032

TESTING FACILITY: Centre Analytical Laboratories, Inc.  
3048 Research Drive  
State College, PA 16801

ANALYTICAL PHASE  
TIMETABLE: Study Initiation Date: 01/09/01  
Analytical Start Date: 01/11/01  
Analytical Termination Date: 05/22/01

## **PROJECT PERSONNEL**

The Study Director for this project at Centre Analytical Laboratories, Inc. was Karen Smith. The following personnel from Centre Analytical Laboratories, Inc., were associated with various phases of the study:

<u>Name</u>	<u>Title</u>
Karen Smith	Scientist
Emily Stauffer	Scientist
Angela Morgan	Technician
Tiffany Proctor	Technician
Rickey Keller	Sample Custodian
Lawrence Ord	Sample Custodian
Dave Bell	Scientist
Sharareh Zolghadr	Technician
Jason Farabaugh	Scientist
Mitra Arjmand	Technician
Yu Qiong Tan	Technician
Ling Ling Liu	Technician
Xiaoming Zhu	Technician

**TABLE OF CONTENTS**

	<u>Page</u>
TITLE PAGE .....	1
GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT .....	2
QUALITY ASSURANCE STATEMENT .....	3
CERTIFICATION OF AUTHENTICITY .....	4
STUDY IDENTIFICATION .....	5
PROJECT PERSONNEL .....	6
TABLE OF CONTENTS .....	7
LIST OF TABLES .....	8
LIST OF FIGURES .....	9
LIST OF APPENDICES .....	11
1.0 SUMMARY .....	12
2.0 OBJECTIVE.....	12
3.0 INTRODUCTION.....	12
4.0 TEST SYSTEM .....	13
5.0 REFERENCE MATERIAL .....	14
6.0 EXPERIMENTAL DESIGN.....	15
7.0 DESCRIPTION OF ANALYTICAL METHOD .....	15
7.1 Extraction Procedure .....	15
7.2 Preparation of Standards and Fortification Solutions .....	15
7.3 Chromatography .....	16
7.4 Instrument Sensitivity .....	17
7.5 Description of Instrument and Operating Conditions.....	17
7.6 Quantitation and Example Calculation .....	18
8.0 RESULTS AND DISCUSSION.....	20
9.0 CIRCUMSTANCES THAT MAY HAVE AFFECTED THE DATA.....	21
10.0 RETENTION OF DATA AND SAMPLES.....	21
11.0 TABLES.....	22
12.0 FIGURES .....	41
13.0 APPENDICES.....	65



**LIST OF TABLES**

	<u>Page</u>
Table I. Summary of PFOS, FOSA and PFOA Fortification Recoveries .....	23
Table II. Summary of PFOS, FOSA and PFOA Residues Found in Samples .....	26

## LIST OF FIGURES

	<u>Page</u>
Figure 1. Typical Calibration Curve for PFOS.....	42
Figure 2. Typical Calibration Curve for FOSA .....	43
Figure 3. Typical Calibration Curve for PFOA .....	44
Figure 4. Chromatogram Representing a 0.5 ng/mL mixed calibration standard of PFOS, FOSA and PFOA.....	45
Figure 5. Chromatogram Representing a 2.5 ng/mL mixed calibration standard of PFOS, FOSA and PFOA.....	46
Figure 6. Chromatogram Representing a Control Apple Sample (Centre ID: 0009347 Blank A1, Data Set: 020901C Apple).....	47
Figure 7. Chromatogram Representing a Control Apple Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009347 Spk A1, Data Set: 020901C Apple) .....	48
Figure 8. Chromatogram Representing an Apple Sample (Centre ID: 0012927, Sponsor ID: Publix, 18257, Data Set: 020901C Apple) .....	49
Figure 9. Chromatogram Representing a Control Green Bean Sample (Centre ID: 0009346 Blank A2, Data Set: 020601C Bean) .....	50
Figure 10. Chromatogram Representing a Control Green Bean Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009346 Spk A2, Data Set: 020601C Bean).....	51
Figure 11. Chromatogram Representing a Green Bean Sample (Centre ID: 0012910, Sponsor ID: Bruno's, 18341, Data Set: 020601C Bean).....	52
Figure 12. Chromatogram Representing a Control Pork Muscle Sample (Centre ID: 0009348 Blank A1, Data Set: 011501A Pork).....	53
Figure 13. Chromatogram Representing a Control Pork Muscle Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009348 Spk A1, Data Set: 011501A Pork) .....	54
Figure 14. Chromatogram Representing a Pork Muscle Sample (Centre ID: 0012795, Sponsor ID: Winn-Dixie, 18227, Data Set: 011501A Pork).....	55
Figure 15. Chromatogram Representing a Control Milk Sample (Centre ID: 0009349 Blank A1, Data Set: 020701A Milk) .....	56
Figure 16. Chromatogram Representing a Control Milk Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009349 Spk A1, Data Set: 020701A Milk).....	57

**LIST OF FIGURES (CONTINUED)**

Figure 17. Chromatogram Representing a Milk Sample (Centre ID: 0012871, Sponsor ID: Winn-Dixie, 18202, Data Set: 020701A Milk) ..... 58

Figure 18. Chromatogram Representing a Control Chicken Egg Sample (Centre ID: 0009342 Blank A3, Data Sets: 050901A Egg for PFOS, 012401A Egg for FOSA, and 052101A Egg for PFOA)..... 59

Figure 19. Chromatogram Representing a Control Chicken Egg Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009342 Spk A3, Data Sets: 050901A Egg for PFOS, 012401A Egg for FOSA, and 052101A Egg for PFOA) ..... 60

Figure 20. Chromatogram Representing a Chicken Egg Sample (Centre ID: 0012861, Sponsor ID: Save, 18167 and 21226, Data Sets: 050901A Egg for PFOS, 012401A Egg for FOSA, and 052101A Egg for PFOA) ..... 61

Figure 21. Chromatogram Representing a Control Bread Sample (Centre ID: 0009343 Blank A1, Data Set: 020901A Bread)..... 62

Figure 22. Chromatogram Representing a Control Bread Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009343 Spk A1, Data Set: 020901A Bread) ..... 63

Figure 23. Chromatogram Representing a Bread Sample (Centre ID: 0012887, Sponsor ID: Albertson's, 18244, Data Set: 020901A Bread) ..... 64

**LIST OF APPENDICES**

	<u>Page</u>
<b>Appendix A</b> Study Protocol 00P-023-057 (Centre Study No. 023-057), Amendments and Deviation .....	65

## **1.0 SUMMARY**

The purpose of this study was to analyze residues of PFOS, FOSA and PFOA in various food matrices as specified in Centre Protocol 00P-023-057. The analytical method used for this study was entitled, "Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples, Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hot Dogs, and Catfish by LC/MS/MS."

The limit of quantification for PFOS, FOSA and PFOA in food was 0.5 ppb (ng/g).

Average fortification recoveries and relative standard deviations were 101%  $\pm$  9.0% for PFOS, 96%  $\pm$  13% for FOSA and 104%  $\pm$  14% for PFOA.

Residues ranging from non-quantifiable levels to 0.852 ppb (ng/g) for PFOS, non-quantifiable levels for FOSA, and non-quantifiable to 2.35 ng/g for PFOA (one value of 14.7 ppb (ng/g) was found for PFOA but was considered suspect) were found in the food samples.

## **2.0 OBJECTIVE**

The objective of this study was to determine levels of PFOS, FOSA and PFOA in food samples collected from local grocery stores from six test cities outlined in 3M Quality Assurance Project Plan for Empirical Human Exposure Assessment Multi-City Study Sampling Task using the analytical method entitled "Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples, Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hot Dogs, and Catfish by LC/MS/MS."

## **3.0 INTRODUCTION**

This report details the results of the residues of PFOS, FOSA and PFOA detected in green beans, apples, pork muscle, cow milk, chicken muscle, chicken eggs, bread, hot dogs, catfish and ground beef, using the analytical method entitled, "Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples, Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hot Dogs, and Catfish by LC/MS/MS." Complete details of the analytical methodology can be found in **Appendix A**.

The study was initiated on January 09, 2001, when the study director signed the protocol 00P-023-057. The complete protocol and amendments can be found in **Appendix A**. The analytical start date was January 11, 2001, and the analytical termination date was May 22, 2001.

#### 4.0 TEST SYSTEM

The food samples analyzed in this study were received frozen from Pace Analytical on December 09, 2000 and stored frozen (<-10°C) upon receipt. Pace Analytical collected the samples for 3M Environmental Laboratory. The samples were then logged in on December 18, 2000 by Centre personnel and transferred to different frozen storage locale. All of the samples were processed according to the method, "Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples, Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hot Dogs, and Catfish by LC/MS/MS." Exact details of processing can be found on page 91 of this report. After processing, the samples were stored in a freezer at a temperature of -10°C or below until used and placed back into a freezer immediately after use.

At the request of the sponsor, the egg samples were returned frozen to 3M Environmental Laboratory on February 26, 2001 for possible reanalysis. The egg samples were returned unanalyzed from 3M Environmental Laboratory to Centre Analytical Laboratories, Inc. These samples were received frozen from 3M Environmental Laboratory on May 03, 2001 for PFOS and PFOA re-extraction and analysis. They were assigned different 3M and Centre ID's.

The food samples used for the controls and matrix spikes were the same samples used in the method validation study (Centre Study No. 023-043). They were purchased from Giant, Store #111 in State College, PA and assigned the following sample IDs upon log in at Centre:

<b>Centre Sample ID</b>	<b>Sample Description</b>
0009341	Chicken Muscle
0009342	Chicken Eggs
0009343	White Bread
0009344	Hot Dogs
0009345	Catfish
0009346	Green Beans
0009347	Apples
0009348	Pork Muscle
0009349	Whole Milk
0100006	Ground Beef

All of the control samples were purchased on September 27, 2000 except for the ground beef, which was purchased on January 03, 2001. The samples were stored in a refrigerator (4° ± 2°C) until processed. All of these control samples were processed with dry ice in a Hobart food chopper on October 02, 2000 except for the ground beef, which was processed on January 03, 2001. After processing, the samples were stored in a freezer at a temperature of -10°C or below until used and placed back into a freezer immediately after use.

Sample login and chain of custody information can be found in the raw data package associated with this study. Storage records will be kept at Centre Analytical Laboratories, Inc. and a true copy of the storage records can be found in the raw data package associated with this study.

## 5.0 REFERENCE MATERIAL

PFOS was received at Centre on June 05, 2000, FOSA was received on February 15, 2000 and PFOA was received on July 06, 2000 from 3M Environmental Technology and Services. Characterization of the reference materials PFOS and PFOA was performed at Centre on August 31, 2000 and November 06, 2000, respectively, and documentation can be found in raw data package associated with this report. The characterization of FOSA is in progress and the necessary information will be added upon complete characterization of the reference materials. All analyses were completed prior to standard expiration dates.

The available information for the reference material is listed below. FOSA was stored at room temperature and PFOS and PFOA were stored frozen at  $\leq -10^{\circ}\text{C}$ .

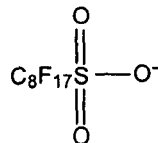
<u>Compound</u>	<u>TCR Substance No.</u>	<u>Batch No.</u>	<u>Purity (%)</u>	<u>Expiration Date</u>
PFOS	TCR00017-46	NA	97.9	08/31/01
FOSA	SD-029	L-15709	95.1	02/15/01
PFOA	TCR99030-30	332	95.0	10/31/01

Molecular structures of PFOS, FOSA and PFOA are given below.

### PFOS

Chemical Name = Perfluorooctane sulfonate

Molecular weight = 499 ( $\text{C}_8\text{F}_{17}\text{SO}_3^-$ )

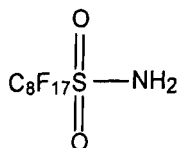


**Note:** The neutral molecule and standard form which PFOS (anion) is derived from is potassium perfluorooctane sulfonate [ $\text{C}_8\text{F}_{17}\text{SO}_3\text{K}$ ], molecular weight 538.

### FOSA

Chemical Name = Perfluorooctane sulfonylamide

Molecular weight = 499



**PFOA**

Chemical Name = Perfluorooctanoate

Molecular weight = 413

C<sub>7</sub>F<sub>15</sub>COO<sup>-</sup>

**Note:** The neutral molecule and standard form which PFOA (anion) is derived from is ammonium perfluorooctanoate [C<sub>7</sub>F<sub>15</sub>COONH<sub>4</sub>], molecular weight 431.

## **6.0 EXPERIMENTAL DESIGN**

Each sample was extracted in duplicate. A set contained one matrix blank and two matrix blanks spiked with known amounts of PFOS, FOSA and PFOA and 6 – 10 samples. The re-extracted egg sets also included one reagent blank.

The extracts were analyzed by LC/MS/MS. Samples with residues below the linear range of the calibration curve were not quantified; however, residues in the control samples were calculated up to 20% of the LOQ by extrapolation of the calibration curve, in order to correct fortification recoveries.

## **7.0 DESCRIPTION OF ANALYTICAL METHOD**

Analytical method entitled “Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples, Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hot Dogs, and Catfish by LC/MS/MS” was used for this study.

### **7.1 Extraction Procedure**

Five grams of processed sample were weighed into 50 mL polypropylene centrifuge tubes and fortified (if necessary). The samples were extracted with ACN followed by a SPE column (florisil, silica gel, carbon and LC-NH<sub>2</sub>) clean up. The column eluate was collected and concentrated to near dryness. Final volume was adjusted to 2 mL with 2% ascorbic acid in methanol. The samples were analyzed using electrospray LC/MS/MS.

### **7.2 Preparation of Standards and Fortification Solutions**

Standard stock solutions of PFOS, PFOA and FOSA were prepared on September 18, 2000, September 26, 2000 and October 03, 2000, respectively. Fortification and calibration standard solutions were prepared on October 17, 2000 as specified in Centre Analytical Laboratories’ analytical method entitled, “Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples, Pork Muscle, Cow Milk,



Chicken Muscle, Chicken Eggs, Bread, Hotdogs, and Catfish by LC/MS/MS.” Individual stock standard solutions of PFOS, FOSA and PFOA were prepared at concentrations of 100 µg/mL by taking 10 mg of the standard (corrected for purity and salt content where applicable) and bringing it up to 100 mL with methanol. From these solutions, a 1 µg/mL mixed fortification standard solution was prepared by taking 1 mL of each stock and bringing the volume up to 100 mL with methanol.

The 1 µg/mL mixed fortification standard was diluted ten-fold with methanol to make the 0.1 µg/mL mixed fortification standard. Then, the 0.1 µg/mL mixed standard was diluted ten-fold with methanol to make the 0.01 µg/mL fortification standard.

LC/MS/MS calibration standards were prepared in methanol at the following concentrations 50, 25, 10, 5.0, 2.5, 1.0, and 0.5 ng/mL according to the following table:

Initial Concentration (ng/mL)	Volume (mL)	Diluted to (mL)	Final concentration (ng/mL)
1000	5.0	100	50
1000	2.5	100	25
1000	1.0	100	10
50	10.0	100	5.0
25	10.0	100	2.5
10	10.0	100	1.0
5.0	10.0	100	0.5

New standard stock, fortification and calibration solutions were made for PFOS and PFOA in order to perform the egg re-extraction and analysis. The PFOS standard stock solution was prepared on March 14, 2001 and the PFOA standard stock solution was prepared on April 11, 2001. Mixed fortification and calibration solutions of PFOS and PFOA were prepared from the stock solutions on May 03, 2001 as described above. The only modification from the above procedure is that FOSA was omitted from the mix since the re-extraction and analysis was for PFOS and PFOA only.

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

### 7.3 Chromatography

Quantification of PFOS, FOSA and PFOA was accomplished by LC/MS/MS analysis using electrospray LC/MS/MS.

The retention times of PFOS, FOSA and PFOA were ~ 5.4 min, ~ 6.0 min and ~ 5.3 min., respectively. The matrices did not contribute any interfering peaks corresponding to the

analyte retention times; however, there were some occasions where the control matrices appeared to contain analyte.

#### 7.4 Instrument Sensitivity

The smallest standard amount injected during the chromatographic run was 0.5 ng/mL which is equivalent to 0.2 ng/g of PFOS, FOSA and PFOA in matrix.

#### 7.5 Description of Instrument and Operating Conditions

A Micromass Quattro Ultima LC/MS/MS coupled to a Hewlett Packard HPLC system was used. Data acquisition and processing were performed using Masslynx 3.4 software. Detailed operating conditions are listed below:

Instrument: Micromass Quattro Ultima

##### ELECTROSPRAY ION SOURCE:

Capillary: 2.0 kV	Hexapole 2: 0 V
Hexapole 1: 0 V	Source Block Temp.: 100°C
Aperture 1: 0 V	Desolvation Temp.: 400°C

##### ANALYZER:

LM Res 1: 10.5 V	LM Res 2: 12.0 V
HM Res 1: 10.5 V	HM Res 2: 12.0 V
IEnergy 1: 1.0 V	IEnergy 2: 2.0 V
Entrance: -2 V	Multiplier: 650 V
Exit: 2 V	

##### GAS FLOWS AND PRESSURE:

Desolvation N<sub>2</sub> Flow Rate: ~700 L/hr  
Nebuliser N<sub>2</sub> Flow Rate: ~150 L/hr  
Gas Cell Pressure: ~0.0031 mbar

Computer: COMPAQ Professional Workstation AP200

Software: Microsoft Windows NT: Version 4 Build 1381: Service Pack 5  
Micromass Limited: Masslynx 3.4 Build 004

HPLC Equipment: Hewlett Packard (HP) Series 1100  
HP Binary Pump  
HP Autosampler  
HP Vacuum Degasser  
HP Column Oven

HPLC Column: Genesis C-8, 5 cm x 2.1 mm i.d. x 4 μ

Column Temperature: 35°C

Mobile Phase (A) : 2 mM Ammonium Acetate in Type I Water

Mobile Phase (B) : Methanol

Time (min)	% A	% B	Flow Rate (mL/min)
0.0	60.0	40.0	0.3
0.4	60.0	40.0	0.3
1.0	10.0	90.0	0.3
7.0	10.0	90.0	0.3
7.5	0.0	100.0	0.3
9.0	0.0	100.0	0.4
9.5	60.0	40.0	0.4
13.5	60.0	40.0	0.4
14.0	60.0	40.0	0.3

Total run time = 14 min

Injected Volume: 15 µL

Ions monitored :

Analyte	Parent ion	Daughter ion	Dwell (secs)	Coll Energy (eV)	Cone (V)
PFOA	413	369	0.2	10	30
FOSA	498	78	0.2	40	30
PFOS	499	99	0.2	50	30

## 7.6 Quantitation and Example Calculation

Fifteen microliters of sample or calibration standard was injected into the LC/MS/MS. The peak area was measured and the standard curve was generated (using 1/x weighted linear regression) by Masslynx software using seven concentrations of standards. The residue concentration in food samples was determined using the following equations:

Equation 1 was used to calculate the amount of analyte found (in ng/mL, based on peak area) using the standard curve (linear regression parameters) generated by the Masslynx software program.

Equation 1:

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

The component residue concentration (in ng/g) in matrix was determined by using Equation 2.

Equation 2:

Analyte found (ng/g) =

$$\frac{[\text{analyte found (ng/mL)} - \text{average analyte found in blanks (ng/mL)}] \times \text{DF} \times \text{FV (mL)}}{\text{sample weight (g)}}$$

where DF = dilution factor and FV = final volume

Note: the process of subtracting the average analyte found in the blanks was done only for control matrix spikes or if a reagent blank contained residue.

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

Equation 3:

$$\text{Recovery (\%)} = \frac{\text{analyte found (ng/g)}}{\text{analyte added (ng/g)}} \times 100$$

An example of a calculation using an actual sample follows:

Pork sample Centre ID 0009348 Spk A1 (Set No.: 011501A Pork), fortified at 2.5 ng/g of PFOS, FOSA and PFOA: (Calculation is using values from FOSA)

Where:

peak area	=	149454
intercept	=	2524.48
slope	=	25194.9
final volume	=	2 mL
dilution factor	=	1
sample weight	=	5 g
ng/g added (fort level)	=	2.5 ng/g
amt found in control	=	0 (NQ = Not quantifiable; assumed to be 0)

**From equation 1:**

$$\begin{aligned} \text{Analyte found (ng/mL)} &= \frac{[149484 - 2524.48]}{25194.9} \\ &= 5.83 \text{ ng/mL} \end{aligned}$$

**From equation 2:**

$$\begin{aligned}\text{Analyte found (ng/g)} &= \frac{(5.832 - 0) \times 1 \times 2}{5.0} \\ &= 2.33 \text{ ng/g}\end{aligned}$$

**From equation 3:**

$$\begin{aligned}\% \text{ Recovery} &= \frac{2.33}{2.5} \times 100 \\ &= 93\%\end{aligned}$$

Note: This example calculation was done using rounded numbers, and therefore may be slightly different from the values shown in the RAW DATA.

Any residues found for PFOA were adjusted for PFOA purity (95%). This was done by taking the residue found (ng/g) for PFOA and multiplying by the purity (0.95).

Other statistical methods used in analyzing this data were:

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \qquad \text{Mean} = \bar{x} = \frac{\sum_{i=1}^n x_i}{n}$$

*Relative Standard Deviation (RSD or Coefficient of Variation (CV)) =*

$$\frac{\text{Standard Deviation} \times 100\%}{\text{Mean}}$$

## **8.0 RESULTS AND DISCUSSION**

Average fortification recoveries and relative standard deviations were 101% ± 9.0% for PFOS, 96% ± 13% for FOSA and 104% ± 14% for PFOA. A summary of all of the fortification recoveries can be found in **Table I**.

Residues ranging from non-quantifiable levels to 0.852 ppb (ng/g) for PFOS, non-quantifiable levels for FOSA, and non-quantifiable to 2.35 ng/g for PFOA (one value of 14.7 ppb (ng/g) was found for PFOA but was considered suspect) were found in the food samples. The residues found in all of the samples are detailed in **Table II**.

Typical calibration curves and chromatograms representing standards, controls, fortifications, and samples are depicted in **Figures 1-23**.

In this study, analytes were not quantified below 0.5 ng/g. If lower detection levels are required, a more definitive study, including extraction blanks in addition to matrix blanks, could be done.

Initially the PFOA standard and fortification solutions were not corrected for purity, as purity information had not been received at the time. Purity data was received after residue analyses were completed and the residues found were then corrected for PFOA purity. All ng/g residues of PFOA in this report have been adjusted to reflect the purity of the PFOA used in this study.

There was some PFOA contamination from the lab while performing the extraction for eggs. It was believed to be coming from the extraction solvent itself. Using a different brand and lot number of ACN alleviated the problem because upon reanalysis, there were no quantifiable residues of PFOA in the egg samples.

## **9.0 CIRCUMSTANCES THAT MAY HAVE AFFECTED THE DATA**

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

## **10.0 RETENTION OF DATA AND SAMPLES**

When the final report is complete, all original paper data generated by Centre Analytical Laboratories, Inc. will be shipped to the sponsor. This does not include facility-specific raw data such as instrument logs, however exact copies of temperature logs will be submitted. Exact copies of all raw data, as well as a signed copy of the final analytical report and all original facility-specific raw data, will be retained in the Centre Analytical Laboratories, Inc. archives for the period of time specified in 40 CFR 792.195 (b). Retained samples of reference substances are archived by the sponsor.

# **11.0 TABLES**

**Table I. Summary of PFOS, FOSA and PFOA Fortification Recoveries**

Centre ID	Sample Description	Extraction Date	Analysis Date	Fort Level (ng/g)	Recovery (%)		
					PFOS	FOSA	PFOA
0009341 Spk A1	Chicken	01/11/01	01/11-12/01	2.5	104	93	94
0009341 Spk B1	Chicken	01/11/01	01/11-12/01	10	89	84	87
0009341 Spk A2	Chicken	01/11/01	01/12/01	2.5	110	94	98
0009341 Spk B2	Chicken	01/11/01	01/12/01	10	91	89	91
0009341 Spk A3	Chicken	01/12/01	01/15/01	2.5	108	107	106
0009341 Spk B3	Chicken	01/12/01	01/15/01	10	106	105	111
0009341 Spk A4	Chicken	01/12/01	01/15/01	2.5	106	105	107
0009341 Spk B4	Chicken	01/12/01	01/15/01	10	97	90	98
0009348 Spk A1	Pork	01/15/01	01/15-16/01	2.5	105	93	116
0009348 Spk B1	Pork	01/15/01	01/15-16/01	10	99	86	110
0009348 Spk A2	Pork	01/15/01	01/16/01	2.5	98	87	105
0009348 Spk B2	Pork	01/15/01	01/16/01	10	84	76	95
0009348 Spk A3	Pork	01/16/01	01/16/01	2.5	98	93	106
0009348 Spk B3	Pork	01/16/01	01/16/01	10	81	78	91
0009348 Spk A4	Pork	01/16/01	01/16-17/01	2.5	90	86	93
0009348 Spk B4	Pork	01/16/01	01/16-17/01	10	82	79	92
0009344 Spk A1	Hot Dog	01/17/01	01/17-18/01	2.5	102	100	*
0009344 Spk B1	Hot Dog	01/17/01	01/17-18/01	10	95	92	*
0009344 Spk A1	Hot Dog	01/17/01	01/18/01	2.5	-	-	97
0009344 Spk B1	Hot Dog	01/17/01	01/18/01	10	-	-	95
0009344 Spk A2	Hot Dog	01/17/01	01/18/01	2.5	113	106	114
0009344 Spk B2	Hot Dog	01/17/01	01/18/01	10	95	91	87
0009344 Spk A3	Hot Dog	01/18/01	01/18-19/01	2.5	99	94	102
0009344 Spk B3	Hot Dog	01/18/01	01/18-19/01	10	89	77	89
0009344 Spk A4	Hot Dog	01/18/01	01/19/01	2.5	104	95	96
0009344 Spk B4	Hot Dog	01/18/01	01/19/01	10	91	84	84
0009345 Spk A1	Fish	01/19/01	01/19-20/01	2.5	100	68	105
0009345 Spk B1	Fish	01/19/01	01/19-20/01	10	87	56	87
0009345 Spk A2	Fish	01/19/01	01/20/01	2.5	86	*	85
0009345 Spk B2	Fish	01/19/01	01/20/01	10	86	*	86
0009345 Spk A2	Fish	01/30/01	01/31/01	2.5	-	126	-
0009345 Spk B2	Fish	01/30/01	01/31/01	10	-	110	-
0009345 Spk A3	Fish	01/22/01	01/22-23/01	2.5	111	76	111
0009345 Spk B3	Fish	01/22/01	01/22-23/01	10	95	65	101
0009345 Spk A4	Fish	01/22/01	01/23/01	2.5	106	73	103
0009345 Spk B4	Fish	01/22/01	01/23/01	10	89	62	89

\* = Rejected due to an unacceptable recovery. Replaced with re-extracted data.

- = Not Analyzed For



**Table I continued: Summary of PFOS, FOSA and PFOA Fortification Recoveries**

Centre ID	Sample Description	Extraction	Analysis	Fort Level (ng/g)	Recovery (%)		
		Date	Date		PFOS	FOSA	PFOA
0009342 Spk A1	Egg	01/23/01	01/23-24/01	2.5	104	96	84
0009342 Spk B1	Egg	01/23/01	01/23-24/01	10	98	80	67
0009342 Spk A2	Egg	01/23/01	01/24/01	2.5	117	106	78
0009342 Spk B2	Egg	01/23/01	01/24/01	10	101	98	77
0009342 Spk A3	Egg	01/24/01	01/24-25/01	2.5	115	109	105
0009342 Spk B3	Egg	01/24/01	01/24-25/01	10	92	90	85
0009342 Spk A4	Egg	01/24/01	01/25/01	2.5	117	111	90
0009342 Spk B4	Egg	01/24/01	01/25/01	10	102	97	82
0009346 Spk A1	Green Bean	02/05/01	02/05/01	2.5	98	114	129
0009346 Spk B1	Green Bean	02/05/01	02/05/01	10	106	106	128
0009346 Spk A2	Green Bean	02/06/01	02/07/01	2.5	92	99	124
0009346 Spk B2	Green Bean	02/06/01	02/07/01	10	111	103	130
0009346 Spk A3	Green Bean	02/08/01	02/09/01	2.5	99	100	125
0009346 Spk B3	Green Bean	02/08/01	02/09/01	10	94	97	125
0009346 Spk A4	Green Bean	02/08/01	02/12/01	2.5	83	85	103
0009346 Spk B4	Green Bean	02/08/01	02/12/01	10	96	95	112
0009349 Spk A1	Milk	02/07/01	02/09-10/01	2.5	99	103	129
0009349 Spk B1	Milk	02/07/01	02/09-10/01	10	91	90	128
0009349 Spk A2	Milk	02/07/01	02/10/01	2.5	114	101	123
0009349 Spk B2	Milk	02/07/01	02/10/01	10	90	91	123
0009349 Spk A3	Milk	02/08/01	02/10/01	2.5	101	103	126
0009349 Spk B3	Milk	02/08/01	02/10/01	10	89	91	129
0009349 Spk A4	Milk	02/08/01	02/10-11/01	2.5	113	103	124
0009349 Spk B4	Milk	02/08/01	02/10-11/01	10	93	94	123
0009347 Spk A1	Apple	02/09/01	02/12/01	2.5	113	110	122
0009347 Spk B1	Apple	02/09/01	02/12/01	10	105	104	119
0009347 Spk A2	Apple	02/12/01	02/13/01	2.5	111	107	108
0009347 Spk B2	Apple	02/12/01	02/13/01	10	100	102	101
0009347 Spk A3	Apple	02/12/01	02/13-14/01	2.5	109	105	110
0009347 Spk B3	Apple	02/12/01	02/13-14/01	10	98	96	100
0009347 Spk A4	Apple	02/12/01	02/14/01	2.5	108	101	102
0009347 Spk B4	Apple	02/12/01	02/14/01	10	99	96	99

**Table I continued: Summary of PFOS, FOSA and PFOA Fortification Recoveries**

Centre ID	Sample Description	Extraction	Analysis	Fort Level (ng/g)	Recovery (%)		
		Date	Date		PFOS	FOSA	PFOA
0009343 Spk A1	Bread	02/08-09/01	02/11/01	2.5	87	106	118
0009343 Spk B1	Bread	02/08-09/01	02/11/01	10	96	96	109
0009343 Spk A2	Bread	02/08-09/01	02/11/01	2.5	106	101	119
0009343 Spk B2	Bread	02/08-09/01	02/11/01	10	94	93	106
0009343 Spk A3	Bread	02/12-13/01	02/14/01	2.5	106	103	103
0009343 Spk B3	Bread	02/12-13/01	02/14/01	10	99	92	97
0009343 Spk A4	Bread	02/12-13/01	02/14/01	2.5	111	109	106
0009343 Spk B4	Bread	02/12-13/01	02/14/01	10	102	100	100
0100006 Spk A1	Ground Beef	02/13/01	02/14-15/01	2.5	104	104	100
0100006 Spk B1	Ground Beef	02/13/01	02/14-15/01	10	103	99	95
0100006 Spk A2	Ground Beef	02/13/01	02/15/01	2.5	111	102	103
0100006 Spk B2	Ground Beef	02/13/01	02/15/01	10	103	98	98
0100006 Spk A3	Ground Beef	02/14/01	02/15/01	2.5	121	123	120
0100006 Spk B3	Ground Beef	02/14/01	02/15/01	10	106	116	114
0100006 Spk A4	Ground Beef	02/14/01	02/15-16/01	2.5	112	108	105
0100006 Spk B4	Ground Beef	02/14/01	02/15-16/01	10	102	101	102
0009342 Spk A1	Egg	05/07-08/01	05/08/01	2.5	111	-	-
0009342 Spk B1	Egg	05/07-08/01	05/08/01	10	99	-	-
0009342 Spk A2	Egg	05/07-08/01	05/08-09/01	2.5	111	-	-
0009342 Spk B2	Egg	05/07-08/01	05/08-09/01	10	102	-	-
0009342 Spk A3	Egg	05/08-09/01	05/09/01	2.5	106	-	-
0009342 Spk B3	Egg	05/08-09/01	05/09/01	10	94	-	-
0009342 Spk A4	Egg	05/08-09/01	05/09-10/01	2.5	109	-	-
0009342 Spk B4	Egg	05/08-09/01	05/09-10/01	10	102	-	-
0009342 Spk A1	Egg	05/18/01	05/18/01	2.5	-	-	110
0009342 Spk B1	Egg	05/18/01	05/18/01	10	-	-	99
0009342 Spk A2	Egg	05/18/01	05/18-19/01	2.5	-	-	102
0009342 Spk B2	Egg	05/18/01	05/18-19/01	10	-	-	108
0009342 Spk A3	Egg	05/20-21/01	05/21/01	2.5	-	-	96
0009342 Spk B3	Egg	05/20-21/01	05/21/01	10	-	-	96
0009342 Spk A4	Egg	05/20-21/01	05/21-22/01	2.5	-	-	95
0009342 Spk B4	Egg	05/20-21/01	05/21-22/01	10	-	-	96
<b>AVERAGE:</b>					<b>101</b>	<b>96</b>	<b>104</b>
<b>STD DEV:</b>					<b>9.0</b>	<b>13</b>	<b>14</b>
<b>RELATIVE STD DEV:</b>					<b>9.0</b>	<b>13</b>	<b>14</b>
<b>n:</b>					<b>88</b>	<b>80</b>	<b>88</b>

- = Not Analyzed For

**Table II. Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*			
						PFOS	FOSA	PFOA	
0012776	011101A	Chicken	18249	Publix	Port St. Lucie, FL	Chicken	NQ	NQ	NQ
0012776 Dup	011101A	Chicken	18249	Publix	Port St. Lucie, FL	Chicken	NQ	NQ	NQ
0012777	011101A	Chicken	18226	Winn-Dixie	Port St. Lucie, FL	Chicken	NQ	NQ	NQ
0012777 Dup	011101A	Chicken	18226	Winn-Dixie	Port St. Lucie, FL	Chicken	NQ	NQ	NQ
0012778	011101A	Chicken	18239	Albertson's	Port St. Lucie, FL	Chicken	NQ	NQ	NQ
0012778 Dup	011101A	Chicken	18239	Albertson's	Port St. Lucie, FL	Chicken	NQ	NQ	NQ
0012779	011101A	Chicken	18356	Delchamps II	Mobile, AL	Chicken	NQ	NQ	NQ
0012779 Dup	011101A	Chicken	18356	Delchamps II	Mobile, AL	Chicken	NQ	NQ	NQ
0012780	011101A	Chicken	18197	Winn-Dixie	Columbus, GA	Chicken	NQ	NQ	NQ
0012780 Dup	011101A	Chicken	18197	Winn-Dixie	Columbus, GA	Chicken	NQ	NQ	NQ
0012781	011101B	Chicken	18345	Delchamps I	Mobile, AL	Chicken	NQ	NQ	NQ
0012781 Dup	011101B	Chicken	18345	Delchamps I	Mobile, AL	Chicken	NQ	NQ	NQ
0012782	011101B	Chicken	18209	Piggly Wiggly	Columbus, GA	Chicken	NQ	NQ	NQ
0012782 Dup	011101B	Chicken	18209	Piggly Wiggly	Columbus, GA	Chicken	NQ	NQ	NQ
0012783	011101B	Chicken	18335	Bruno's	Mobile, AL	Chicken	NQ	NQ	NQ
0012783 Dup	011101B	Chicken	18335	Bruno's	Mobile, AL	Chicken	NQ	NQ	NQ
0012784	011101B	Chicken	18323	Kroger's	Decatur, AL	Chicken	NQ	NQ	NQ
0012784 Dup	011101B	Chicken	18323	Kroger's	Decatur, AL	Chicken	NQ	NQ	NQ
0012785	011101B	Chicken	18312	Winn-Dixie	Decatur, AL	Chicken	NQ	NQ	NQ
0012785 Dup	011101B	Chicken	18312	Winn-Dixie	Decatur, AL	Chicken	NQ	NQ	NQ
0012786	011201A	Chicken	18301	Haloway	Decatur, AL	Chicken	NQ	NQ	NQ
0012786 Dup	011201A	Chicken	18301	Haloway	Decatur, AL	Chicken	NQ	NQ	NQ
0012787	011201A	Chicken	18187	Cub	Columbus, GA	Chicken	NQ	NQ	NQ
0012787 Dup	011201A	Chicken	18187	Cub	Columbus, GA	Chicken	NQ	NQ	NQ
0012788	011201A	Chicken	18174	Bi-Lo	Cleveland, TN	Chicken	NQ	NQ	NQ
0012788 Dup	011201A	Chicken	18174	Bi-Lo	Cleveland, TN	Chicken	NQ	NQ	NQ
0012789	011201A	Chicken	18163	Save-a-Lot	Cleveland, TN	Chicken	NQ	NQ	NQ
0012789 Dup	011201A	Chicken	18163	Save-a-Lot	Cleveland, TN	Chicken	NQ	NQ	NQ
0012790	011201A	Chicken	18283	Albertson's	Pensacola, FL	Chicken	NQ	NQ	NQ
0012790 Dup	011201A	Chicken	18283	Albertson's	Pensacola, FL	Chicken	NQ	NQ	NQ
0012791	011201B	Chicken	18260	Food World	Pensacola, FL	Chicken	NQ	NQ	NQ
0012791 Dup	011201B	Chicken	18260	Food World	Pensacola, FL	Chicken	NQ	NQ	NQ

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012792	011201B	18151	Watson's	Cleveland, TN	Chicken	NQ	NQ	NQ
0012792 Dup	011201B	18151	Watson's	Cleveland, TN	Chicken	NQ	NQ	NQ
0012793	011201B	18272	Food World II	Pensacola, FL	Chicken	NQ	NQ	NQ
0012793 Dup	011201B	18272	Food World II	Pensacola, FL	Chicken	NQ	NQ	NQ
0012794	011501A	18250	Publix	Port St. Lucie, FL	Pork	NQ	NQ	NQ
0012794 Dup	011501A	18250	Publix	Port St. Lucie, FL	Pork	NQ	NQ	NQ
0012795	011501A	18227	Winn-Dixie	Port St. Lucie, FL	Pork	NQ	NQ	NQ
0012795 Dup	011501A	18227	Winn-Dixie	Port St. Lucie, FL	Pork	NQ	NQ	NQ
0012796	011501A	18238	Albertson's	Port St. Lucie, FL	Pork	NQ	NQ	NQ
0012796 Dup	011501A	18238	Albertson's	Port St. Lucie, FL	Pork	NQ	NQ	NQ
0012797	011501A	18357	Delchamps II	Mobile, AL	Pork	NQ	NQ	NQ
0012797 Dup	011501A	18357	Delchamps II	Mobile, AL	Pork	NQ	NQ	NQ
0012798	011501A	18198	Winn-Dixie	Columbus, GA	Pork	NQ	NQ	NQ
0012798 Dup	011501A	18198	Winn-Dixie	Columbus, GA	Pork	NQ	NQ	NQ
0012799	011501B	18346	Delchamps I	Mobile, AL	Pork	NQ	NQ	NQ
0012799 Dup	011501B	18346	Delchamps I	Mobile, AL	Pork	NQ	NQ	NQ
0012800	011501B	18210	Piggly Wiggly	Columbus, GA	Pork	NQ	NQ	NQ
0012800 Dup	011501B	18210	Piggly Wiggly	Columbus, GA	Pork	NQ	NQ	NQ
0012801	011501B	18334	Bruno's	Mobile, AL	Pork	NQ	NQ	NQ
0012801 Dup	011501B	18334	Bruno's	Mobile, AL	Pork	NQ	NQ	NQ
0012802	011501B	18322	Kroger's	Decatur, AL	Pork	NQ	NQ	NQ
0012802 Dup	011501B	18322	Kroger's	Decatur, AL	Pork	NQ	NQ	NQ
0012803	011501B	18311	Winn-Dixie	Decatur, AL	Pork	NQ	NQ	NQ
0012803 Dup	011501B	18311	Winn-Dixie	Decatur, AL	Pork	NQ	NQ	NQ
0012804	011601A	18299	Haloway	Decatur, AL	Pork	NQ	NQ	NQ
0012804 Dup	011601A	18299	Haloway	Decatur, AL	Pork	NQ	NQ	NQ
0012805	011601A	18186	Cub	Columbus, GA	Pork	NQ	NQ	NQ
0012805 Dup	011601A	18186	Cub	Columbus, GA	Pork	NQ	NQ	NQ
0012806	011601A	18175	Bi-Lo	Cleveland, TN	Pork	NQ	NQ	NQ
0012806 Dup	011601A	18175	Bi-Lo	Cleveland, TN	Pork	NQ	NQ	NQ
0012807	011601A	18164	Save-a-Lot	Cleveland, TN	Pork	NQ	NQ	NQ
0012807 Dup	011601A	18164	Save-a-Lot	Cleveland, TN	Pork	NQ	NQ	NQ

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012808	011601A	18282	Albertson's	Pensacola, FL	Pork	NQ	NQ	NQ
0012808 Dup	011601A	18282	Albertson's	Pensacola, FL	Pork	NQ	NQ	NQ
0012809	011601B	18261	Food World	Pensacola, FL	Pork	NQ	NQ	NQ
0012809 Dup	011601B	18261	Food World	Pensacola, FL	Pork	NQ	NQ	NQ
0012810	011601B	18152	Watson's	Cleveland, TN	Pork	NQ	NQ	NQ
0012810 Dup	011601B	18152	Watson's	Cleveland, TN	Pork	NQ	NQ	NQ
0012811	011601B	18271	Food World II	Pensacola, FL	Pork	NQ	NQ	NQ
0012811 Dup	011601B	18271	Food World II	Pensacola, FL	Pork	NQ	NQ	NQ
0012812	011701A	18251	Publix	Port St. Lucie, FL	Hot Dog	NQ	NQ	**
0012812 Dup	011701A	18251	Publix	Port St. Lucie, FL	Hot Dog	NQ	NQ	**
0012813	011701A	18228	Winn-Dixie	Port St. Lucie, FL	Hot Dog	NQ	NQ	**
0012813 Dup	011701A	18228	Winn-Dixie	Port St. Lucie, FL	Hot Dog	NQ	NQ	**
0012814	011701A	18240	Albertson's	Port St. Lucie, FL	Hot Dog	NQ	NQ	**
0012814 Dup	011701A	18240	Albertson's	Port St. Lucie, FL	Hot Dog	NQ	NQ	**
0012815	011701A	18358	Delchamps II	Mobile, AL	Hot Dog	NQ	NQ	**
0012815 Dup	011701A	18358	Delchamps II	Mobile, AL	Hot Dog	NQ	NQ	**
0012816	011701A	18199	Winn-Dixie	Columbus, GA	Hot Dog	NQ	NQ	**
0012816 Dup	011701A	18199	Winn-Dixie	Columbus, GA	Hot Dog	NQ	NQ	**
0012812	011701AR	18251	Publix	Port St. Lucie, FL	Hot Dog	-	-	NQ
0012812 Dup	011701AR	18251	Publix	Port St. Lucie, FL	Hot Dog	-	-	NQ
0012813	011701AR	18228	Winn-Dixie	Port St. Lucie, FL	Hot Dog	-	-	NQ
0012813 Dup	011701AR	18228	Winn-Dixie	Port St. Lucie, FL	Hot Dog	-	-	NQ
0012814	011701AR	18240	Albertson's	Port St. Lucie, FL	Hot Dog	-	-	NQ
0012814 Dup	011701AR	18240	Albertson's	Port St. Lucie, FL	Hot Dog	-	-	NQ
0012815	011701AR	18358	Delchamps II	Mobile, AL	Hot Dog	-	-	NQ
0012815 Dup	011701AR	18358	Delchamps II	Mobile, AL	Hot Dog	-	-	NQ
0012816	011701AR	18199	Winn-Dixie	Columbus, GA	Hot Dog	-	-	NQ
0012816 Dup	011701AR	18199	Winn-Dixie	Columbus, GA	Hot Dog	-	-	NQ
0012817	011701B	18347	Delchamps I	Mobile, AL	Hot Dog	NQ	NQ	NQ
0012817 Dup	011701B	18347	Delchamps I	Mobile, AL	Hot Dog	NQ	NQ	NQ
0012818	011701B	18211	Piggly Wiggly	Columbus, GA	Hot Dog	NQ	NQ	NQ
0012818 Dup	011701B	18211	Piggly Wiggly	Columbus, GA	Hot Dog	NQ	NQ	NQ

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

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\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012819	011701B Hot Dog	18336	Bruno's	Mobile, AL	Hot Dog	NQ	NQ	NQ
0012819 Dup	011701B Hot Dog	18336	Bruno's	Mobile, AL	Hot Dog	NQ	NQ	NQ
0012820	011701B Hot Dog	18324	Kroger's	Decatur, AL	Hot Dog	NQ	NQ	NQ
0012820 Dup	011701B Hot Dog	18324	Kroger's	Decatur, AL	Hot Dog	NQ	NQ	NQ
0012821	011701B Hot Dog	18313	Winn-Dixie	Decatur, AL	Hot Dog	NQ	NQ	NQ
0012821 Dup	011701B Hot Dog	18313	Winn-Dixie	Decatur, AL	Hot Dog	NQ	NQ	NQ
0012822	011801A Hot Dog	18302	Haloway	Decatur, AL	Hot Dog	NQ	NQ	NQ
0012822 Dup	011801A Hot Dog	18302	Haloway	Decatur, AL	Hot Dog	NQ	NQ	NQ
0012823	011801A Hot Dog	18188	Cub	Columbus, GA	Hot Dog	NQ	NQ	NQ
0012823 Dup	011801A Hot Dog	18188	Cub	Columbus, GA	Hot Dog	NQ	NQ	NQ
0012824	011801A Hot Dog	18176	Bi-Lo	Cleveland, TN	Hot Dog	NQ	NQ	NQ
0012824 Dup	011801A Hot Dog	18176	Bi-Lo	Cleveland, TN	Hot Dog	NQ	NQ	NQ
0012825	011801A Hot Dog	18165	Save-a-Lot	Cleveland, TN	Hot Dog	NQ	NQ	NQ
0012825 Dup	011801A Hot Dog	18165	Save-a-Lot	Cleveland, TN	Hot Dog	NQ	NQ	NQ
0012826	011801A Hot Dog	18285	Albertson's	Pensacola, FL	Hot Dog	NQ	NQ	NQ
0012826 Dup	011801A Hot Dog	18285	Albertson's	Pensacola, FL	Hot Dog	NQ	NQ	NQ
0012827	011801B Hot Dog	18263	Food World	Pensacola, FL	Hot Dog	NQ	NQ	NQ
0012827 Dup	011801B Hot Dog	18263	Food World	Pensacola, FL	Hot Dog	NQ	NQ	NQ
0012828	011801B Hot Dog	18153	Watson's	Cleveland, TN	Hot Dog	NQ	NQ	NQ
0012828 Dup	011801B Hot Dog	18153	Watson's	Cleveland, TN	Hot Dog	NQ	NQ	NQ
0012829	011801B Hot Dog	18273	Food World II	Pensacola, FL	Hot Dog	NQ	NQ	NQ
0012829 Dup	011801B Hot Dog	18273	Food World II	Pensacola, FL	Hot Dog	NQ	NQ	NQ
0012830	011901A Fish	18252	Publix	Port St. Lucie, FL	Fish	NQ	NQ	NQ
0012830 Dup	011901A Fish	18252	Publix	Port St. Lucie, FL	Fish	NQ	NQ	NQ
0012831	011901A Fish	18229	Winn-Dixie	Port St. Lucie, FL	Fish	NQ	NQ	NQ
0012831 Dup	011901A Fish	18229	Winn-Dixie	Port St. Lucie, FL	Fish	NQ	NQ	NQ
0012832	011901A Fish	18241	Albertson's	Port St. Lucie, FL	Fish	NQ	NQ	NQ
0012832 Dup	011901A Fish	18241	Albertson's	Port St. Lucie, FL	Fish	NQ	NQ	NQ
0012833	011901A Fish	18359	Delchamps II	Mobile, AL	Fish	NQ	NQ	NQ
0012833 Dup	011901A Fish	18359	Delchamps II	Mobile, AL	Fish	NQ	NQ	NQ
0012834	011901A Fish	18200	Winn-Dixie	Columbus, GA	Fish	NQ	NQ	NQ
0012834 Dup	011901A Fish	18200	Winn-Dixie	Columbus, GA	Fish	NQ	NQ	NQ

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^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012835	011901B Fish	18348	Delchamps	Mobile, AL	Fish	NQ	**	NQ
0012835 Dup	011901B Fish	18348	Delchamps	Mobile, AL	Fish	NQ	**	NQ
0012836	011901B Fish	18212	Piggly Wiggly	Columbus, GA	Fish	NQ	**	NQ
0012836 Dup	011901B Fish	18212	Piggly Wiggly	Columbus, GA	Fish	NQ	**	NQ
0012837	011901B Fish	18337	Bruno's	Mobile, AL	Fish	NQ	**	NQ
0012837 Dup	011901B Fish	18337	Bruno's	Mobile, AL	Fish	NQ	**	NQ
0012838	011901B Fish	18325	Kroger's	Decatur, AL	Fish	NQ	**	NQ
0012838 Dup	011901B Fish	18325	Kroger's	Decatur, AL	Fish	NQ	**	NQ
0012839	011901B Fish	18314	Winn-Dixie	Decatur, AL	Fish	NQ	**	NQ
0012839 Dup	011901B Fish	18314	Winn-Dixie	Decatur, AL	Fish	NQ	**	NQ
0012835	013001A Fish	18348	Delchamps	Mobile, AL	Fish	-	NQ	-
0012835 Dup	013001A Fish	18348	Delchamps	Mobile, AL	Fish	-	NQ	-
0012836	013001A Fish	18212	Piggly Wiggly	Columbus, GA	Fish	-	NQ	-
0012836 Dup	013001A Fish	18212	Piggly Wiggly	Columbus, GA	Fish	-	NQ	-
0012837	013001A Fish	18337	Bruno's	Mobile, AL	Fish	-	NQ	-
0012837 Dup	013001A Fish	18337	Bruno's	Mobile, AL	Fish	-	NQ	-
0012838	013001A Fish	18325	Kroger's	Decatur, AL	Fish	-	NQ	-
0012838 Dup	013001A Fish	18325	Kroger's	Decatur, AL	Fish	-	NQ	-
0012839	013001A Fish	18314	Winn-Dixie	Decatur, AL	Fish	-	NQ	-
0012839 Dup	013001A Fish	18314	Winn-Dixie	Decatur, AL	Fish	-	NQ	-
0012840	012201A Fish	18303	Haloway	Decatur, AL	Fish	NQ	NQ	NQ
0012840 Dup	012201A Fish	18303	Haloway	Decatur, AL	Fish	NQ	NQ	NQ
0012841	012201A Fish	18177	Bi-Lo	Cleveland, TN	Fish	NQ	NQ	NQ
0012841 Dup	012201A Fish	18177	Bi-Lo	Cleveland, TN	Fish	NQ	NQ	NQ
0012842	012201A Fish	18166	Save-a-Lot	Cleveland, TN	Fish	NQ	NQ	NQ
0012842 Dup	012201A Fish	18166	Save-a-Lot	Cleveland, TN	Fish	NQ	NQ	NQ
0012843	012201A Fish	18284	Albertson's	Pensacola, FL	Fish	NQ	NQ	NQ
0012843 Dup	012201A Fish	18284	Albertson's	Pensacola, FL	Fish	NQ	NQ	NQ
0012844	012201A Fish	18262	Food World	Pensacola, FL	Fish	NQ	NQ	NQ
0012844 Dup	012201A Fish	18262	Food World	Pensacola, FL	Fish	NQ	NQ	NQ
0012845	012201B Fish	18154	Watson's	Cleveland, TN	Fish	NQ	NQ	NQ
0012845 Dup	012201B Fish	18154	Watson's	Cleveland, TN	Fish	NQ	NQ	NQ

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**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012846	012201B Fish	18274	Food World II	Pensacola, FL	Fish	NQ	NQ	NQ
0012846 Dup	012201B Fish	18274	Food World II	Pensacola, FL	Fish	NQ	NQ	NQ
0012903	020501A Bean	18256	Publix	Port St. Lucie, FL	Green Bean	NQ	NQ	NQ
0012903 Dup	020501A Bean	18256	Publix	Port St. Lucie, FL	Green Bean	NQ	NQ	NQ
0012904	020501A Bean	18234	Winn-Dixie	Port St. Lucie, FL	Green Bean	NQ	NQ	NQ
0012904 Dup	020501A Bean	18234	Winn-Dixie	Port St. Lucie, FL	Green Bean	NQ	NQ	NQ
0012905	020501A Bean	18245	Albertson's	Port St. Lucie, FL	Green Bean	NQ	NQ	NQ
0012905 Dup	020501A Bean	18245	Albertson's	Port St. Lucie, FL	Green Bean	NQ	NQ	NQ
0012906	020501A Bean	18363	Delchamps II	Mobile, AL	Green Bean	NQ	NQ	0.543
0012906 Dup	020501A Bean	18363	Delchamps II	Mobile, AL	Green Bean	NQ	NQ	NQ
0012907	020501A Bean	18204	Winn-Dixie	Columbus, GA	Green Bean	NQ	NQ	NQ
0012907 Dup	020501A Bean	18204	Winn-Dixie	Columbus, GA	Green Bean	NQ	NQ	NQ
0012908	020601C Bean	18352	Delchamps I	Mobile, AL	Green Bean	NQ	NQ	NQ
0012908 Dup	020601C Bean	18352	Delchamps I	Mobile, AL	Green Bean	NQ	NQ	NQ
0012909	020601C Bean	18216	Piggly Wiggly	Columbus, GA	Green Bean	NQ	NQ	NQ
0012909 Dup	020601C Bean	18216	Piggly Wiggly	Columbus, GA	Green Bean	NQ	NQ	NQ
0012910	020601C Bean	18341	Bruno's	Mobile, AL	Green Bean	NQ	NQ	NQ
0012910 Dup	020601C Bean	18341	Bruno's	Mobile, AL	Green Bean	NQ	NQ	NQ
0012911	020601C Bean	18329	Kroger's	Decatur, AL	Green Bean	NQ	NQ	NQ
0012911 Dup	020601C Bean	18329	Kroger's	Decatur, AL	Green Bean	NQ	NQ	NQ
0012912	020601C Bean	18318	Winn-Dixie	Decatur, AL	Green Bean	NQ	NQ	NQ
0012912 Dup	020601C Bean	18318	Winn-Dixie	Decatur, AL	Green Bean	NQ	NQ	NQ
0012913	020801C Bean	18307	Haloway	Decatur, AL	Green Bean	NQ	NQ	NQ
0012913 Dup	020801C Bean	18307	Haloway	Decatur, AL	Green Bean	NQ	NQ	NQ
0012914	020801C Bean	18193	Cub	Columbus, GA	Green Bean	NQ	NQ	NQ
0012914 Dup	020801C Bean	18193	Cub	Columbus, GA	Green Bean	NQ	NQ	NQ
0012915	020801C Bean	18182	Bi-Lo	Cleveland, TN	Green Bean	NQ	NQ	NQ
0012915 Dup	020801C Bean	18182	Bi-Lo	Cleveland, TN	Green Bean	NQ	NQ	NQ
0012916	020801C Bean	18289	Albertson's	Pensacola, FL	Green Bean	NQ	NQ	NQ
0012916 Dup	020801C Bean	18289	Albertson's	Pensacola, FL	Green Bean	NQ	NQ	NQ
0012917	020801C Bean	18267	Food World	Pensacola, FL	Green Bean	NQ	NQ	NQ
0012917 Dup	020801C Bean	18267	Food World	Pensacola, FL	Green Bean	NQ	NQ	NQ

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**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012918	020801D Bean	36868	Watson's	Cleveland, TN	Green Bean	NQ	NQ	NQ
0012918 Dup	020801D Bean	36868	Watson's	Cleveland, TN	Green Bean	NQ	NQ	NQ
0012919	020801D Bean	18278	Food World II	Pensacola, FL	Green Bean	NQ	NQ	NQ
0012919 Dup	020801D Bean	18278	Food World II	Pensacola, FL	Green Bean	NQ	NQ	NQ
0012867	020701A Milk	18254	Publix	Port St. Lucie, FL	Milk	NQ	NQ	NQ
0012867 Dup	020701A Milk	18254	Publix	Port St. Lucie, FL	Milk	NQ	NQ	NQ
0012868	020701A Milk	18231	Winn-Dixie	Port St. Lucie, FL	Milk	NQ	NQ	NQ
0012868 Dup	020701A Milk	18231	Winn-Dixie	Port St. Lucie, FL	Milk	NQ	NQ	NQ
0012869	020701A Milk	18243	Albertson's	Port St. Lucie, FL	Milk	NQ	NQ	NQ
0012869 Dup	020701A Milk	18243	Albertson's	Port St. Lucie, FL	Milk	NQ	NQ	NQ
0012870	020701A Milk	18361	Delchamps II	Mobile, AL	Milk	NQ	NQ	NQ
0012870 Dup	020701A Milk	18361	Delchamps II	Mobile, AL	Milk	NQ	NQ	NQ
0012871	020701A Milk	18202	Winn-Dixie	Columbus, GA	Milk	NQ	NQ	NQ
0012871 Dup	020701A Milk	18202	Winn-Dixie	Columbus, GA	Milk	0.693	NQ	NQ
0012872	020701B Milk	18350	Delchamps I	Mobile, AL	Milk	NQ	NQ	NQ
0012872 Dup	020701B Milk	18350	Delchamps I	Mobile, AL	Milk	NQ	NQ	NQ
0012873	020701B Milk	18214	Piggly Wiggly	Columbus, GA	Milk	NQ	NQ	NQ
0012873 Dup	020701B Milk	18214	Piggly Wiggly	Columbus, GA	Milk	NQ	NQ	NQ
0012874	020701B Milk	18339	Bruno's	Mobile, AL	Milk	NQ	NQ	NQ
0012874 Dup	020701B Milk	18339	Bruno's	Mobile, AL	Milk	NQ	NQ	NQ
0012875	020701B Milk	18327	Kroger's	Decatur, AL	Milk	NQ	NQ	NQ
0012875 Dup	020701B Milk	18327	Kroger's	Decatur, AL	Milk	NQ	NQ	NQ
0012876	020701B Milk	18316	Winn-Dixie	Decatur, AL	Milk	NQ	NQ	NQ
0012876 Dup	020701B Milk	18316	Winn-Dixie	Decatur, AL	Milk	NQ	NQ	NQ
0012877	020801A Milk	18305	Haloway	Decatur, AL	Milk	NQ	NQ	NQ
0012877 Dup	020801A Milk	18305	Haloway	Decatur, AL	Milk	NQ	NQ	NQ
0012878	020801A Milk	18190	Cub	Columbus, GA	Milk	NQ	NQ	NQ
0012878 Dup	020801A Milk	18190	Cub	Columbus, GA	Milk	NQ	NQ	NQ
0012879	020801A Milk	18179	Bi-Lo	Cleveland, TN	Milk	NQ	NQ	NQ
0012879 Dup	020801A Milk	18179	Bi-Lo	Cleveland, TN	Milk	NQ	NQ	NQ
0012880	020801A Milk	18168	Save-a-Lot	Cleveland, TN	Milk	NQ	NQ	NQ
0012880 Dup	020801A Milk	18168	Save-a-Lot	Cleveland, TN	Milk	0.573	NQ	NQ

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^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

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**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012881	020801A Milk	18287	Albertson's	Pensacola, FL	Milk	NQ	NQ	NQ
0012881 Dup	020801A Milk	18287	Albertson's	Pensacola, FL	Milk	NQ	NQ	NQ
0012882	020801B Milk	18265	Food World	Pensacola, FL	Milk	0.852	NQ	NQ
0012882 Dup	020801B Milk	18265	Food World	Pensacola, FL	Milk	NQ	NQ	NQ
0012883	020801B Milk	18156	Watson's	Cleveland, TN	Milk	NQ	NQ	NQ
0012883 Dup	020801B Milk	18156	Watson's	Cleveland, TN	Milk	NQ	NQ	NQ
0012884	020801B Milk	18276	Food World II	Pensacola, FL	Milk	NQ	NQ	NQ
0012884 Dup	020801B Milk	18276	Food World II	Pensacola, FL	Milk	0.605	NQ	NQ
0012926	020901C Apple	18205	Winn-Dixie	Columbus, GA	Apple	NQ	NQ	NQ
0012926 Dup	020901C Apple	18205	Winn-Dixie	Columbus, GA	Apple	NQ	NQ	NQ
0012927	020901C Apple	18257	Publix	Port St. Lucie, FL	Apple	NQ	NQ	NQ
0012927 Dup	020901C Apple	18257	Publix	Port St. Lucie, FL	Apple	NQ	NQ	NQ
0012928	020901C Apple	18235	Winn-Dixie	Port St. Lucie, FL	Apple	NQ	NQ	NQ
0012928 Dup	020901C Apple	18235	Winn-Dixie	Port St. Lucie, FL	Apple	NQ	NQ	NQ
0012929	020901C Apple	18246	Albertson's	Port St. Lucie, FL	Apple	NQ	NQ	NQ
0012929 Dup	020901C Apple	18246	Albertson's	Port St. Lucie, FL	Apple	NQ	NQ	NQ
0012930	020901C Apple	18364	Delchamps II	Mobile, AL	Apple	NQ	NQ	NQ
0012930 Dup	020901C Apple	18364	Delchamps II	Mobile, AL	Apple	NQ	NQ	NQ
0012931	021201A Apple	18353	Delchamps I	Mobile, AL	Apple	NQ	NQ	NQ
0012931 Dup	021201A Apple	18353	Delchamps I	Mobile, AL	Apple	NQ	NQ	NQ
0012932	021201A Apple	18217	Piggly Wiggly	Columbus, GA	Apple	NQ	NQ	NQ
0012932 Dup	021201A Apple	18217	Piggly Wiggly	Columbus, GA	Apple	NQ	NQ	NQ
0012933	021201A Apple	18342	Bruno's	Mobile, AL	Apple	NQ	NQ	NQ
0012933 Dup	021201A Apple	18342	Bruno's	Mobile, AL	Apple	NQ	NQ	NQ
0012934	021201A Apple	18330	Kroger's	Decatur, AL	Apple	NQ	NQ	NQ
0012934 Dup	021201A Apple	18330	Kroger's	Decatur, AL	Apple	NQ	NQ	2.35
0012935	021201A Apple	18319	Winn-Dixie	Decatur, AL	Apple	NQ	NQ	NQ
0012935 Dup	021201A Apple	18319	Winn-Dixie	Decatur, AL	Apple	NQ	NQ	NQ
0012936	021201B Apple	18308	Haloway	Decatur, AL	Apple	NQ	NQ	NQ
0012936 Dup	021201B Apple	18308	Haloway	Decatur, AL	Apple	NQ	NQ	NQ
0012937	021201B Apple	18194	Cub	Columbus, GA	Apple	NQ	NQ	NQ
0012937 Dup	021201B Apple	18194	Cub	Columbus, GA	Apple	NQ	NQ	NQ

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- = Not analyzed for

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012938	021201B Apple	18183	Bi-Lo	Cleveland, TN	Apple	NQ	NQ	NQ
0012938 Dup	021201B Apple	18183	Bi-Lo	Cleveland, TN	Apple	NQ	NQ	NQ
0012939	021201B Apple	18170	Save-a-Lot	Cleveland, TN	Apple	NQ	NQ	NQ
0012939 Dup	021201B Apple	18170	Save-a-Lot	Cleveland, TN	Apple	NQ	NQ	NQ
0012940	021201B Apple	18290	Albertson's	Pensacola, FL	Apple	NQ	NQ	NQ
0012940 Dup	021201B Apple	18290	Albertson's	Pensacola, FL	Apple	NQ	NQ	NQ
0012941	021201C Apple	18268	Food World	Pensacola, FL	Apple	NQ	NQ	1.13
0012941 Dup	021201C Apple	18268	Food World	Pensacola, FL	Apple	NQ	NQ	NQ
0012942	021201C Apple	18159	Watson's	Cleveland, TN	Apple	NQ	NQ	NQ
0012942 Dup	021201C Apple	18159	Watson's	Cleveland, TN	Apple	NQ	NQ	NQ^
0012943	021201C Apple	18279	Food World II	Pensacola, FL	Apple	NQ	NQ	NQ
0012943 Dup	021201C Apple	18279	Food World II	Pensacola, FL	Apple	NQ	NQ	NQ
0012885	020901A Bread	18255	Publix	Port St. Lucie, FL	Bread	NQ	NQ	NQ
0012885 Dup	020901A Bread	18255	Publix	Port St. Lucie, FL	Bread	NQ	NQ	NQ
0012886	020901A Bread	18233	Winn-Dixie	Port St. Lucie, FL	Bread	NQ	NQ	NQ
0012886 Dup	020901A Bread	18233	Winn-Dixie	Port St. Lucie, FL	Bread	NQ	NQ	NQ
0012887	020901A Bread	18244	Albertson's	Port St. Lucie, FL	Bread	NQ	NQ	NQ
0012887 Dup	020901A Bread	18244	Albertson's	Port St. Lucie, FL	Bread	NQ	NQ	NQ
0012888	020901A Bread	18362	Delchamps II	Mobile, AL	Bread	NQ	NQ	NQ
0012888 Dup	020901A Bread	18362	Delchamps II	Mobile, AL	Bread	NQ	NQ	NQ
0012889	020901A Bread	18203	Winn-Dixie	Columbus, GA	Bread	NQ	NQ	NQ
0012889 Dup	020901A Bread	18203	Winn-Dixie	Columbus, GA	Bread	NQ	NQ	NQ
0012890	020901B Bread	18351	Delchamps I	Mobile, AL	Bread	NQ	NQ	NQ
0012890 Dup	020901B Bread	18351	Delchamps I	Mobile, AL	Bread	NQ	NQ	NQ
0012891	020901B Bread	18215	Piggly Wiggly	Columbus, GA	Bread	NQ	NQ	NQ
0012891 Dup	020901B Bread	18215	Piggly Wiggly	Columbus, GA	Bread	NQ	NQ	NQ
0012892	020901B Bread	18340	Bruno's	Mobile, AL	Bread	NQ	NQ	NQ
0012892 Dup	020901B Bread	18340	Bruno's	Mobile, AL	Bread	NQ	NQ	NQ
0012893	020901B Bread	18328	Kroger's	Decatur, AL	Bread	NQ	NQ	NQ
0012893 Dup	020901B Bread	18328	Kroger's	Decatur, AL	Bread	NQ	NQ	NQ
0012894	020901B Bread	18317	Winn-Dixie	Decatur, AL	Bread	NQ	NQ	NQ
0012894 Dup	020901B Bread	18317	Winn-Dixie	Decatur, AL	Bread	NQ	NQ	NQ

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

^ = Sample result fell below 0.5 ng/g when adjusting for purity of PFOA.

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012895	021301A Bread	18306	Haloway	Decatur, AL	Bread	NQ	NQ	NQ
0012895 Dup	021301A Bread	18306	Haloway	Decatur, AL	Bread	NQ	NQ	NQ
0012896	021301A Bread	18192	Cub	Columbus, GA	Bread	NQ	NQ	NQ
0012896 Dup	021301A Bread	18192	Cub	Columbus, GA	Bread	NQ	NQ	NQ
0012897	021301A Bread	18181	Bi-Lo	Cleveland, TN	Bread	NQ	NQ	NQ
0012897 Dup	021301A Bread	18181	Bi-Lo	Cleveland, TN	Bread	NQ	NQ	NQ
0012898	021301A Bread	18172	Save-a-Lot	Cleveland, TN	Bread	NQ	NQ	NQ
0012898 Dup	021301A Bread	18172	Save-a-Lot	Cleveland, TN	Bread	NQ	NQ	14.7 <sup>^</sup>
0012899	021301A Bread	18288	Albertson's	Pensacola, FL	Bread	NQ	NQ	NQ
0012899 Dup	021301A Bread	18288	Albertson's	Pensacola, FL	Bread	NQ	NQ	NQ
0012900	021301B Bread	18266	Food World	Pensacola, FL	Bread	NQ	NQ	NQ
0012900 Dup	021301B Bread	18266	Food World	Pensacola, FL	Bread	NQ	NQ	NQ
0012901	021301B Bread	18158	Watson's	Cleveland, TN	Bread	NQ	NQ	NQ
0012901 Dup	021301B Bread	18158	Watson's	Cleveland, TN	Bread	NQ	NQ	NQ
0012902	021301B Bread	18277	Food World II	Pensacola, FL	Bread	NQ	NQ	NQ
0012902 Dup	021301B Bread	18277	Food World II	Pensacola, FL	Bread	NQ	NQ	0.524
0012758	021301C Beef	18248	Publix	Port St. Lucie, FL	Ground Beef	NQ	NQ	NQ
0012758 Dup	021301C Beef	18248	Publix	Port St. Lucie, FL	Ground Beef	NQ	NQ	0.504
0012759	021301C Beef	18225	Winn-Dixie	Port St. Lucie, FL	Ground Beef	NQ	NQ	NQ
0012759 Dup	021301C Beef	18225	Winn-Dixie	Port St. Lucie, FL	Ground Beef	NQ	NQ	NQ
0012760	021301C Beef	18237	Albertson's	Port St. Lucie, FL	Ground Beef	NQ	NQ	1.09
0012760 Dup	021301C Beef	18237	Albertson's	Port St. Lucie, FL	Ground Beef	NQ	NQ	NQ
0012761	021301C Beef	18355	Delchamps II	Mobile, AL	Ground Beef	NQ	NQ	NQ
0012761 Dup	021301C Beef	18355	Delchamps II	Mobile, AL	Ground Beef	NQ	NQ	NQ
0012762	021301C Beef	18196	Winn-Dixie	Columbus, GA	Ground Beef	NQ	NQ	NQ
0012762 Dup	021301C Beef	18196	Winn-Dixie	Columbus, GA	Ground Beef	NQ	NQ	NQ
0012763	021301D Beef	18344	Delchamps I	Mobile, AL	Ground Beef	0.570	NQ	NQ
0012763 Dup	021301D Beef	18344	Delchamps I	Mobile, AL	Ground Beef	0.587	NQ	NQ
0012764	021301D Beef	18208	Piggly Wiggly	Columbus, GA	Ground Beef	NQ	NQ	NQ
0012764 Dup	021301D Beef	18208	Piggly Wiggly	Columbus, GA	Ground Beef	NQ	NQ	NQ
0012765	021301D Beef	18333	Bruno's	Mobile, AL	Ground Beef	NQ	NQ	NQ
0012765 Dup	021301D Beef	18333	Bruno's	Mobile, AL	Ground Beef	NQ	NQ	NQ

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

<sup>^</sup> = Result is suspect. Apparent contamination.

<sup>^^</sup> = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012766	021301D Beef	18321	Kroger's	Decatur, AL	Ground Beef	NQ	NQ	NQ
0012766 Dup	021301D Beef	18321	Kroger's	Decatur, AL	Ground Beef	NQ	NQ	NQ
0012767	021301D Beef	18310	Winn-Dixie	Decatur, AL	Ground Beef	NQ	NQ	NQ
0012767 Dup	021301D Beef	18310	Winn-Dixie	Decatur, AL	Ground Beef	NQ	NQ	NQ
0012768	021401A Beef	18300	Haloway	Decatur, AL	Ground Beef	NQ	NQ	NQ
0012768 Dup	021401A Beef	18300	Haloway	Decatur, AL	Ground Beef	NQ	NQ	NQ
0012769	021401A Beef	18185	Cub	Columbus, GA	Ground Beef	NQ	NQ	NQ
0012769 Dup	021401A Beef	18185	Cub	Columbus, GA	Ground Beef	NQ	NQ	NQ
0012770	021401A Beef	18173	Bi-Lo	Cleveland, TN	Ground Beef	NQ	NQ	NQ
0012770 Dup	021401A Beef	18173	Bi-Lo	Cleveland, TN	Ground Beef	NQ	NQ	NQ
0012771	021401A Beef	18162	Save-a-Lot	Cleveland, TN	Ground Beef	NQ	NQ	NQ
0012771 Dup	021401A Beef	18162	Save-a-Lot	Cleveland, TN	Ground Beef	NQ	NQ	NQ
0012772	021401A Beef	18281	Albertson's	Pensacola, FL	Ground Beef	NQ	NQ	NQ
0012772 Dup	021401A Beef	18281	Albertson's	Pensacola, FL	Ground Beef	NQ	NQ	NQ
0012773	021401B Beef	18259	Food World	Pensacola, FL	Ground Beef	NQ	NQ	NQ
0012773 Dup	021401B Beef	18259	Food World	Pensacola, FL	Ground Beef	NQ	NQ	NQ
0012774	021401B Beef	18161	Watson's	Cleveland, TN	Ground Beef	NQ	NQ	NQ
0012774 Dup	021401B Beef	18161	Watson's	Cleveland, TN	Ground Beef	NQ	NQ	NQ
0012775	021401B Beef	18270	Food World II	Pensacola, FL	Ground Beef	NQ	NQ	NQ
0012775 Dup	021401B Beef	18270	Food World II	Pensacola, FL	Ground Beef	NQ	NQ	NQ
0012847	012301A Egg	18253	Publix	Port St. Lucie, FL	Egg	^^	NQ	^^
0012847 Dup	012301A Egg	18253	Publix	Port St. Lucie, FL	Egg	^^	NQ	^^
0012848	012301A Egg	18230	Winn-Dixie	Port St. Lucie, FL	Egg	^^	NQ	^^
0012848 Dup	012301A Egg	18230	Winn-Dixie	Port St. Lucie, FL	Egg	^^	NQ	^^
0012849	012301A Egg	18242	Albertson's	Port St. Lucie, FL	Egg	^^	NQ	^^
0012849 Dup	012301A Egg	18242	Albertson's	Port St. Lucie, FL	Egg	^^	NQ	^^
0012850	012301A Egg	18360	Delchamps II	Mobile, AL	Egg	^^	NQ	^^
0012850 Dup	012301A Egg	18360	Delchamps II	Mobile, AL	Egg	^^	NQ	^^
0012851	012301A Egg	18201	Winn-Dixie	Columbus, GA	Egg	^^	NQ	^^
0012851 Dup	012301A Egg	18201	Winn-Dixie	Columbus, GA	Egg	^^	NQ	^^
0012852	012301B Egg	18349	Delchamps I	Mobile, AL	Egg	^^	NQ	^^
0012852 Dup	012301B Egg	18349	Delchamps I	Mobile, AL	Egg	^^	NQ	^^

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0012853	012301B Egg	18219	Peggy's	Port St. Lucie, FL	Egg	^^	NQ	^^
0012853 Dup	012301B Egg	18219	Peggy's	Port St. Lucie, FL	Egg	^^	NQ	^^
0012854	012301B Egg	18213	Piggly Wiggly	Columbus, GA	Egg	^^	NQ	^^
0012854 Dup	012301B Egg	18213	Piggly Wiggly	Columbus, GA	Egg	^^	NQ	^^
0012855	012301B Egg	18338	Bruno's	Mobile, AL	Egg	^^	NQ	^^
0012855 Dup	012301B Egg	18338	Bruno's	Mobile, AL	Egg	^^	NQ	^^
0012856	012301B Egg	18326	Kroger's	Decatur, AL	Egg	^^	NQ	^^
0012856 Dup	012301B Egg	18326	Kroger's	Decatur, AL	Egg	^^	NQ	^^
0012857	012401A Egg	18315	Winn-Dixie	Decatur, AL	Egg	^^	NQ	^^
0012857 Dup	012401A Egg	18315	Winn-Dixie	Decatur, AL	Egg	^^	NQ	^^
0012858	012401A Egg	18304	Haloway	Decatur, AL	Egg	^^	NQ	^^
0012858 Dup	012401A Egg	18304	Haloway	Decatur, AL	Egg	^^	NQ	^^
0012859	012401A Egg	18189	Cub	Columbus, GA	Egg	^^	NQ	^^
0012859 Dup	012401A Egg	18189	Cub	Columbus, GA	Egg	^^	NQ	^^
0012860	012401A Egg	18178	Bi-Lo	Cleveland, TN	Egg	^^	NQ	^^
0012860 Dup	012401A Egg	18178	Bi-Lo	Cleveland, TN	Egg	^^	NQ	^^
0012861	012401A Egg	18167	Save-a-Lot	Cleveland, TN	Egg	^^	NQ	^^
0012861 Dup	012401A Egg	18167	Save-a-Lot	Cleveland, TN	Egg	^^	NQ	^^
0012862	012401B Egg	18286	Albertson's	Pensacola, FL	Egg	^^	NQ	^^
0012862 Dup	012401B Egg	18286	Albertson's	Pensacola, FL	Egg	^^	NQ	^^
0012863	012401B Egg	18264	Food World	Pensacola, FL	Egg	^^	NQ	^^
0012863 Dup	012401B Egg	18264	Food World	Pensacola, FL	Egg	^^	NQ	^^
0012864	012401B Egg	18296	Farmer's Market	Mobile, AL	Egg	^^	NQ	^^
0012864 Dup	012401B Egg	18296	Farmer's Market	Mobile, AL	Egg	^^	NQ	^^
0012865	012401B Egg	18155	Watson's	Cleveland, TN	Egg	^^	NQ	^^
0012865 Dup	012401B Egg	18155	Watson's	Cleveland, TN	Egg	^^	NQ	^^
0012866	012401B Egg	18275	Food World II	Pensacola, FL	Egg	^^	NQ	^^
0012866 Dup	012401B Egg	18275	Food World II	Pensacola, FL	Egg	^^	NQ	^^
0106429	050801A Egg	21213	Winn-Dixie	Port St. Lucie, FL	Egg	NQ	-	^^
0106429 Dup	050801A Egg	21213	Winn-Dixie	Port St. Lucie, FL	Egg	NQ	-	^^
0106430	050801A Egg	21214	Albertson's	Port St. Lucie, FL	Egg	NQ	-	^^
0106430 Dup	050801A Egg	21214	Albertson's	Port St. Lucie, FL	Egg	NQ	-	^^

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0106431	050801A Egg	21216	Winn-Dixie	Columbus, GA	Egg	NQ	-	^^
0106431 Dup	050801A Egg	21216	Winn-Dixie	Columbus, GA	Egg	NQ	-	^^
0106432	050801A Egg	21217	Delchamps I	Mobile, AL	Egg	NQ	-	^^
0106432 Dup	050801A Egg	21217	Delchamps I	Mobile, AL	Egg	NQ	-	^^
0106433	050801A Egg	21218	Peggy's	Port St. Lucie, FL	Egg	NQ	-	^^
0106433 Dup	050801A Egg	21218	Peggy's	Port St. Lucie, FL	Egg	NQ	-	^^
0106434	050801B Egg	21219	Piggly Wiggly	Columbus, GA	Egg	NQ	-	^^
0106434 Dup	050801B Egg	21219	Piggly Wiggly	Columbus, GA	Egg	NQ	-	^^
0106435	050801B Egg	21221	Kroger's	Decatur, AL	Egg	NQ	-	^^
0106435 Dup	050801B Egg	21221	Kroger's	Decatur, AL	Egg	NQ	-	^^
0106436	050801B Egg	21222	Winn-Dixie	Decatur, AL	Egg	NQ	-	^^
0106436 Dup	050801B Egg	21222	Winn-Dixie	Decatur, AL	Egg	NQ	-	^^
0106437	050801B Egg	21223	Haloway	Decatur, AL	Egg	NQ	-	^^
0106437 Dup	050801B Egg	21223	Haloway	Decatur, AL	Egg	NQ	-	^^
0106438	050801B Egg	21224	Cub	Columbus, GA	Egg	NQ	-	^^
0106438 Dup	050801B Egg	21224	Cub	Columbus, GA	Egg	NQ	-	^^
0106439	050901A Egg	21225	Bi-Lo	Cleveland, TN	Egg	NQ	-	^^
0106439 Dup	050901A Egg	21225	Bi-Lo	Cleveland, TN	Egg	NQ	-	^^
0106440	050901A Egg	21226	Save-a-Lot	Cleveland, TN	Egg	NQ	-	^^
0106440 Dup	050901A Egg	21226	Save-a-Lot	Cleveland, TN	Egg	NQ	-	^^
0106441	050901A Egg	21227	Albertson's	Pensacola, FL	Egg	NQ	-	^^
0106441 Dup	050901A Egg	21227	Albertson's	Pensacola, FL	Egg	NQ	-	^^
0106442	050901A Egg	21228	Food World	Pensacola, FL	Egg	NQ	-	^^
0106442 Dup	050901A Egg	21228	Food World	Pensacola, FL	Egg	NQ	-	^^
0106443	050901A Egg	21229	Farmer's Market	Mobile, AL	Egg	NQ	-	^^
0106443 Dup	050901A Egg	21229	Farmer's Market	Mobile, AL	Egg	NQ	-	^^
0106444	050901B Egg	21230	Watson's	Cleveland, TN	Egg	NQ	-	^^
0106444 Dup	050901B Egg	21230	Watson's	Cleveland, TN	Egg	NQ	-	^^
0106445	050901B Egg	21231	Food World II	Pensacola, FL	Egg	NQ	-	^^
0106445 Dup	050901B Egg	21231	Food World II	Pensacola, FL	Egg	NQ	-	^^
0106446	050901B Egg	na	\$	\$	Egg	NQ	-	^^
0106446 Dup	050901B Egg	na	\$	\$	Egg	NQ	-	^^

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

\$ = Sample labels fell off during second shipping and there was no way of telling samples apart.

**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0106447	050901B Egg	na	\$	\$	Egg	NQ	-	^^
0106447 Dup	050901B Egg	na	\$	\$	Egg	NQ	-	^^
0106448	050901B Egg	na	\$	\$	Egg	NQ	-	^^
0106448 Dup	050901B Egg	na	\$	\$	Egg	NQ	-	^^
0106429	051801A Egg	21213	Winn-Dixie	Port St. Lucie, FL	Egg	-	-	NQ
0106429 Dup	051801A Egg	21213	Winn-Dixie	Port St. Lucie, FL	Egg	-	-	NQ
0106430	051801A Egg	21214	Albertson's	Port St. Lucie, FL	Egg	-	-	NQ
0106430 Dup	051801A Egg	21214	Albertson's	Port St. Lucie, FL	Egg	-	-	NQ
0106431	051801A Egg	21216	Winn-Dixie	Columbus, GA	Egg	-	-	NQ
0106431 Dup	051801A Egg	21216	Winn-Dixie	Columbus, GA	Egg	-	-	NQ
0106432	051801A Egg	21217	Delchamps I	Mobile, AL	Egg	-	-	NQ
0106432 Dup	051801A Egg	21217	Delchamps I	Mobile, AL	Egg	-	-	NQ
0106433	051801A Egg	21218	Peggy's	Port St. Lucie, FL	Egg	-	-	NQ
0106433 Dup	051801A Egg	21218	Peggy's	Port St. Lucie, FL	Egg	-	-	NQ
0106434	051801B Egg	21219	Piggly Wiggly	Columbus, GA	Egg	-	-	NQ
0106434 Dup	051801B Egg	21219	Piggly Wiggly	Columbus, GA	Egg	-	-	NQ
0106435	051801B Egg	21221	Kroger's	Decatur, AL	Egg	-	-	NQ
0106435 Dup	051801B Egg	21221	Kroger's	Decatur, AL	Egg	-	-	NQ
0106436	051801B Egg	21222	Winn-Dixie	Decatur, AL	Egg	-	-	NQ
0106436 Dup	051801B Egg	21222	Winn-Dixie	Decatur, AL	Egg	-	-	NQ
0106437	051801B Egg	21223	Haloway	Decatur, AL	Egg	-	-	NQ
0106437 Dup	051801B Egg	21223	Haloway	Decatur, AL	Egg	-	-	NQ
0106438	051801B Egg	21224	Cub	Columbus, GA	Egg	-	-	NQ
0106438 Dup	051801B Egg	21224	Cub	Columbus, GA	Egg	-	-	NQ
0106439	052101A Egg	21225	Bi-Lo	Cleveland, TN	Egg	-	-	NQ
0106439 Dup	052101A Egg	21225	Bi-Lo	Cleveland, TN	Egg	-	-	NQ
0106440	052101A Egg	21226	Save-a-Lot	Cleveland, TN	Egg	-	-	NQ
0106440 Dup	052101A Egg	21226	Save-a-Lot	Cleveland, TN	Egg	-	-	NQ
0106441	052101A Egg	21227	Albertson's	Pensacola, FL	Egg	-	-	NQ
0106441 Dup	052101A Egg	21227	Albertson's	Pensacola, FL	Egg	-	-	NQ
0106442	052101A Egg	21228	Food World	Pensacola, FL	Egg	-	-	NQ
0106442 Dup	052101A Egg	21228	Food World	Pensacola, FL	Egg	-	-	NQ

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

\$ = Sample labels fell off during second shipping and there was no way of telling samples apart.



**Table II continued: Summary of PFOS, FOSA and PFOA Residues Found in Samples**

Centre ID	Data Set No.	Sponsor ID	Store Name	Location	Matrix	Residue Found (ng/g)*		
						PFOS	FOSA	PFOA
0106443	052101A Egg	21229	Farmer's Market	Mobile, AL	Egg	-	-	NQ
0106443 Dup	052101A Egg	21229	Farmer's Market	Mobile, AL	Egg	-	-	NQ
0106444	052101B Egg	21230	Watson's	Cleveland, TN	Egg	-	-	NQ
0106444 Dup	052101B Egg	21230	Watson's	Cleveland, TN	Egg	-	-	NQ
0106445	052101B Egg	21231	Food World II	Pensacola, FL	Egg	-	-	NQ
0106445 Dup	052101B Egg	21231	Food World II	Pensacola, FL	Egg	-	-	NQ
0106446	052101B Egg	na	\$	\$	Egg	-	-	NQ
0106446 Dup	052101B Egg	na	\$	\$	Egg	-	-	NQ
0106447	052101B Egg	na	\$	\$	Egg	-	-	NQ
0106447 Dup	052101B Egg	na	\$	\$	Egg	-	-	NQ
0106448	052101B Egg	na	\$	\$	Egg	-	-	NQ
0106448 Dup	052101B Egg	na	\$	\$	Egg	-	-	NQ

NQ = Not Quantifiable. The residue detected, if any, was below the limit of the study which is 0.5 ng/g.

\*Value is based on wet weight (5.0 g sample processed as received)

\*\* Values rejected based on unacceptable recoveries. Data has been replaced with either reanalysis or re-extraction values.

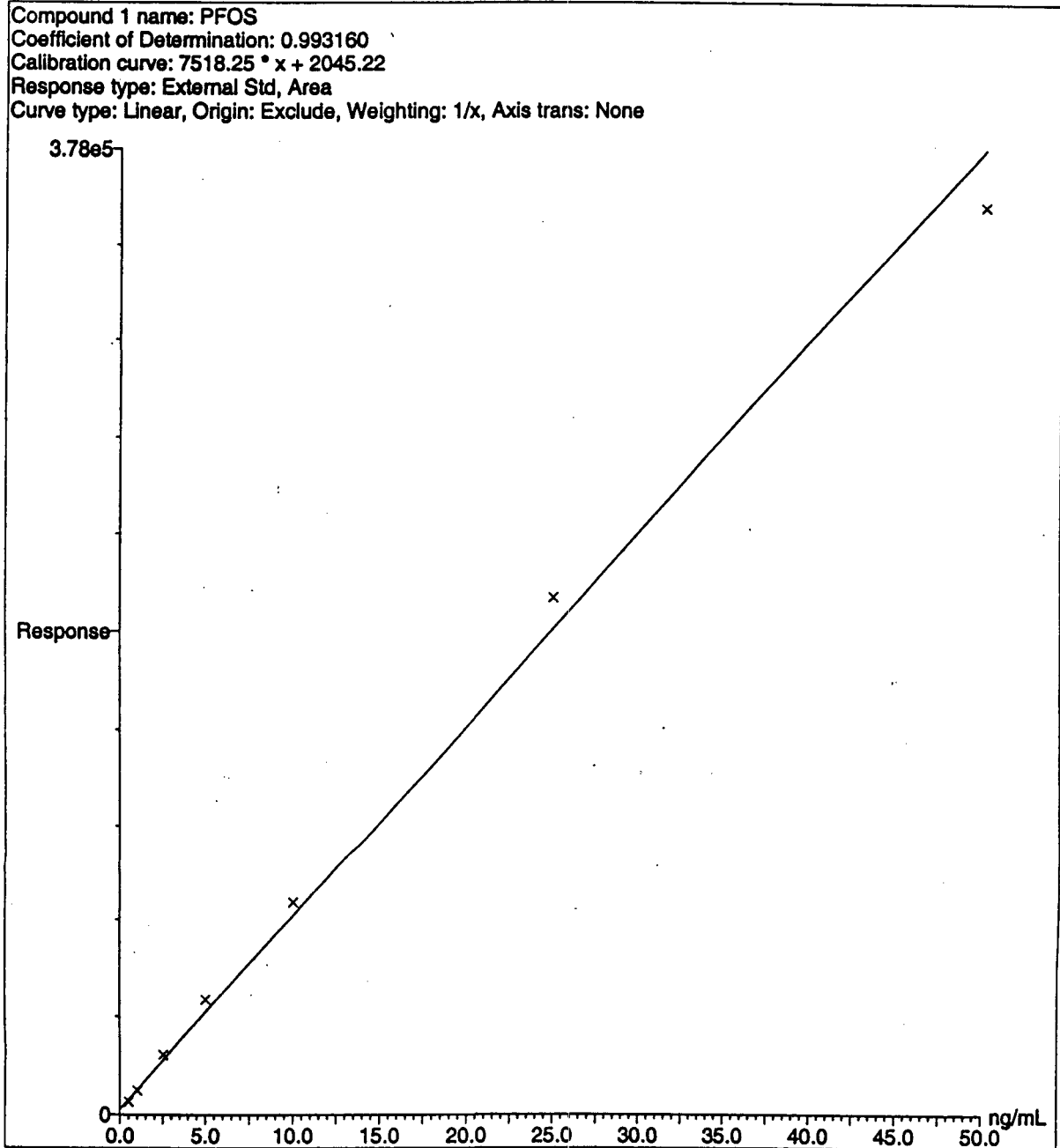
^^ = Results rejected based on apparent contamination. Data has been replaced with re-extracted values.

- = Not analyzed for

\$ = Sample labels fell off during second shipping and there was no way of telling samples apart.

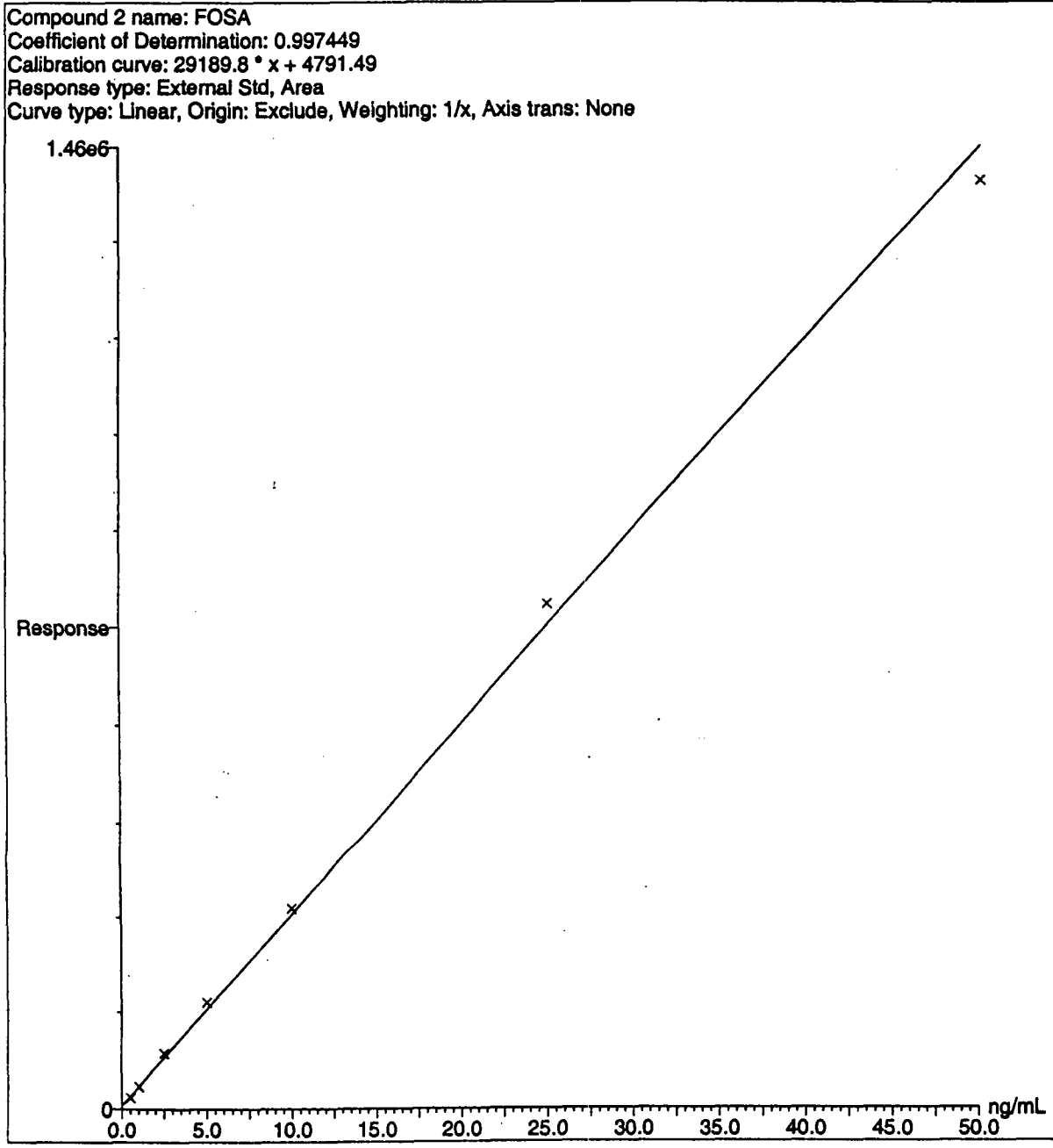
# **12.0 FIGURES**

Figure 1. Typical Calibration Curve for PFOS



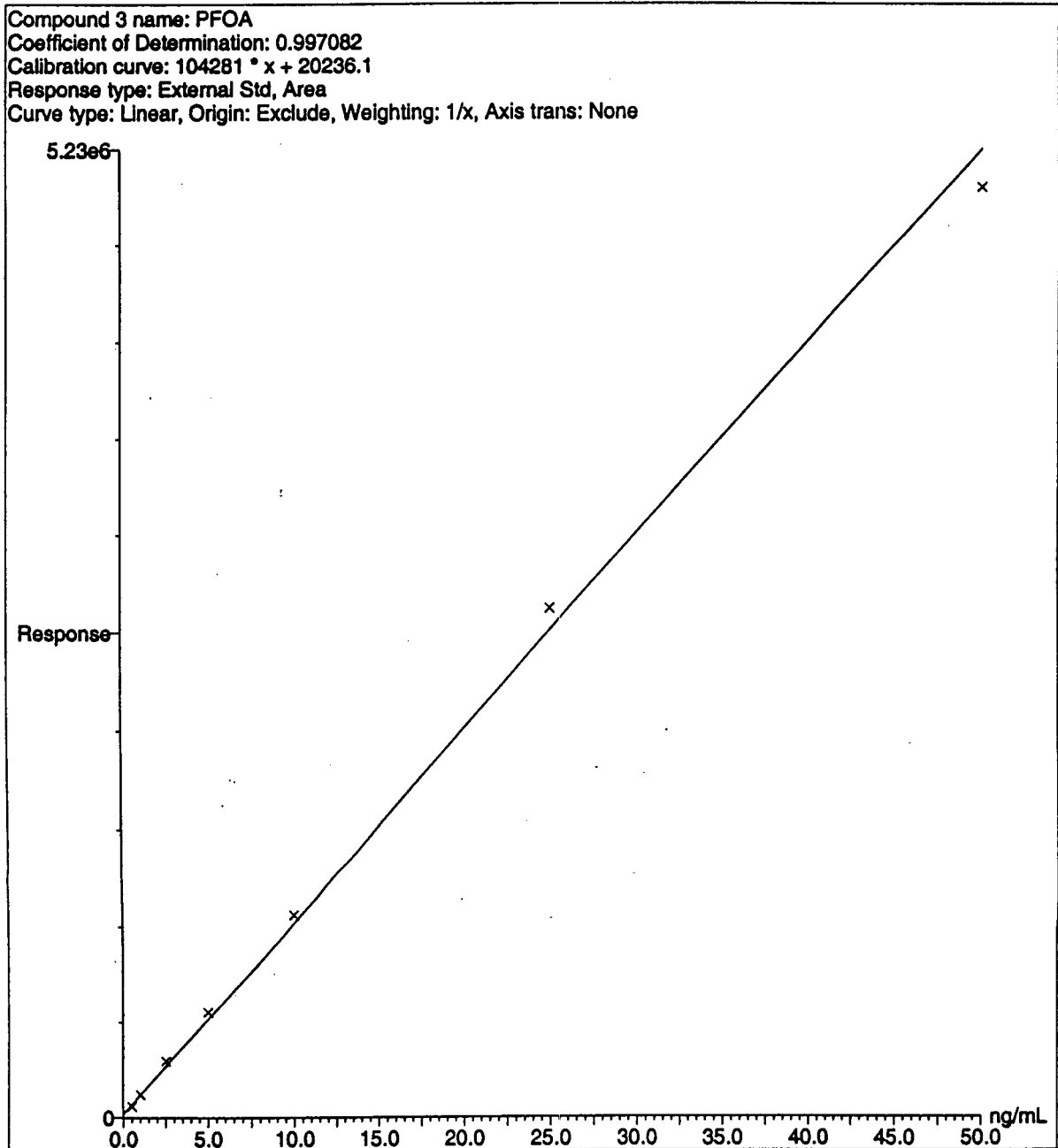
Centre Analytical Laboratories, Inc.

Figure 2. Typical Calibration Curve for FOSA



Centre Analytical Laboratories, Inc.

Figure 3. Typical Calibration Curve for PFOA

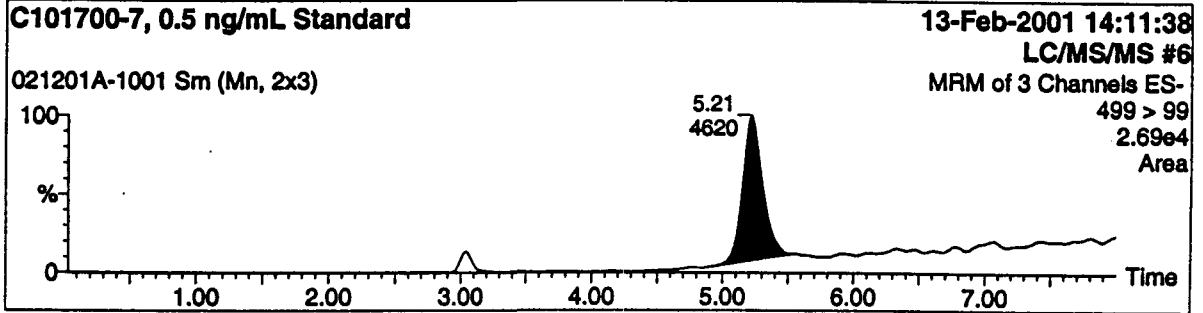


Centre Analytical Laboratories, Inc.

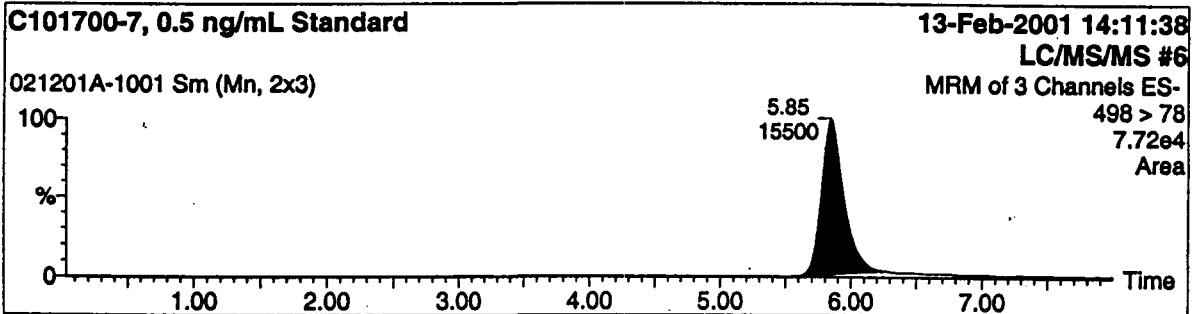
**Figure 4. Chromatogram Representing a 0.5 ng/mL mixed calibration standard of PFOS, FOSA and PFOA**

Name: 021201A-1001  
Text: C101700-7, 0.5 ng/mL Standard

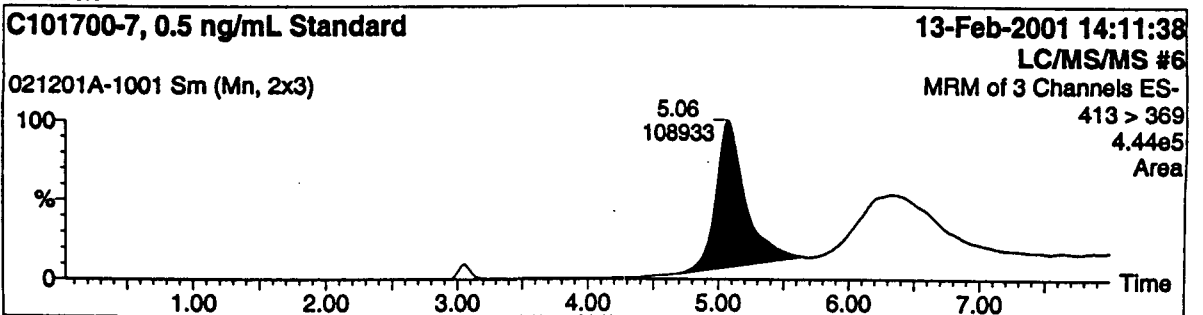
1: PFOS



2: FOSA



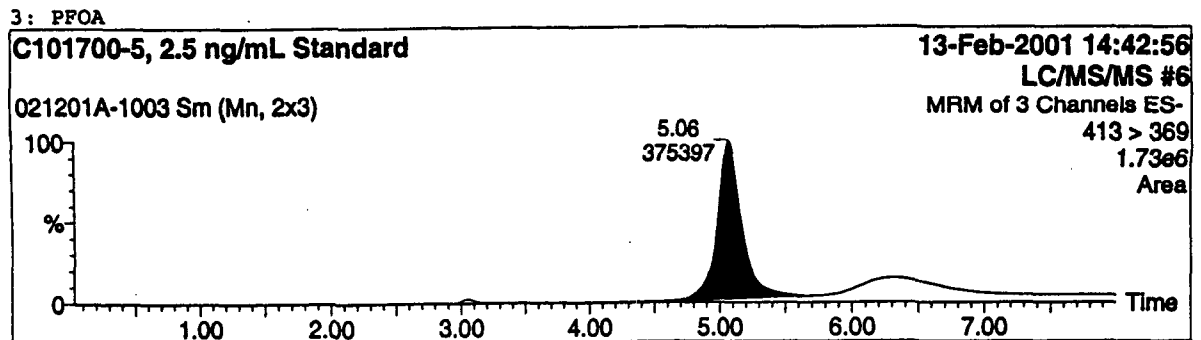
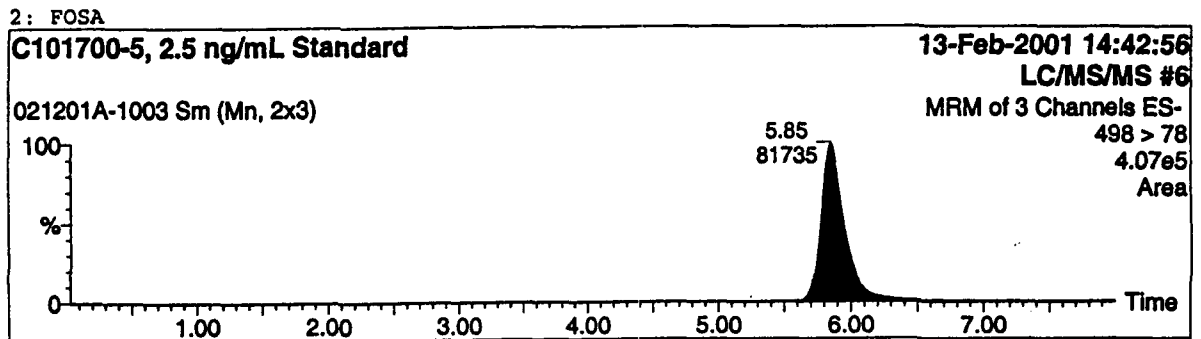
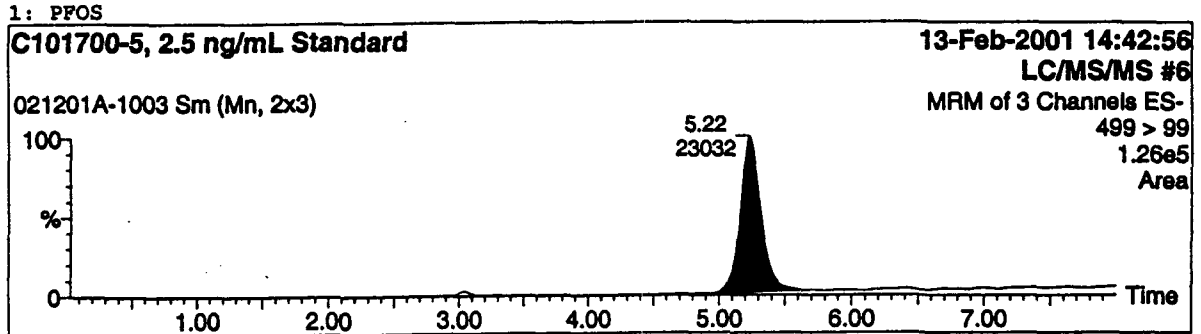
3: PFOA



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**Figure 5. Chromatogram Representing a 2.5 ng/mL mixed calibration standard of PFOS, FOSA and PFOA**

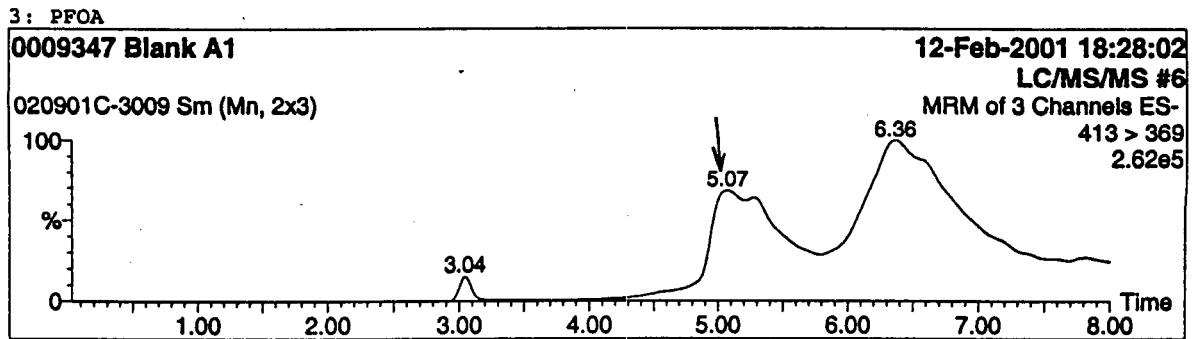
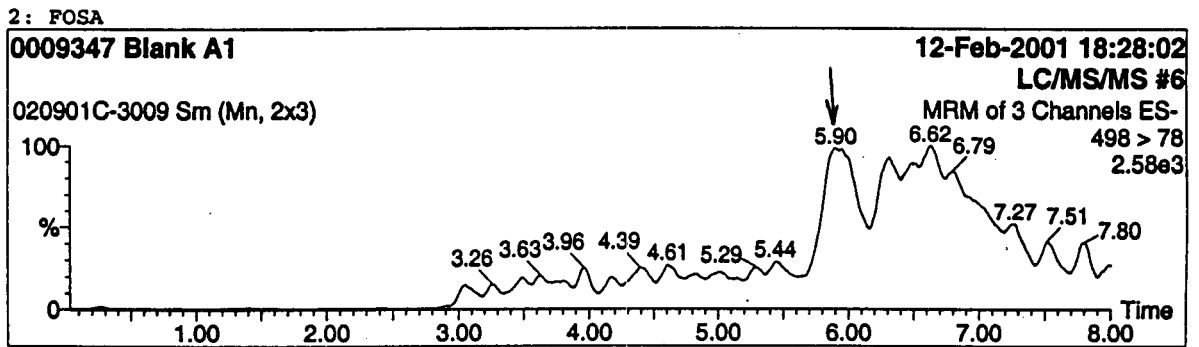
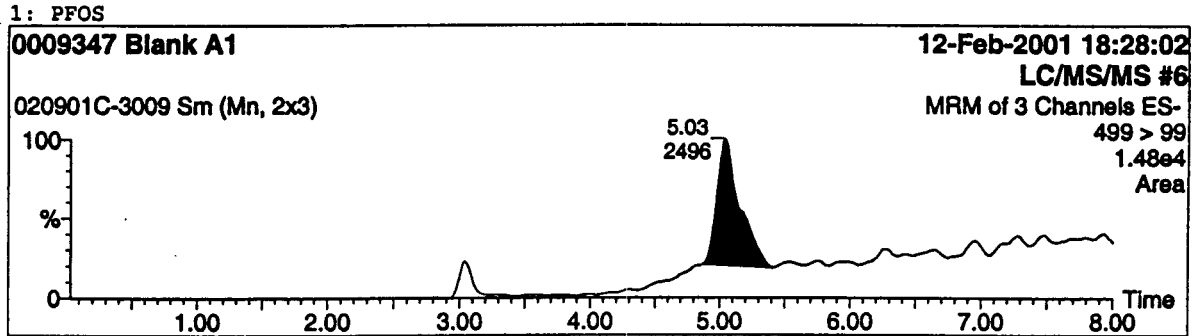
Name: 021201A-1003  
Text: C101700-5, 2.5 ng/mL Standard



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**Figure 6. Chromatogram Representing a Control Apple Sample  
(Centre ID: 0009347 Blank A1, Data Set: 020901C Apple)**

Name: 020901C-3009  
Text: 0009347 Blank A1



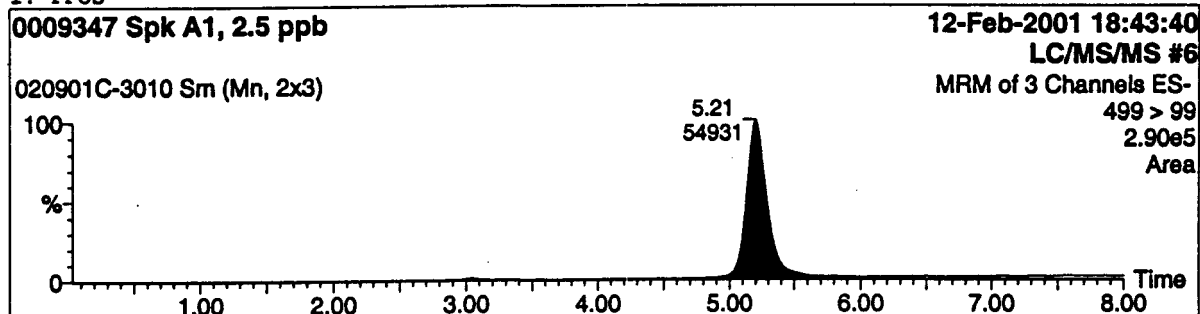
Centre Analytical Laboratories, Inc.



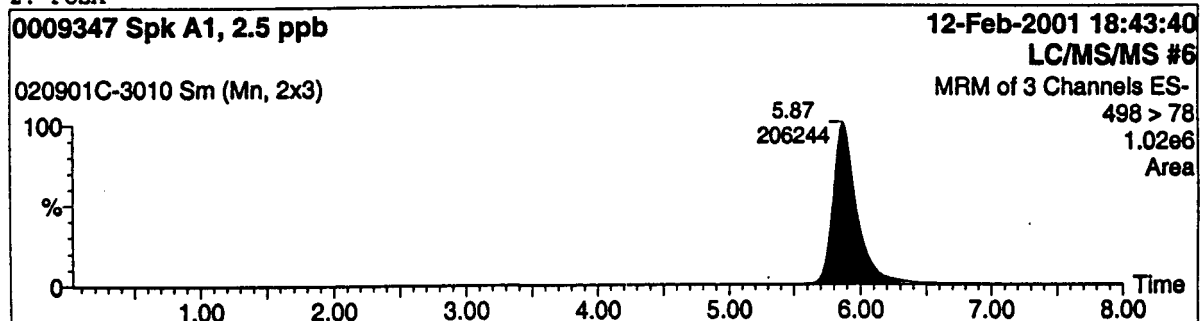
**Figure 7. Chromatogram Representing a Control Apple Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009347 Spk A1, Data Set: 020901C Apple)**

Name: 020901C-3010  
Text: 0009347 Spk A1, 2.5 ppb

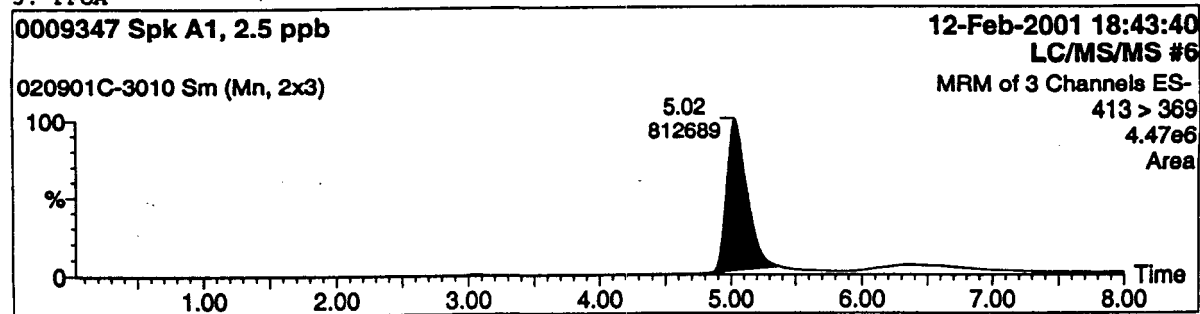
1: PFOS



2: FOSA



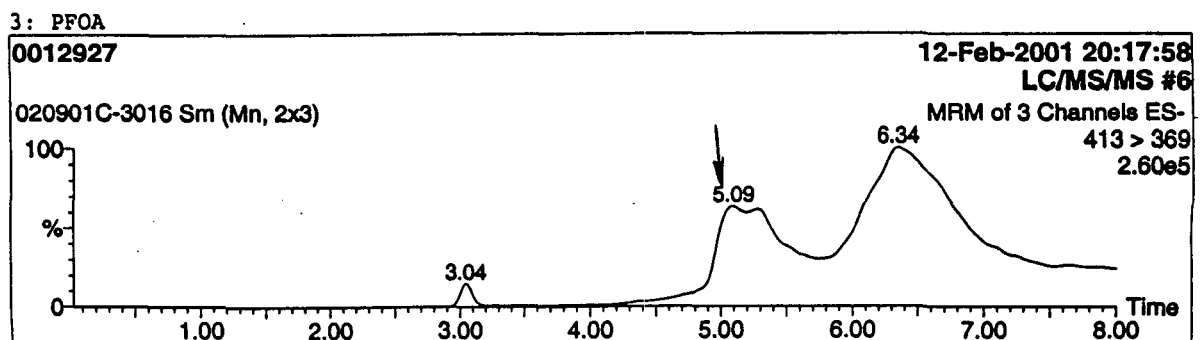
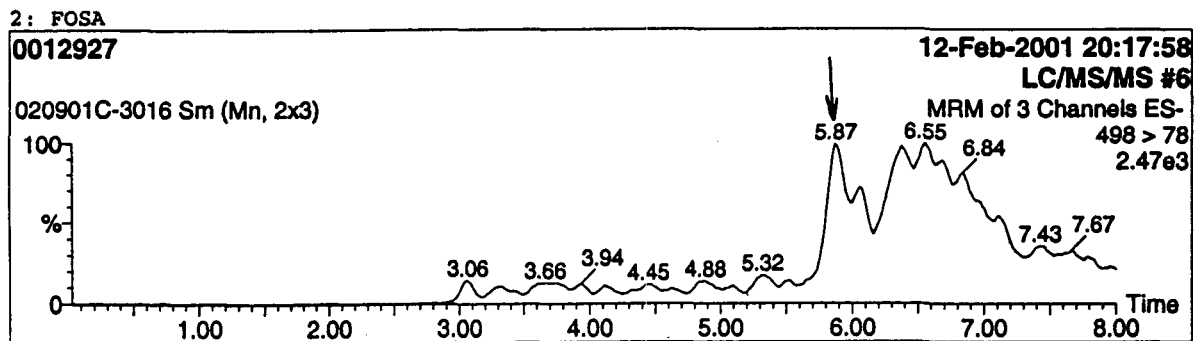
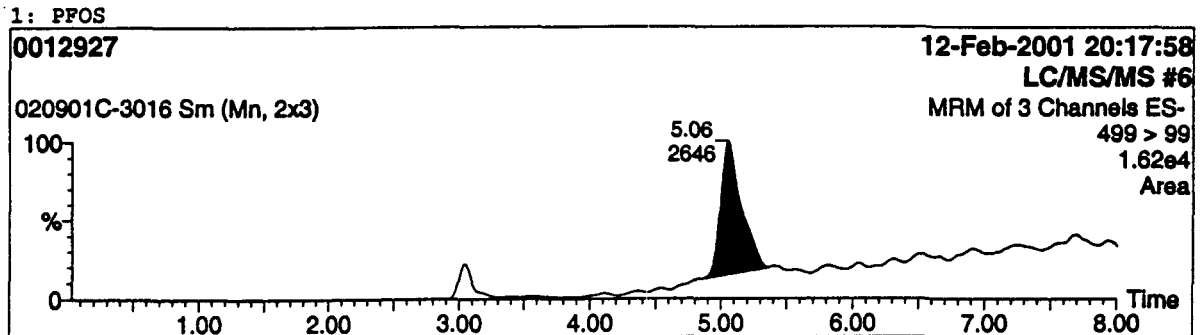
3: PFOA



Centre Analytical Laboratories, Inc.

**Figure 8. Chromatogram Representing an Apple Sample (Centre ID: 0012927, Sponsor ID: Publix, 18257, Data Set: 020901C Apple)**

Name: 020901C-3016  
Text: 0012927

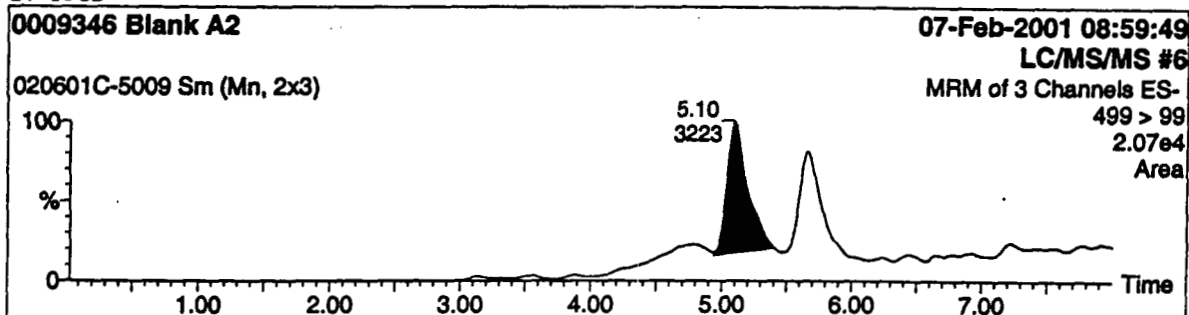


Centre Analytical Laboratories, Inc.

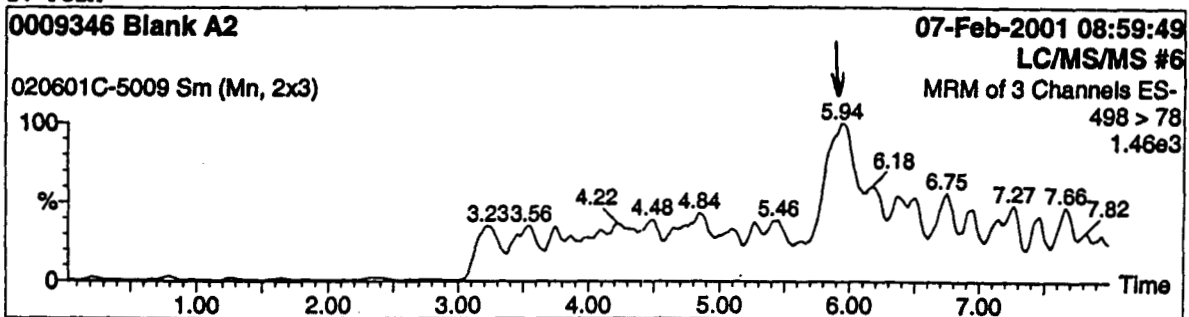
**Figure 9. Chromatogram Representing a Control Green Bean Sample (Centre ID: 0009346 Blank A2, Data Set: 020601C Bean)**

Name: 020601C-5009  
Text: 0009346 Blank A2

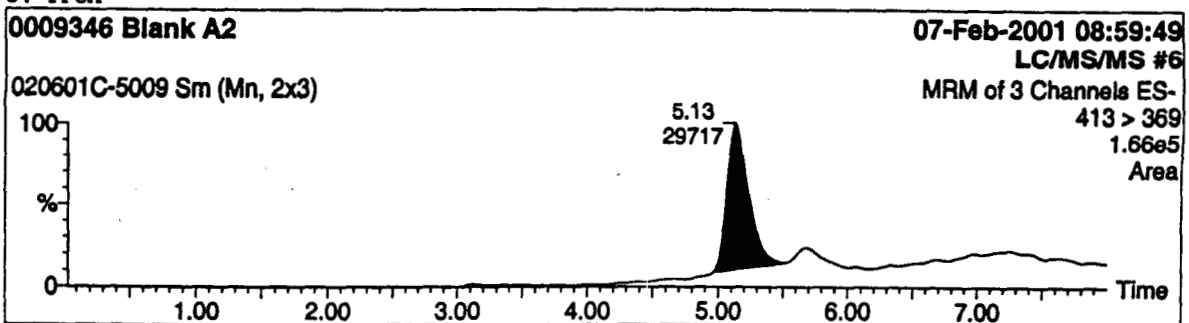
1: PFOS



2: FOSA



3: PFOA

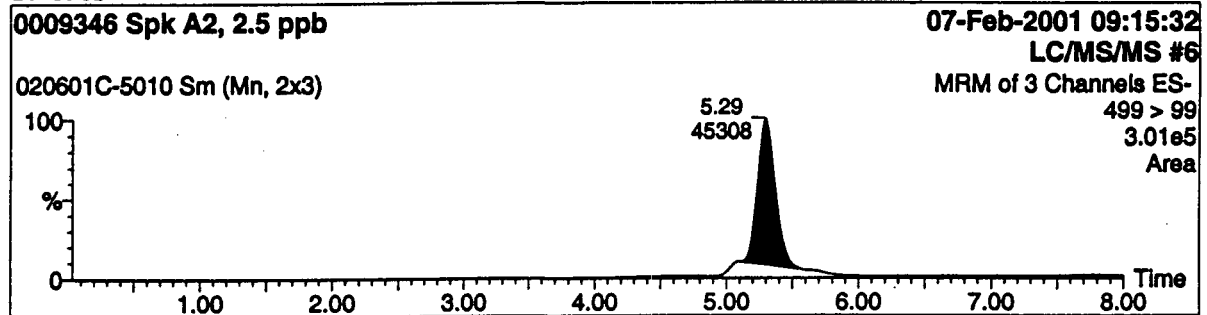


Centre Analytical Laboratories, Inc.

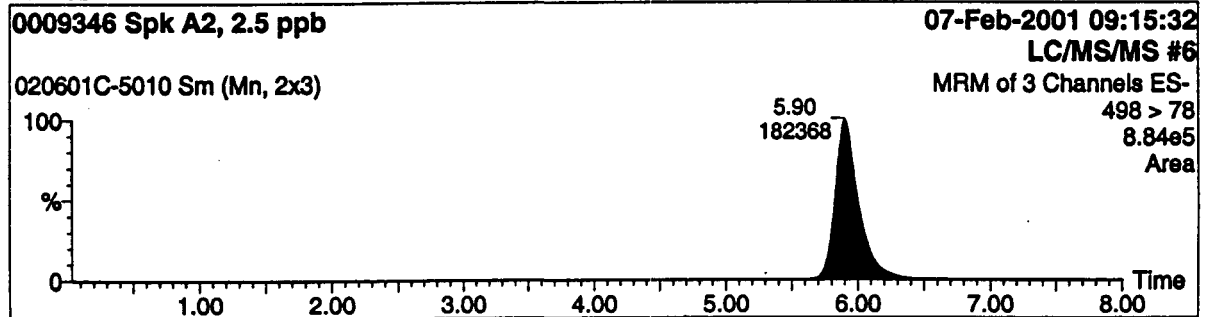
**Figure 10. Chromatogram Representing a Control Green Bean  
Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA  
(Centre ID: 0009346 Spk A2, Data Set: 020601C Bean)**

Name: 020601C-5010  
Text: 0009346 Spk A2, 2.5 ppb

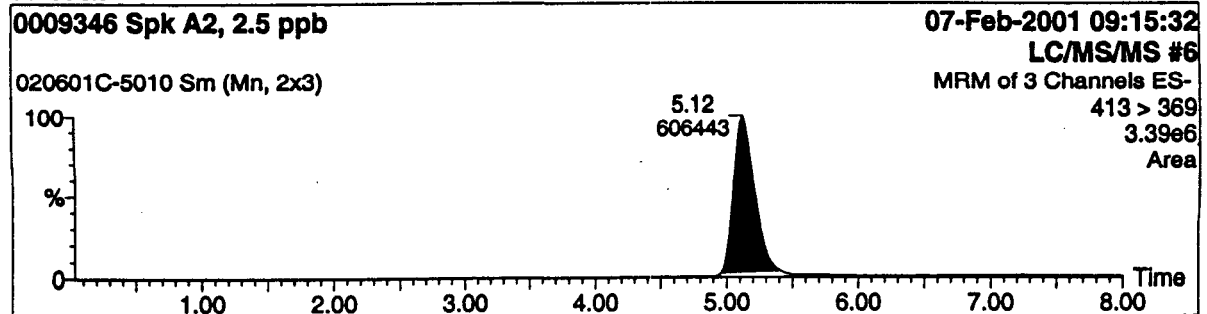
1: PFOS



2: FOSA



3: PFOA

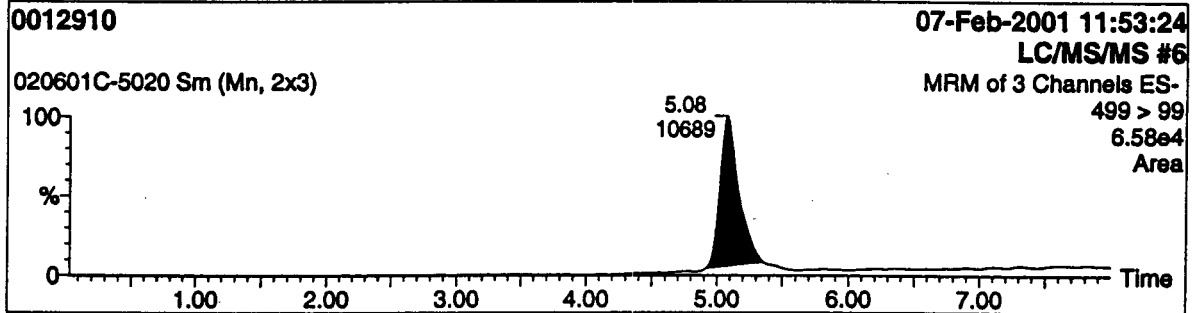


Centre Analytical Laboratories, Inc.

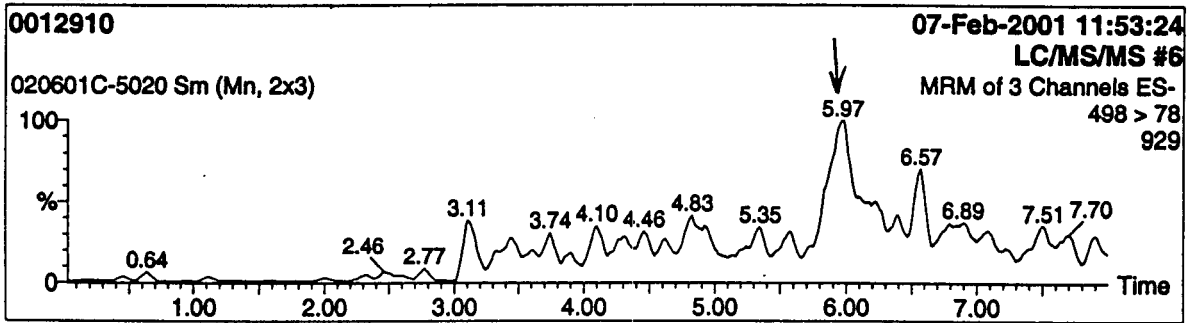
**Figure 11. Chromatogram Representing a Green Bean Sample (Centre ID: 0012910, Sponsor ID: Bruno's, 18341, Data Set: 020601C Bean)**

Name: 020601C-5020  
Text: 0012910

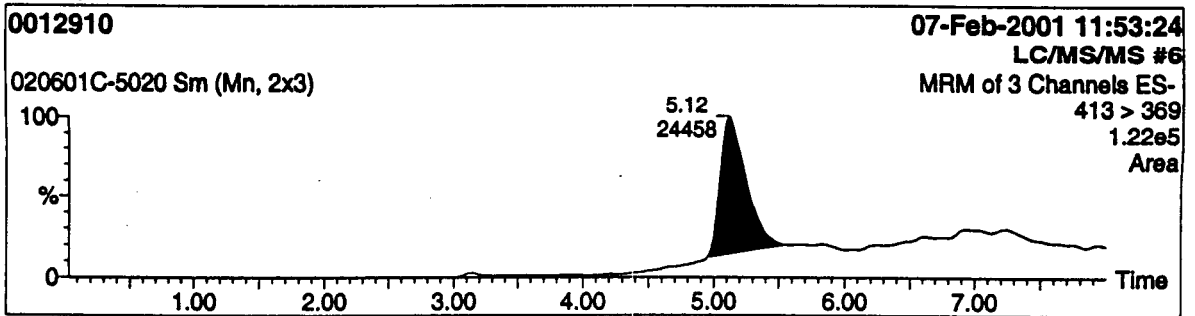
1: PFOS



2: FOSA



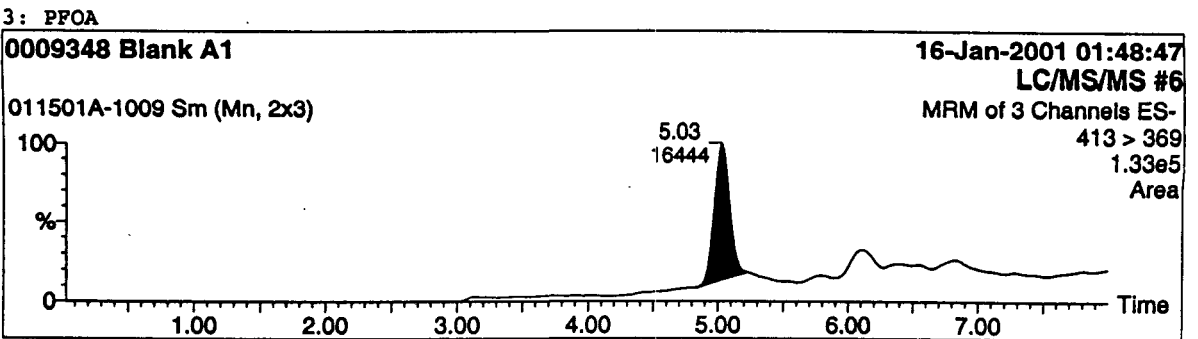
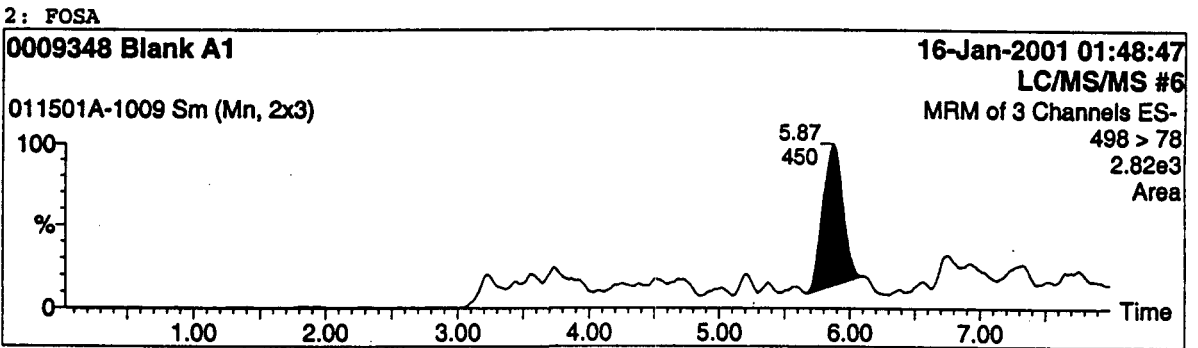
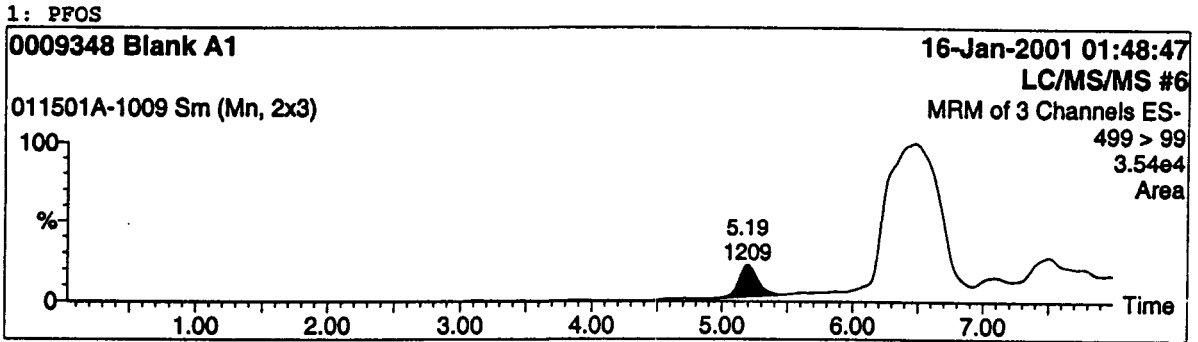
3: PFOA



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**Figure 12. Chromatogram Representing a Control Pork Muscle Sample (Centre ID: 0009348 Blank A1, Data Set: 011501A Pork)**

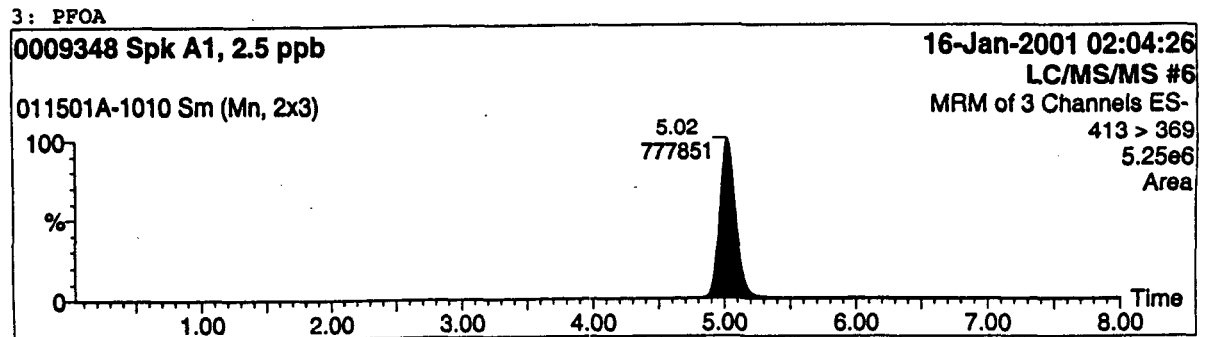
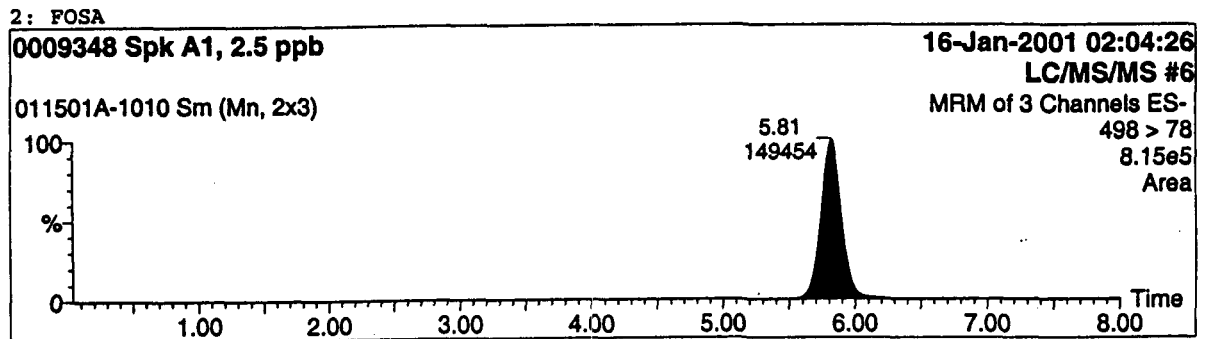
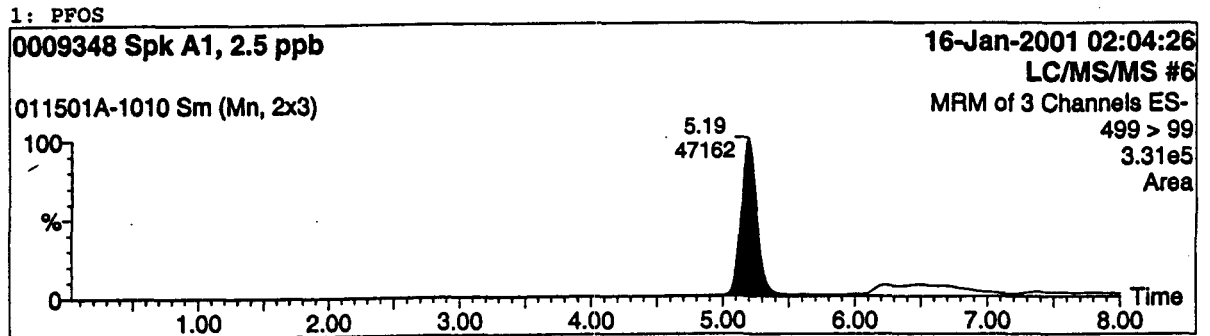
Name: 011501A-1009  
Text: 0009348 Blank A1



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**Figure 13. Chromatogram Representing a Control Pork Muscle  
Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA  
(Centre ID: 0009348 Spk A1, Data Set: 011501A Pork)**

Name: 011501A-1010  
Text: 0009348 Spk A1, 2.5 ppb

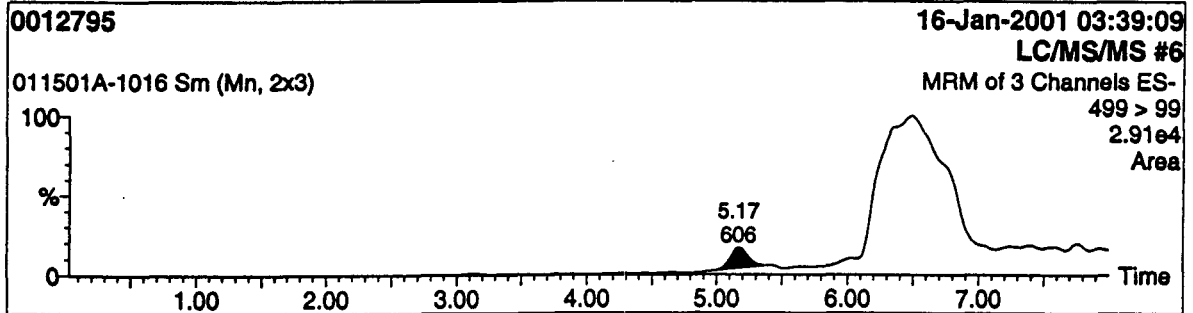


Centre Analytical Laboratories, Inc.

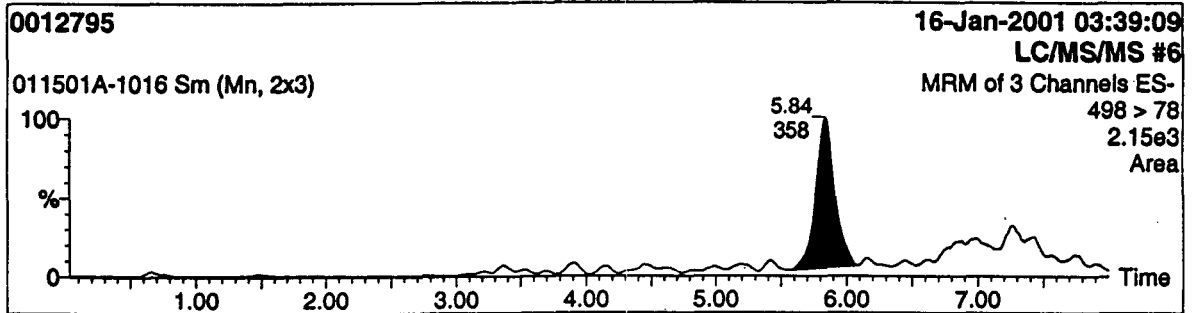
**Figure 14. Chromatogram Representing a Pork Muscle Sample  
(Centre ID: 0012795, Sponsor ID: Winn-Dixie, 18227, Data  
Set: 011501A Pork)**

Name: 011501A-1016  
Text: 0012795

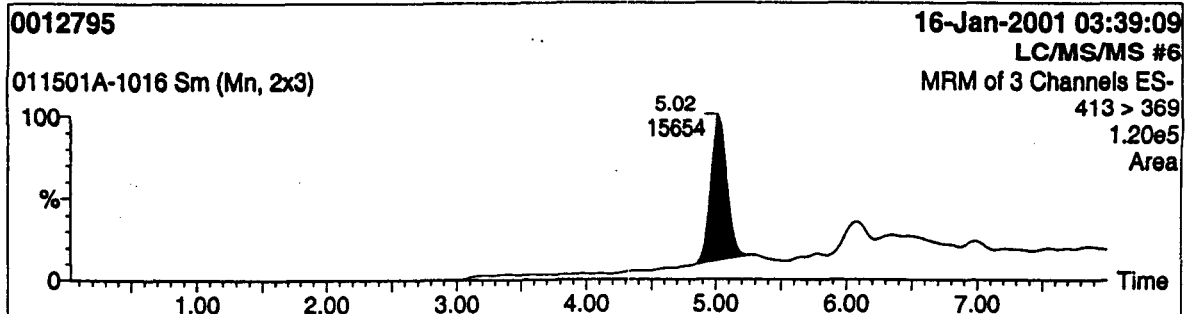
1: PFOS



2: FOSA



3: PFOA



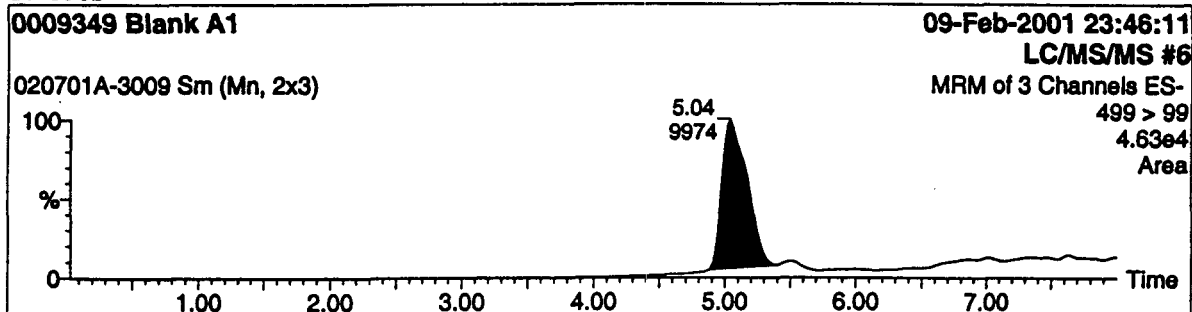
Centre Analytical Laboratories, Inc.



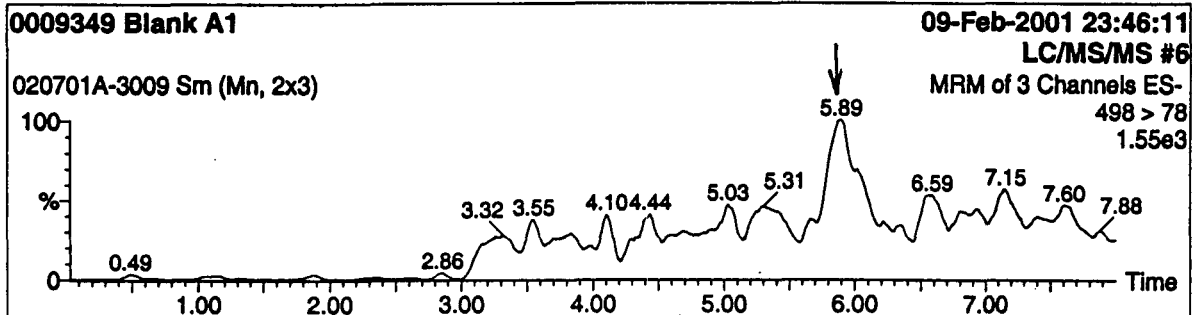
Figure 15. Chromatogram Representing a Control Milk Sample  
(Centre ID: 0009349 Blank A1, Data Set: 020701A Milk)

Name: 020701A-3009  
Text: 0009349 Blank A1

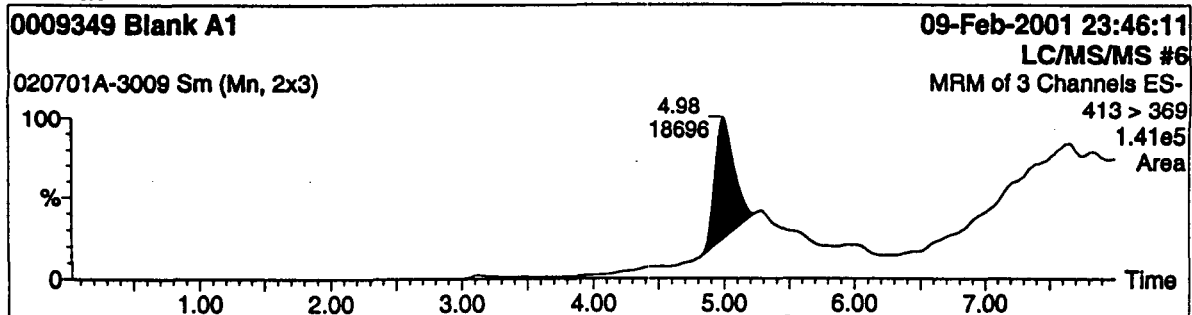
1: PFOS



2: FOSA



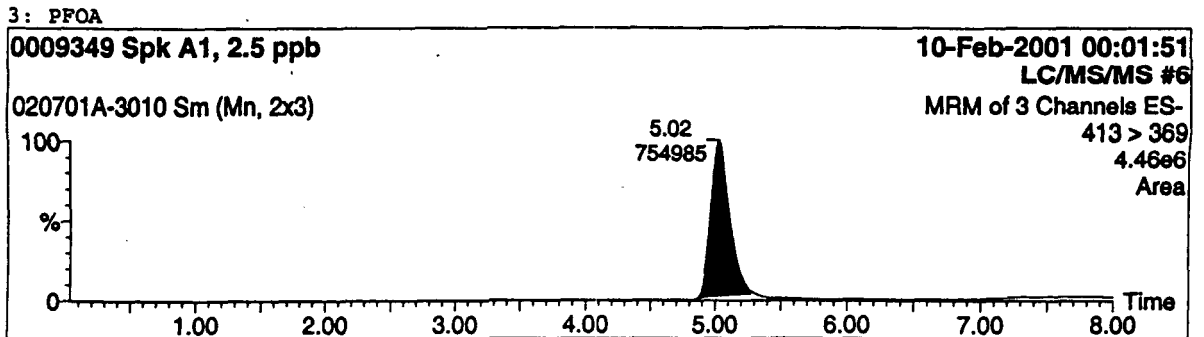
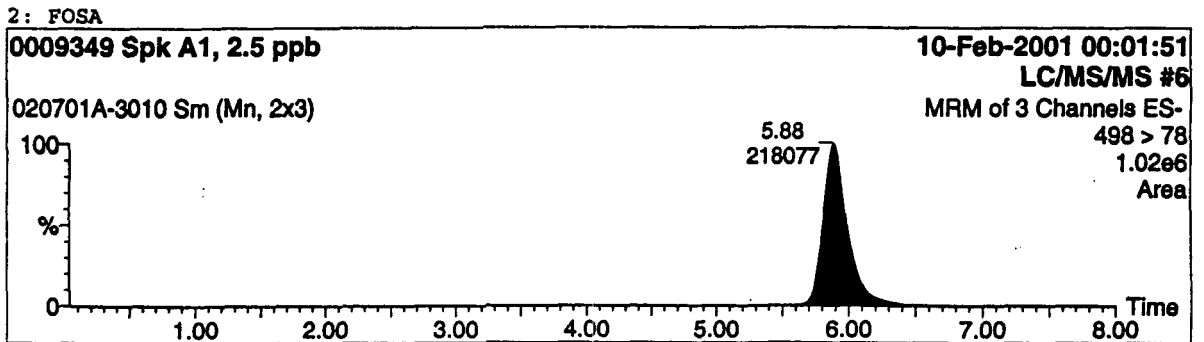
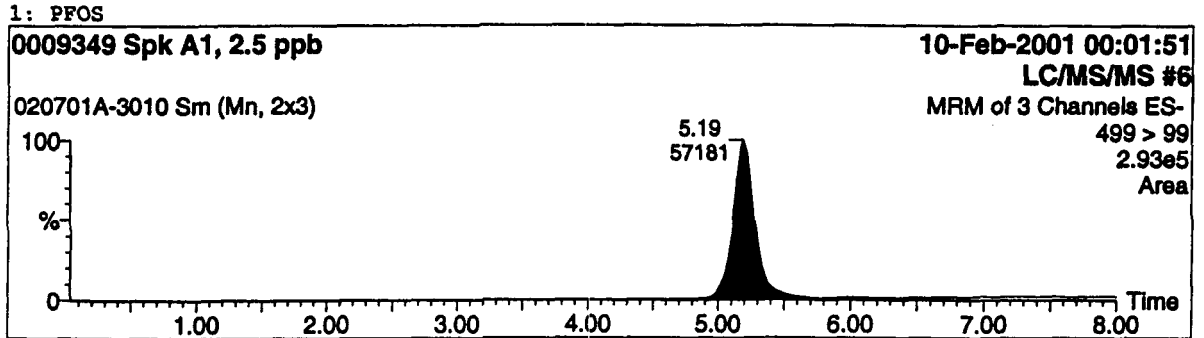
3: PFOA



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**Figure 16. Chromatogram Representing a Control Milk Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009349 Spk A1, Data Set: 020701A Milk)**

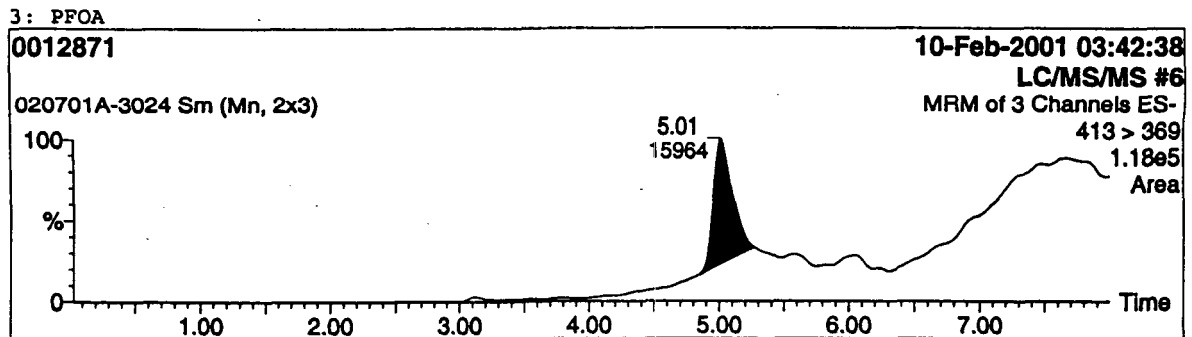
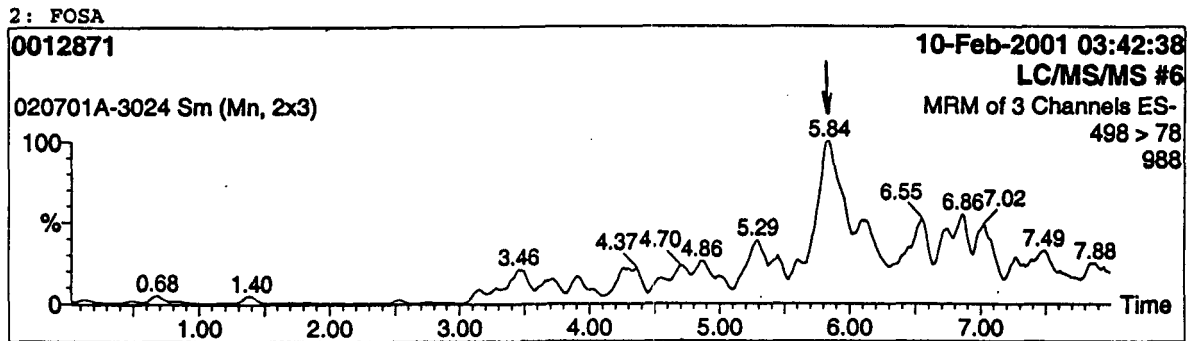
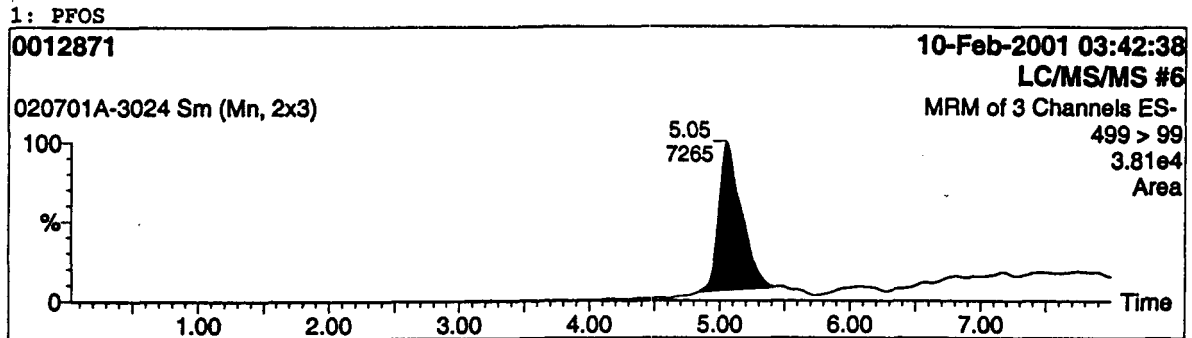
Name: 020701A-3010  
Text: 0009349 Spk A1, 2.5 ppb



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**Figure 17. Chromatogram Representing a Milk Sample (Centre ID: 0012871, Sponsor ID: Winn-Dixie, 18202, Data Set: 020701A Milk)**

Name: 020701A-3024  
Text: 0012871

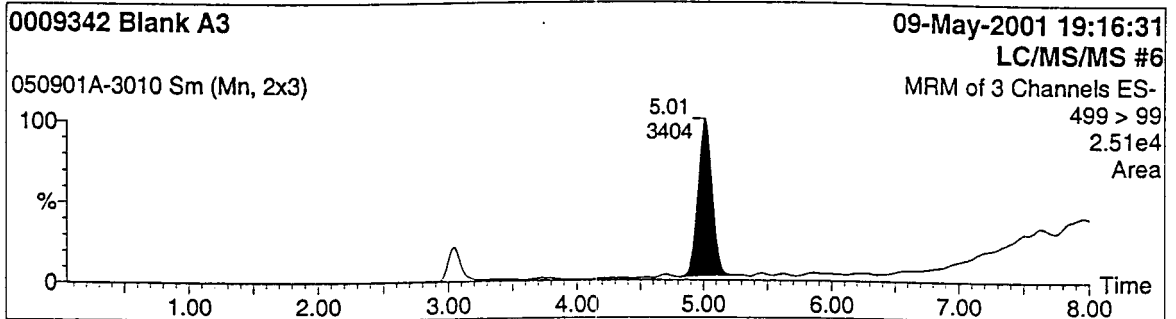


Centre Analytical Laboratories, Inc.

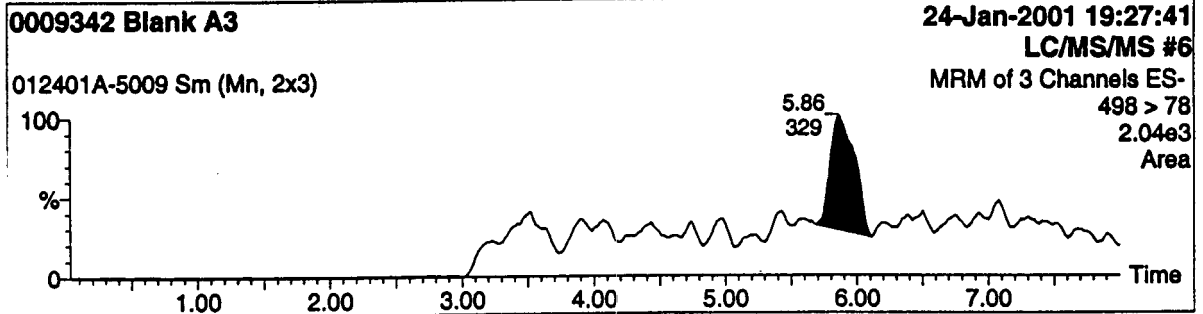
**Figure 18. Chromatogram Representing a Control Chicken Egg Sample (Centre ID: 0009342 Blank A3, Data Sets: 050901A Egg for PFOS, 012401A Egg for FOSA, and 052101A Egg for PFOA)**

Name: 050901A-3010  
Text: 0009342 Blank A3

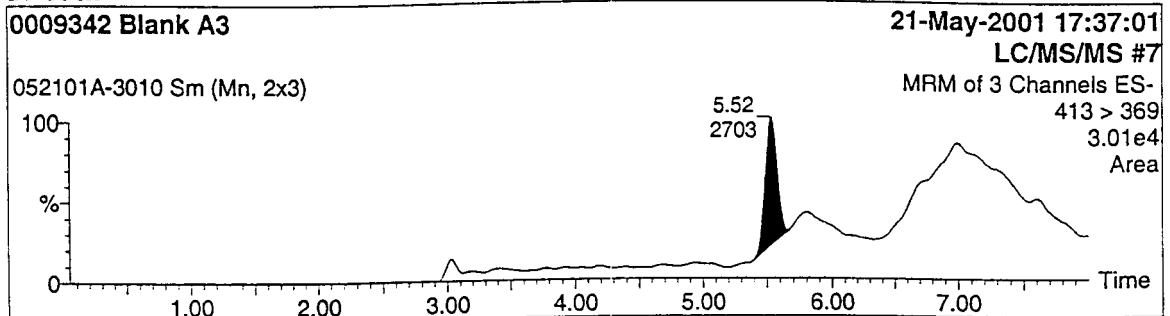
1: PFOS



2: FOSA



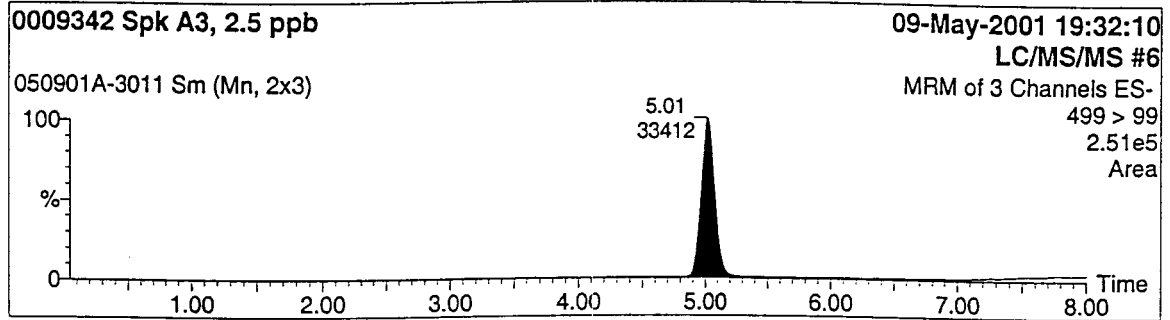
1: PFOA



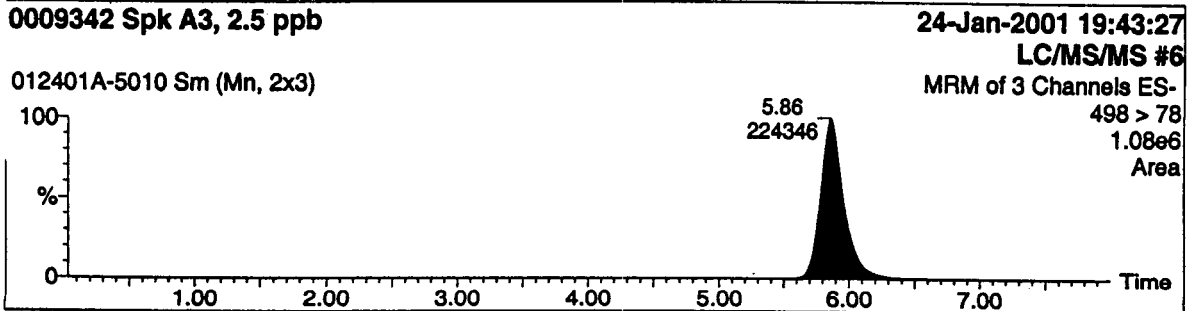
**Figure 19. Chromatogram Representing a Control Chicken Egg Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009342 Spk A3, Data Sets: 050901A Egg for PFOS, 012401A Egg for FOSA, and 052101A Egg for PFOA)**

Name: 050901A-3011  
Text: 0009342 Spk A3, 2.5 ppb

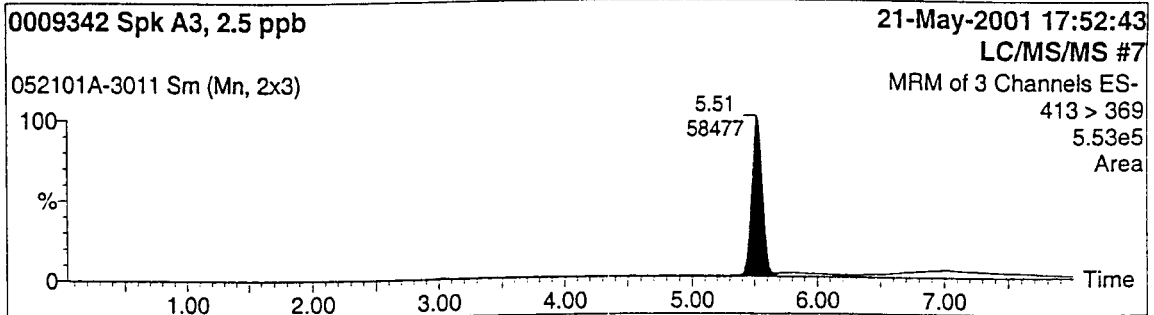
1: PFOS



2: FOSA

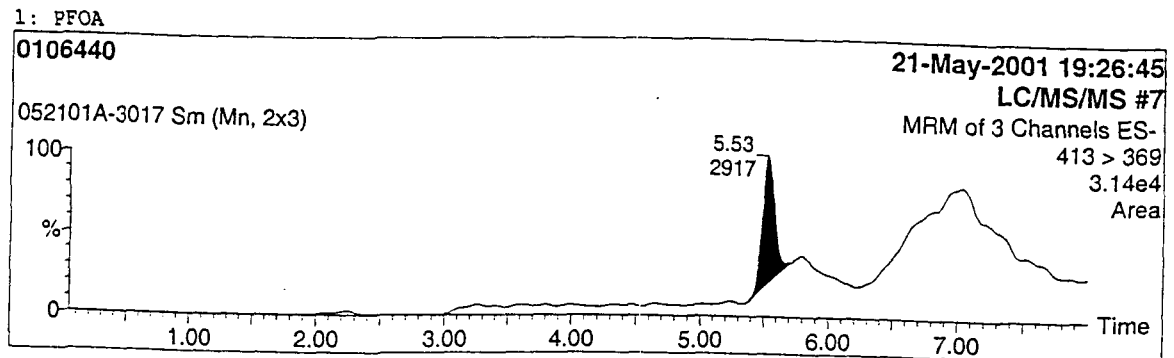
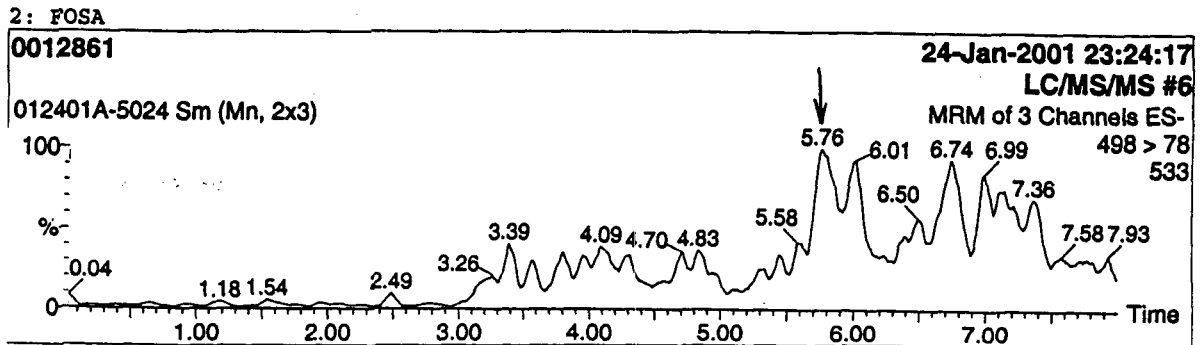
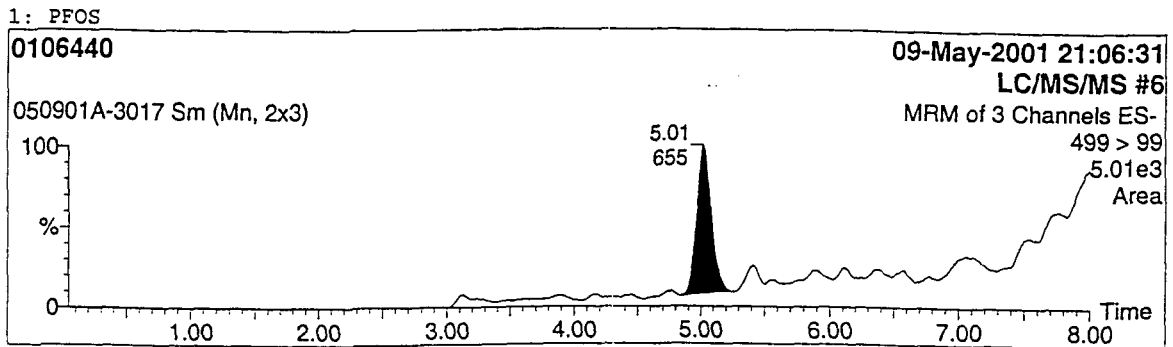


1: PFOA



**Figure 20. Chromatogram Representing a Chicken Egg Sample**  
(Centre ID: 0012861, Sponsor ID: Save, 18167 and 21226,  
Data Sets: 050901A Egg for PFOS, 012401A Egg for FOSA,  
and 052101A Egg for PFOA)

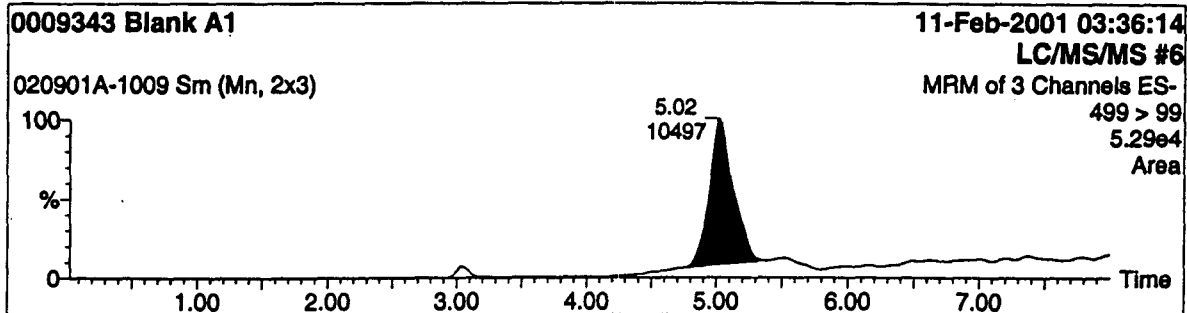
Name: 050901A-3017  
Text: 0106440



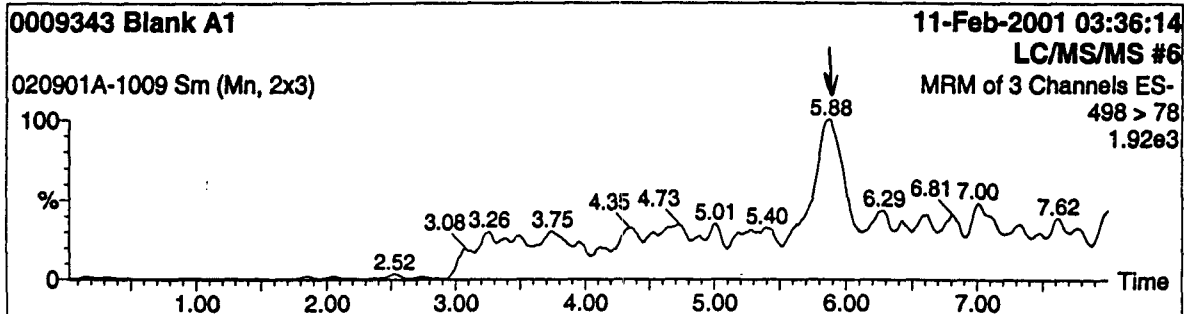
**Figure 21. Chromatogram Representing a Control Bread Sample  
(Centre ID: 0009343 Blank A1, Data Set: 020901A Bread)**

Name: 020901A-1009  
Text: 0009343 Blank A1

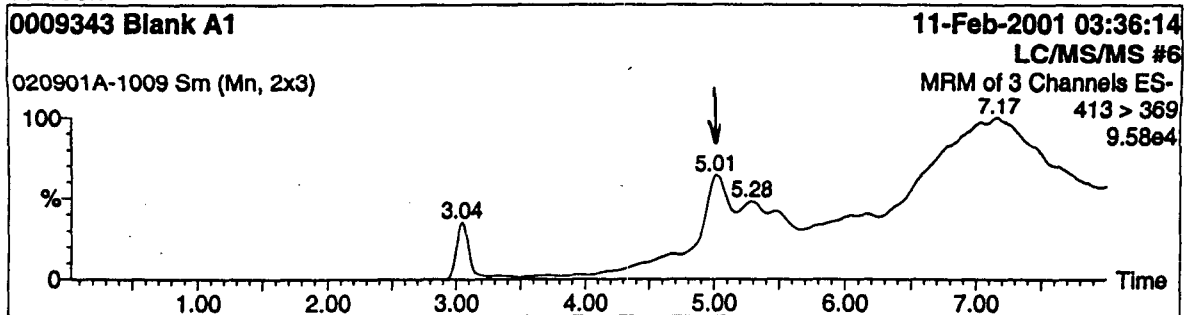
1: PFOS



2: FOSA



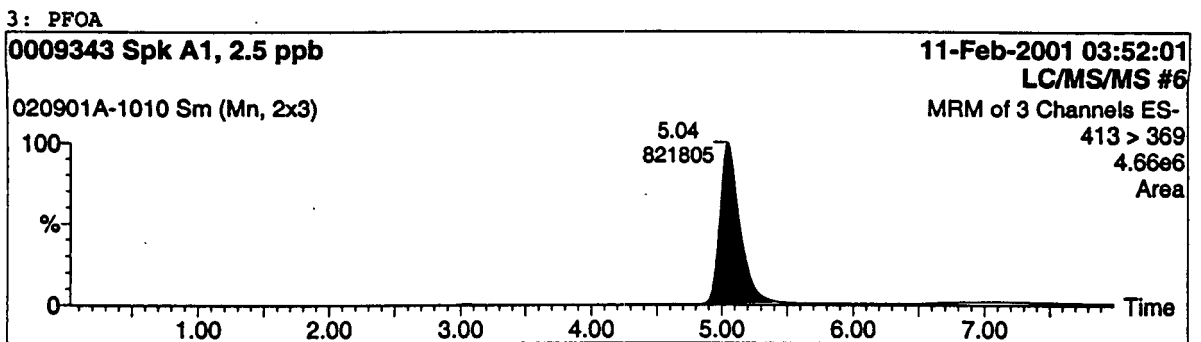
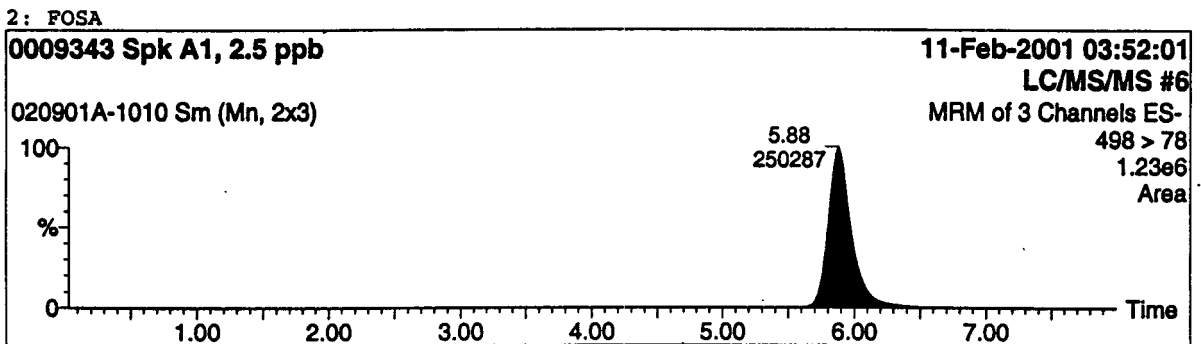
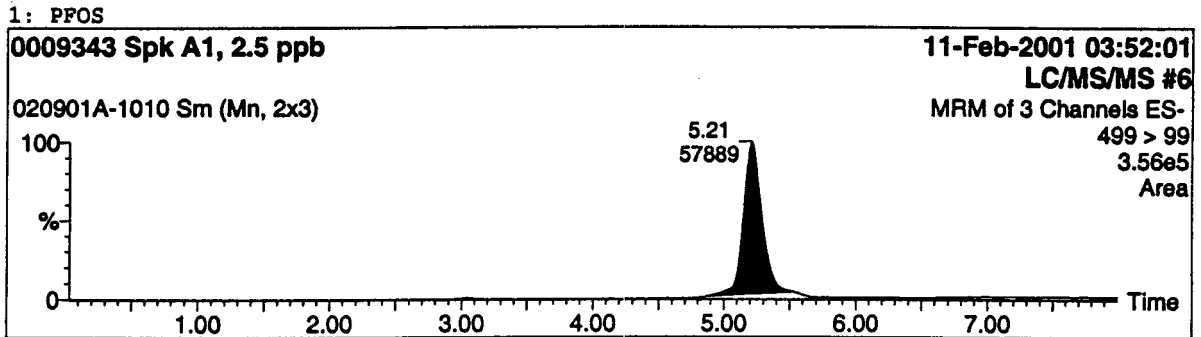
3: PFOA



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**Figure 22. Chromatogram Representing a Control Bread Sample Fortified with 2.5 ng/g of PFOS, FOSA and PFOA (Centre ID: 0009343 Spk A1, Data Set: 020901A Bread)**

Name: 020901A-1010  
Text: 0009343 Spk A1, 2.5 ppb



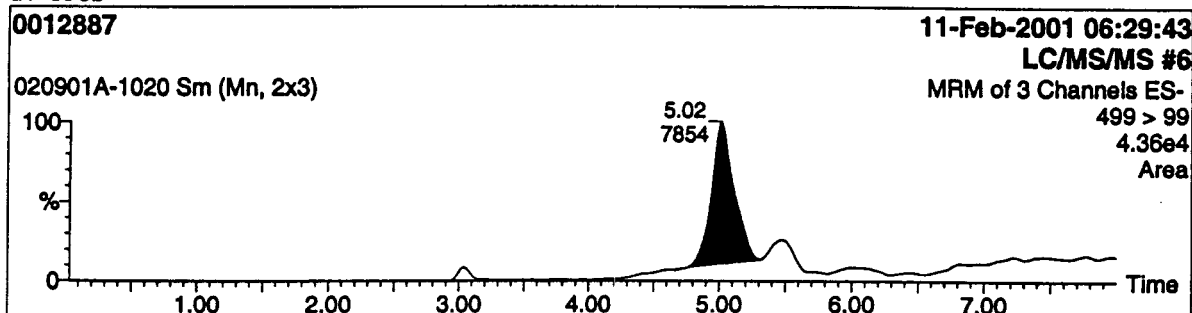
Centre Analytical Laboratories, Inc.



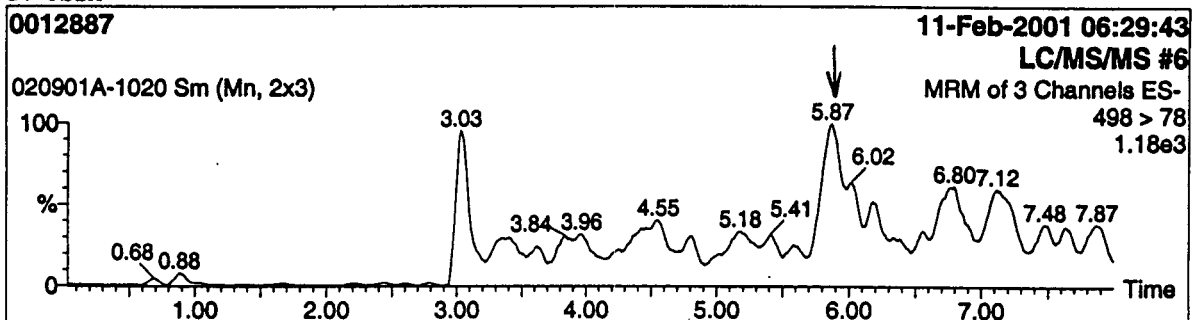
**Figure 23. Chromatogram Representing a Bread Sample (Centre ID: 0012887, Sponsor ID: Albertson's, 18244, Data Set: 020901A Bread)**

Name: 020901A-1020  
Text: 0012887

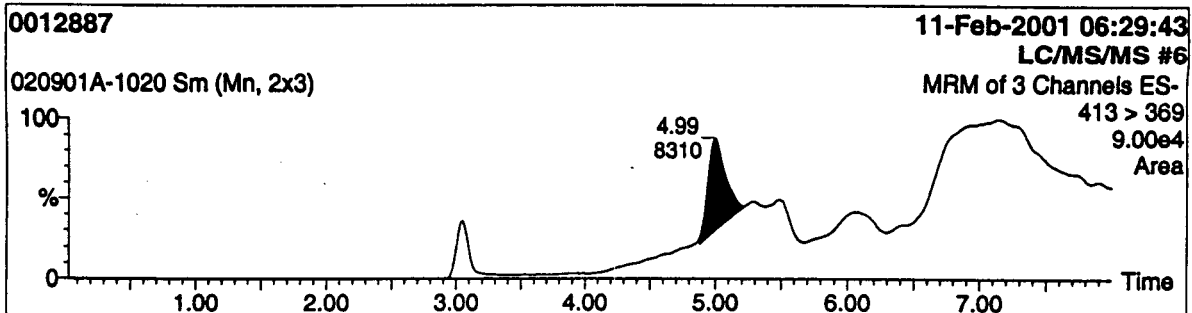
1: PFOS



2: FOSA



3: PFOA



Centre Analytical Laboratories, Inc.

# **13.0 APPENDICES**

# **APPENDIX A**

**Study Protocol  
00P-023-057  
(Centre Study No. 023-057),  
Amendments  
and  
Deviation**

## STUDY PROTOCOL

### STUDY TITLE

EXTRACTION OF PFOS, FOSA AND PFOA FROM VARIOUS FOOD MATRICES  
FOR ANALYSIS USING HPLC ELECTROSPRAY/MASS SPECTROMETRY

### SPONSOR

3M Environmental Technology and Safety Services  
Building 2-3E-09  
PO Box 33331  
St. Paul, MN 55133-3331

### DATA REQUIREMENTS

EPA TSCA Good Laboratory Practice Standards 40 CFR 792

### TESTING LABORATORY

Centre Analytical Laboratories, Inc. (Centre)  
3048 Research Drive  
State College, PA 16801  
Phone 814-231-8032

### PROTOCOL IDENTIFICATION NUMBER

00P-023-057

Title: EXTRACTION OF PFOS, FOSA AND PFOA FROM VARIOUS FOOD  
MATRICES FOR ANALYSIS USING HPLC ELECTROSPRAY/MASS  
SPECTROMETRY TABLE OF CONTENTS

	Page
STUDY TITLE .....	1
1. PURPOSE .....	3
2. TEST MATERIALS .....	3
3. SPONSOR.....	5
4. TESTING FACILITY AND PERFORMING LABORATORY .....	5
5. PROPOSED EXPERIMENTAL TIME-FRAME.....	5
6. TEST SYSTEM.....	5
7. JUSTIFICATION FOR THE SELECTION OF THE TEST SYSTEM.....	6
8. SAMPLE PROCESSING, STORAGE AND IDENTIFICATION .....	6
9. ANALYTICAL METHOD .....	6
10. EXPERIMENTAL DESIGN.....	7
11. STATISTICAL METHODS TO BE USED .....	7
12. PROTOCOL AMENDMENTS AND DEVIATIONS.....	7
13. RECORDS AND DATA REPORTING .....	8
14. QUALITY ASSURANCE .....	8
15. DATA AND REPORT.....	8
16. ARCHIVE STATEMENT .....	10
17. REFERENCES.....	10
18. PROTOCOL APPROVAL.....	11
APPENDIX: ANALYTICAL METHOD.....	12

Study title:

EXTRACTION OF PFOS, FOSA AND PFOA FROM VARIOUS FOOD MATRICES  
FOR ANALYSIS USING HPLC ELECTROSPRAY/MASS SPECTROMETRY

1. **PURPOSE**

The purpose of this study is to analyze PFOS, FOSA and PFOA in various food matrices using the analytical method entitled "Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples, Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hotdogs, and Catfish by LC/MS/MS". A copy of the method is appended to this protocol (Appendix).

The Quality Assurance Unit of Centre Analytical Laboratories, Inc. will audit the study for compliance with EPA TSCA Good Laboratory Practice standards 40 CFR 792 (1).

2. **TEST MATERIALS**

The following analytical standards will be used:

Test Material	Lot or TCR Number	Purity (%)	Expiration Date
PFOS	TCR00017-46	97.9	08/31/01
PFOA	332	TBD	07/06/01
FOSA	L-15709	95.1	02/15/01

TBD= To be determined

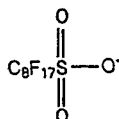
Note: GLP characterization, solubility and stability data for PFOA and FOSA are unavailable at this time. However, available characterization data for the test materials will be supplied to the testing facility (Centre) by the sponsor (3M). Therefore, do not correct for percent purity when preparing standard solutions for these two fluorochemicals.

Centre Protocol No. 00P-023-057

Chemical names and structures of the compounds are presented below.

**PFOS**

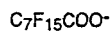
Chemical Name: 1-Octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptafluoro-, potassium salt.  
Molecular Weight: 499 (C<sub>8</sub>F<sub>17</sub>SO<sub>3</sub><sup>-</sup>)  
CAS Number: 2795-39-3



Note: The neutral molecule and standard form which PFOS (anion) is derived from is perfluorooctanesulfonate potassium salt [C<sub>8</sub>F<sub>17</sub>SO<sub>3</sub>K], molecular weight 538.

**PFOA**

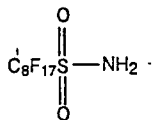
Chemical Name: Octanoic acid, pentafluoro-, ammonium salt  
Molecular Weight: 413  
CAS Number: 3825-26-1 (FC-143)



Note: The neutral molecule and standard form which PFOA (anion) is derived from is perfluorooctanoate ammonium salt [C<sub>7</sub>F<sub>15</sub>COONH<sub>4</sub>], molecular weight 431.

**FOSA**

Chemical Name: 1-Octanesulfonamide, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptafluoro-  
Molecular Weight: 499  
CAS Number: 754-91-6



A record of test and reference substance receipt, storage conditions, and a record of use will be maintained at Centre Analytical Laboratories, Inc. Chain-of-custody forms and shipping documents for tracking of the test substances will be included as part of the raw data package.

Centre Protocol No. 00P-023-057

All standards/test substances and any prepared solutions must be identified with a unique label or number on the container or cross-referenced to the container.

HAZARD INFORMATION

A current MSDS for the chemical(s) used in this study will be maintained at the testing facility.

3. **SPONSOR**

3M Environmental Technology and Safety Services  
Building 2-3E-09  
PO Box 33331  
St. Paul, MN 55133-3331

STUDY DIRECTOR:  
Susan A. Beach, 3M  
TELEPHONE: 651-778-7452  
FAX NUMBER: 651-778-6176

4. **TESTING FACILITY AND PERFORMING LABORATORY**

Centre Analytical Laboratories, Inc. (Centre)  
3048 Research Drive  
State College, PA 16801

PRINCIPAL INVESTIGATOR:  
Karen Smith, Centre  
TELEPHONE: 814-231-8032  
FAX NUMBER: 814-231-1580

5. **PROPOSED EXPERIMENTAL TIME-FRAME**

Experimental Start Date	January 8, 2001
Experimental Termination Date	February 8, 2001
Report Issued	March 8, 2001

6. **TEST SYSTEM**

Food samples obtained by Centre Analytical Laboratories, Inc. and used in the validation study (Centre Study No. 023-043) will be used as the control samples. Samples will be collected from local grocery stores from six test cities outlined in



Centre Protocol No. 00P-023-057

3M Quality Assurance Project Plan for Empirical Human Exposure Assessment Multi-City Study Sampling Task. All food samples obtained from grocery stores in the six test cities by the sponsor will be shipped to Centre Analytical Laboratories, Inc. for analysis.

**7. JUSTIFICATION FOR THE SELECTION OF THE TEST SYSTEM**

Representative samples such as, honey, vegetable (green beans, carrot, radish, lettuce, potato), fruit (apple), livestock (pork muscle, cow milk), poultry (chicken muscle, egg), fish and processed foods (bread, hot dogs), will be collected by the sponsor and shipped to Centre for trace-level analysis of fluorochemical residues. The cities chosen as the collection sites either have major fluorochemical production facilities, major fluorochemical users, or no major fluorochemical production or usage facilities. The matrices were chosen to determine if fluorochemicals are being transferred from the environment to food sources.

**8. SAMPLE PROCESSING, STORAGE AND IDENTIFICATION**

All food samples will be processed according to the method entitled, "Method of Analysis for the Determination of Fluorochemicals In Green Beans, Apples, Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hotdogs, and Catfish by LC/MS/MS."

Upon receipt at Centre, the food samples will be assigned unique sample identification numbers, which will be used for tracking and identification of the samples. The samples will be stored in a temperature monitored freezer, maintained at  $< -10^{\circ}$  C, except when removed for analysis as described in the method. The samples will be kept isolated from the test substance during storage.

Sample receipt and storage location and conditions during the study must be documented. All samples and any resulting sample extracts must be identified with a unique label or sample number. Such identification will be either on the container or cross-referenced to the container.

**9. ANALYTICAL METHOD**

The samples will be analyzed according to the analytical method entitled "Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples, Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hotdogs, and Catfish by LC/MS/MS" (see Appendix).

10. **EXPERIMENTAL DESIGN**

For each matrix, a set of samples will be extracted in duplicate and analyzed. Each set of samples analyzed will contain at least one matrix control and two matrix control samples fortified at known concentrations and carried through the extraction procedure to verify recovery.

Methods to control bias will include assay of untreated control samples, fortification of untreated control samples to obtain recovery data, and replicate analysis of fortified samples to provide an indication of reproducibility.

11. **STATISTICAL METHODS TO BE USED**

Standard (linear, 1/x weighted) curves used to determine the concentration of the compound being analyzed will be constructed from a minimum of six concentrations. Quantification of residues will only occur within the range of the standard curve (residues in control samples may be calculated up to 20% of the LOQ [20% of LOQ = 0.1 ppb] by extrapolation of the calibration curve, in order to correct fortification recoveries). The correlation coefficient (r) for calibration curves generated must be greater than or equal to 0.9925 ( $r^2 \geq 0.985$ ).

Overall averages, standard deviations and relative standard deviations of fortification recoveries will be calculated.

Other than these descriptive statistics, no other statistical methods will be used during the course of this study.

12. **PROTOCOL AMENDMENTS AND DEVIATIONS**

Any deviations from the protocol or from the analytical method as provided will be documented and reported promptly to the Study Director. Planned changes to this protocol or to the analytical method will be made in writing as an amendment or modification and approved by the Principal Investigator and the Study Director. Any amendments will be appended to this protocol and included in the final report.

1. Protocol amendments: Planned changes to the approved protocol shall be documented by amendments that clearly describe the change, justification for the change, and impact on the study. Amendments will be signed and dated by the Principal Investigator and Study Director. Copies of amendments will be sent to the quality assurance unit.
2. Protocol deviations: Protocol deviations, which are one time and unplanned deviations from the protocol shall be documented in the study records, noting the nature of the deviation, potential effect or impact on the study, and corrective action if required. Protocol deviations are signed by the Principal Investigator and Study Director and reviewed by QAU.

**13. RECORDS AND DATA REPORTING**

Records to be maintained include, but are not limited to the following (as appropriate):

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

All chromatograms will contain the following:

- a. Sample identification, date, arrow or other indication of the area of interest, and injection number corresponding to the run.
- b. Additionally, fortifications will include the fortification level of each analyte.
- c. Analytical standard chromatograms will additionally include the concentration (e.g.,  $\mu\text{g/ml}$ ,  $\text{ng/mL}$ , ppb, ppt, etc.).

Each data set will contain information on temperatures, flow rates, column parameters, gases, instrument parameters, and instrument type, etc. if any of these differ from the method. The Centre study number will also be recorded on the first chromatogram of each daily analytical run.

**14. QUALITY ASSURANCE**

Centre QA Unit will review the protocol and audit the study conduct and the study documentation (including raw data and final report) to ascertain that all QA/GLP procedures are adhered to. Centre QA Unit will inspect the study at intervals adequate to assure compliance to GLP's, and will report the findings of the audits to the Principal Investigator, Centre Management, and the Study Director.

**15. DATA AND REPORT**

1. All raw data and the original signed protocol will be maintained in the study file. This data includes the protocol amendments, protocol deviations, laboratory notebooks, analytical standard solution preparation, sample chain of custody sheets, sample work sheets, chromatograms, calibration curves, and any other appropriate data generated. Raw data not used will be stored in the study file. The reason for exclusion will be documented.

Centre Protocol No. 00P-023-057

2. A final report will be issued by Centre, approved and signed by the Study Director and sufficient for submission to EPA. The report contents should include, but are not limited to:
  1. Objectives and procedures stated in the protocol
  2. Analytical and statistical methods used
  3. Test materials identified by name, lot, purity and other characteristics
  4. Name of testing facility and study initiation and completion dates
  5. Statement prepared and signed by the quality assurance unit
  6. Tables containing all applicable data
  7. All chromatographic and instrumental conditions
  8. A complete listing of Centre study personnel

Centre will send a copy of the draft report to the Study Director, who will return the report with comments. Centre Analytical will make revisions and finalize the report with the approval of the Study Director. The Centre QA unit will conduct an audit of the final draft report and data files to assure accuracy and GLP compliance. The final report and copies of the raw data must be approved by the Study Director prior to signature. A statement of inspections and a statement of compliance will be included in the final report.

Any corrections or additions to the final report shall be in the form of an amendment by the Principal Investigator. The amendment shall clearly identify the part of the final report that is being corrected and the justification for correction. The amendment shall be signed and dated by the Principal Investigator and the Study Director.

**REPORT DISTRIBUTION:**

Original: Study Director  
Copy: Centre Archives

Centre Protocol No. 00P-023-057

**16. ARCHIVE STATEMENT**

Study records to be maintained: Records to be maintained for the study include all raw data, observations recorded during the conduct of the study, documentation, chromatograms, specimens, and study related correspondence. This includes a description of equipment used during the conduct of the study. All characterization data and any shipping records shall be retained.

Document archives: Upon completion of the study, the study records, protocol and amendments, and the final report and amendments shall be retained in the 3M document archives. If it is necessary to substitute a copy for an original record, it will be certified as an exact copy. Centre will retain facility-related originals and a copy of the final report and raw data package.

**17. REFERENCES**

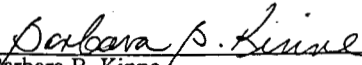
1. U.S. Environmental Protection Agency, Toxic Substances Control Act (TSCA). Good Laboratory Practice Standards. Final Rule, 40 CFR Part 792.

Centre Protocol No. 00P-023-057

18. PROTOCOL APPROVAL

This protocol was audited by the Quality Assurance Unit of Centre Analytical Laboratories, Inc.

Quality Assurance Reviewer, Centre

  
\_\_\_\_\_  
Barbara B. Kinne  
Quality Assurance Auditor

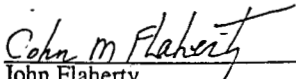
01/03/01  
Date

Principal Investigator, Centre

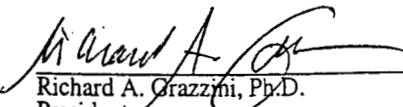
  
\_\_\_\_\_  
Karen Smith  
Scientist

01/03/01  
Date

Facility Management, Centre

  
\_\_\_\_\_  
John Flaherty  
Laboratory Manager

01/03/01  
Date

  
\_\_\_\_\_  
Richard A. Grazzini, Ph.D.  
President

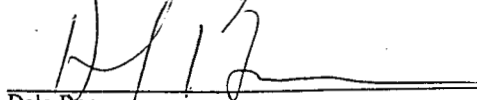
03-JAN-01  
Date

Study Director, 3M

  
\_\_\_\_\_  
Susan A. Beach

01/09/01  
Date

Sponsor Management, 3M

  
\_\_\_\_\_  
Dale Bacon  
Technical Director

01/09/01  
Date

## **APPENDIX: ANALYTICAL METHOD**

METHOD OF ANALYSIS FOR THE DETERMINATION OF  
FLUORO-CHEMICALS IN GREEN BEANS, APPLES, PORK MUSCLE,  
COW MILK; CHICKEN MUSCLE, CHICKEN EGGS, BREAD,  
HOTDOGS, AND CATFISH BY LC/MS/MS.

Centre Protocol No. 00P-023-057

TITLE

Method of Analysis for the Determination of Fluorochemicals in Green Beans, Apples,  
Pork Muscle, Cow Milk, Chicken Muscle, Chicken Eggs, Bread, Hotdogs, and Catfish by  
LC/MS/MS

AUTHORS

Enaksha Wickremesinha, Shaozhi Zheng and John Flaherty

DATE ISSUED

September 13, 2000

SPONSOR

3M Environmental Technology and Safety Services  
Building 2-3E-09  
PO Box 33331  
St. Paul, MN 55133-3331

PERFORMING LABORATORY

Centre Analytical Laboratories, Inc. (Centre)  
3048 Research Drive  
State College, PA 16801

CENTRE STUDY NUMBER

023-001

TOTAL NUMBER OF PAGES

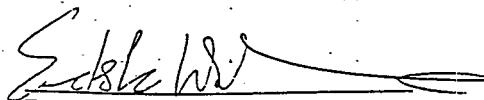
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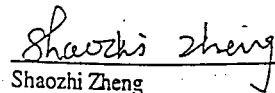


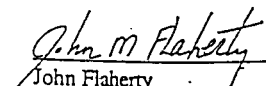
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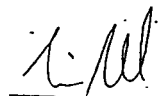
MANAGEMENT APPROVAL

As per 40 CFR 160.3, method development is not required to be conducted in compliance with GLP's. However, the work was in general conformance with applicable standard operating procedures and general GLP regulations.

 Sept 13, 2000  
Enaksha Wickremesinhe, Ph.D. Date  
Author, Principal Investigator  
Centre Analytical Laboratories, Inc

 9-13-00  
Shaozhi Zheng Date  
Author, Principal Investigator  
Centre Analytical Laboratories, Inc.

 9/13/00  
John Flaherty Date  
Laboratory Manager  
Centre Analytical Laboratories, Inc.

 for Rick Grazzini 9/13/00  
Richard A. Grazzini, Ph.D. Date  
President  
Centre Analytical Laboratories, Inc.

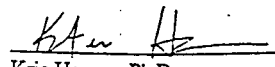
 Sept. 14, 2000  
Kris Hansen, Ph.D. Date  
Sponsor Representative  
3M Environmental Laboratory

TABLE OF CONTENTS

TITLE PAGE ..... 1  
MANAGEMENT APPROVAL ..... 2  
TABLE OF CONTENTS ..... 3  
LIST OF TABLES ..... 4  
LIST OF FIGURES ..... 5  
1. SUMMARY ..... 7  
2. EXPERIMENTAL COMPOUNDS ..... 8  
3. CHEMICALS AND SUPPLIES ..... 9  
    3.1. CHEMICALS ..... 9  
    3.2. STANDARDS ..... 9  
    3.3. EQUIPMENT AND SUPPLIES ..... 10  
    3.4. SOLUTIONS ..... 11  
    3.5. PREPARATION OF STOCK, FORTIFICATION, AND CALIBRATION SOLUTIONS ..... 11  
        3.5.1. Stock Solutions ..... 11  
        3.5.2. Fortification Solutions ..... 11  
        3.5.3. Calibration Standards ..... 12  
4. METHOD ..... 12  
    4.1. FLOW DIAGRAM ..... 12  
    4.2. SAMPLE PROCESSING ..... 13  
    4.3. EXTRACTION ..... 13  
        4.3.1. Apple, Green Beans, Bread, Hot Dog, and Pork ..... 13  
        4.3.2. Chicken and Milk ..... 14  
        4.3.3. Egg and Catfish ..... 15  
        4.3.4. SPE Column Preparation ..... 16  
        4.3.5. Silanization of Pear-Shaped Flasks ..... 16  
        4.3.6. Standardization of SPE Columns ..... 16  
    4.4. ANALYSIS BY LC/MS/MS ..... 16  
        4.4.1. LC/MS/MS System and Operating Conditions (Electrospray) ..... 16  
        4.4.2. Example Tune File Parameters ..... 18  
        4.4.3. Calibration Procedures ..... 19  
        4.4.4. Sample Analysis ..... 19  
    4.5. PERFORMANCE CRITERIA ..... 20  
    4.6. TIME REQUIRED FOR ANALYSIS ..... 21  
5. CALCULATIONS ..... 21  
    5.1. COMPONENT RESIDUE CONCENTRATION ..... 21  
    5.2. FORTIFICATION RECOVERY ..... 22  
6. SAFETY ..... 22

LIST OF TABLES

Table 1: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH,PFOS, POAA and PFOSA in Whole Apple .....	24
Table 2: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Green Beans .....	25
Table 3: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Chicken Eggs .....	26
Table 4: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Chicken Muscle.....	27
Table 5: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Cow Milk .....	28
Table 6: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Bread.....	29
Table 7: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Hot Dog.....	30
Table 8: Summary of Recoveries for PFOS, POAA and PFOSA in Catfish .....	31
Table 9: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH,PFOS, POAA and PFOSA in Pork Muscle .....	32

LIST OF FIGURES

Figure 1: Representative Chromatograms of a 0.5 ng/mL Mixed Calibration Standard of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA..... 34

Figure 2: Representative Chromatograms of a Control Whole Apple Sample (Set 111799 Apple, Centre ID 983732 Apple Blank A) ..... 36

Figure 3: Representative Chromatograms from the Injection of Control Whole Apple Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111799 Apple, Centre ID 983732 Spk A) ..... 38

Figure 4: Representative Chromatograms of a Control Green Bean Sample (Set 111299 Beans, Centre ID 9908032 Green Beans Blank A)..... 40

Figure 5: Representative Chromatograms from the Injection of Control Green Beans Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111299 Beans, Centre ID 9908032 Spk B)..... 42

Figure 6: Representative Chromatograms of a Control Chicken Egg Sample (Set 122999 Egg, Centre ID 9912031 Egg Blank A) ..... 44

Figure 7: Representative Chromatograms from the Injection of Control Chicken Egg Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 122999 Egg, Centre ID 9912031 Spk B) ..... 46

Figure 8: Representative Chromatograms of a Control Chicken Muscle Sample (Set 111999 Chicken, Centre ID 9910292 Chicken Blank B)..... 48

Figure 9: Representative Chromatograms from the Injection of Control Chicken Muscle Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111999 Chicken, Centre ID 9910292 Spk A)..... 50

Figure 10: Representative Chromatograms of a Control Cow Milk Sample (Set 110999 Milk, Centre ID 972627 Milk Blank B)..... 52

Figure 11: Representative Chromatograms from the Injection of Control Cow Milk Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 110999 Milk, Centre ID 972627 Spk A) ..... 54

Figure 12: Representative Chromatograms of a Control Bread Sample (Set 111999 Bread, Centre ID 9908031 Bread Blank A)..... 56

Figure 13: Representative Chromatograms from the Injection of Control Bread Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111999 Bread, Centre ID 9908031 Spk A)..... 58

Figure 14: Representative Chromatograms of a Control Hot Dog Sample (Set 110899 Hot Dog, Centre ID 9908035 Hot Dog Blank B) ..... 60

Figure 15: Representative Chromatograms from the Injection of Control Hot Dog Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 110899 Hot Dog, Centre ID 9908035 Spk B) ..... 62

Figure 16: Representative Chromatograms of a Control Catfish Sample (Set 012400 Catfish, Centre ID 9912032 Catfish Blank A)..... 64

LIST OF FIGURES (CONTINUED)

Figure 17: Representative Chromatograms from the Injection of Control Catfish Fortified at 500 ppt each of PFOS, POAA, and PFOSA (Set 012400 Catfish, Centre ID 9912032 Spk A)..... 65

Figure 18: Representative Chromatograms of a Control Pork Muscle Sample (Set 122199 Pork, Centre ID 9911783 Pork Blank B)..... 66

Figure 19: Representative Chromatograms from the Injection of Control Pork Muscle Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 122199 Pork, Centre ID 9911783 Spk B)..... 68

## 1. SUMMARY

This document details a method of analysis for residues of 2(N-ethylperfluorooctane-sulfonamido)-ethyl alcohol (Et-FOSE-OH), 2(N-methylperfluorooctanesulfonamido) ethyl alcohol (Me-FOSE-OH), perfluorooctane sulfonate (PFOS), perfluorooctanoate (POAA), and perfluorooctane sulfonamide (PFOSA), in green beans, apples, pork muscle, cow milk, chicken muscle, chicken eggs, bread, and hotdogs, and PFOS, POAA and PFOSA in catfish. The chemical formulas of the analytes are given in Section 2 of this method.

Residues of these five fluorochemicals are extracted from each matrix with acetonitrile (ACN). The ACN extract is passed through a multi-layered (florisil, silica gel, carbon and amino) solid-phase extraction (SPE) column. Approximately 2-3 drops of 1-octanol are added to the extract and evaporated to near dryness using rotary evaporation. Each extract is then reconstituted with methanol. Quantification of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA is accomplished by liquid chromatography/tandem mass spectrometry (LC/MS/MS) analysis using selected reaction monitoring (SRM).

The proposed limit of quantitation, LOQ; is the lowest fortification specified by the method which gives adequate recovery according to EPA guidelines [OPPTS 860.1340 (c) (2)] and for this method is 0.5 parts-per-billion (ppb) each for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA. This is based on studies conducted with whole apple, green bean, bread, chicken egg, chicken muscle, pork muscle, cow milk, hot dog, and catfish control samples during the development of this method.

The theoretical limit of detection (LOD) will be based on the signal to noise ratio and will be at least greater than 3 times the level of noise, based on the instrumentation system used. For all five analytes (Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA), the lowest analytical standard corresponds to 0.5 ng/mL, which is equivalent to 0.2 ng/g (0.2 ppb) of matrix.

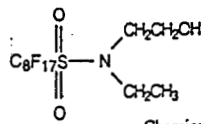
Recoveries for all matrices can be found in Tables 1-9. Lower recoveries for Me-FOSE-OH and broad peak shapes for Et-FOSE-OH have been observed in some animal matrices (chicken, pork and chicken egg).

NOTE: Perfluorononane sulfonate (PFNS) may be used as a surrogate standard, to verify extraction efficiency.

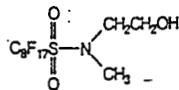
## 2. EXPERIMENTAL COMPOUNDS

Molecular structures of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA are given below.

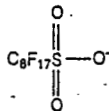
Et-FOSE-OH  
molecular weight: 571



Me-FOSE-OH  
molecular weight: 557



PFOS  
molecular weight: 499 (C<sub>8</sub>F<sub>17</sub>SO<sub>3</sub><sup>-</sup>)



Note: The neutral molecule and standard form which PFOS (anion) is derived from is potassium perfluorooctane sulfonate [C<sub>8</sub>F<sub>17</sub>SO<sub>3</sub>K], molecular weight 538.

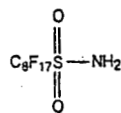
POAA  
molecular weight: 413

C<sub>7</sub>F<sub>15</sub>COO<sup>-</sup>  
Chemical Name = Perfluorooctanoate

Note: The neutral molecule and standard form which POAA (anion) is derived from is ammonium perfluorooctanoate [C<sub>7</sub>F<sub>15</sub>COONH<sub>4</sub>], molecular weight 431.

Centre Protocol No. 00P-023-057

PFOSA  
molecular weight: 499



Chemical Name = Perfluorooctane sulfonylamide

### 3. CHEMICALS AND SUPPLIES

#### 3.1. CHEMICALS

Chemical	Grade	Source	Catalog No.
Acetonitrile (ACN)	HPLC	(VWR) J. T. Baker	JT9017-3
Carbon	120/400	Supelco	57210-U
Methanol (MeOH)	HPLC	(VWR) J. T. Baker	JT9093-2
Silica gel (60-200 mesh)	Reagent	(VWR) J. T. Baker	3405-05
Florisil (60-100 mesh)	Reagent	(VWR) J. T. Baker	3372-08
Superclean LC-NH <sub>2</sub>	Reagent	Supelco	5-7205
1-Octanol	Reagent	Aldrich Chemical	111-87-5
Ammonium Acetate	Reagent	Sigma-Aldrich	A-7330
L-Ascorbic acid	Reagent	Sigma-Aldrich	L-5960
Dimetyldichlorosilane	Reagent	Supelco	3-3009
Toluene	Reagent	(VWR) J. T. Baker	JT9460-3
Water	Type I	in house	-

(Type I water = electrical resistivity, minimum of 16.67 MΩ/cm at 25°C, from a Labconco waterpro workstation)

#### 3.2. STANDARDS

Standard	Grade	Source
Et-FOSE-OH	Analytical	3M
Me-FOSE-OH	Analytical	3M
PFOS	Analytical	3M
POAA	Analytical	3M
PFOSA	Analytical	3M



3.3. EQUIPMENT AND SUPPLIES

EQUIPMENT	SOURCE
Balance, analytical	Mettler
Balance, top-loading	Mettler
Rotary evaporator	Buchi
Rotary evaporator trap	Buchi
Tissumizer	Tekmar
Sorvall RC 5C plus Centrifuge	Dupont
IEC HN SII centrifuge	IEC
Pear-shaped flasks	Pyrex
SPE tubes (20 mL) cat. no. N057177	Supelco
Vacuum pump	Buchi
Visiprep vacuum manifold	Supelco
Wrist-action shaker.	Burrell
Bench-top centrifuge	IEC
50 mL disposable polypropylene centrifuge tubes	VWR
Disposable pipets, test tubes etc.	VWR
125-mL LDPE narrow-mouth bottles	Nalgene
2-mL clear HPLC vial kit (cat # 5181-3400)	HP
Standard lab equipment (class A pipets and volumetric flasks, graduated cylinders, etc.)	
LC/MS/MS and HPLC systems are	As described in Section 4.4.1.

Notes:

1. In order to avoid contamination, the use of disposable labware is highly recommended (containers, tubes, pipets etc.).
2. Teflon or teflon-lined containers or equipment, including teflon-lined HPLC vial caps should not be used.
3. Silanize pear-shaped flasks before use.
4. It is necessary to check the solvents (methanol) for the presence of contaminants (especially POAA) by LC/MS/MS before use. Certain lot numbers have been found to be unsuitable for use.
5. Use disposable micropipets or pipets to aliquot standard solutions, when preparing standards and samples for extraction.
6. Equivalent materials may be substituted for those specified in this method. However, the use of carbon from Supelco is strongly recommended.

### 3.4. SOLUTIONS

1. 50 mM ammonium acetate: Dissolve 3.85 g of ammonium acetate in 1 L of type I water.
2. 2 mM ammonium acetate: Dilute 40 mL of the 50 mM ammonium acetate solution in a liter of type I water, for mobile phase A.
3. 2% ascorbic acid in methanol: Dissolve 2 g ascorbic acid in 100 mL methanol.

**Note:** The volumes shown are provided for guidance; alternative volumes may be prepared.

### 3.5. PREPARATION OF STOCK, FORTIFICATION, AND CALIBRATION SOLUTIONS

Analytical standards are prepared for two purposes. They are used to fortify untreated samples to determine analytical recovery, and to calibrate the response of the detector used in the analysis.

The analyst may vary the absolute volumes of the standards as long as the correct proportions of solute to solvent are maintained. The solutions cited below are given as an example; alternative concentrations may be prepared if needed.

#### 3.5.1. Stock Solutions

Prepare individual stock solutions of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA at 100 µg/mL each, by weighing out 10.0 mg of analytical standard (corrected for percent salt). Adjust final volume to 100 mL with methanol in a 100-mL volumetric flask. Prepare a separate solution for each analyte. Store these stock solutions (in 125-mL LDPE bottles) in a refrigerator at 2°C to 6°C for a maximum period of 6 months from the date of preparation.

#### 3.5.2. Fortification Solutions

- a. 1.0 µg/mL Combined Fortification Solution - Pipet 1.0 mL each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA 100 µg/mL stock solution in to a 100 mL volumetric flask. Bring up to volume with methanol.
- b. 0.1 µg/mL Combined Fortification Solution - Pipet 10.0 mL of the 1.0 µg/mL combined fortification to a 100-mL volumetric flask and bring up to volume with methanol.

- c. 0.01 µg/mL Combined Fortification Solution – Pipet 10.0 mL of the 0.1 µg/mL combined fortification to a 100-mL volumetric flask and bring up to volume with methanol.

Store all fortification standard solutions (in 125-mL LDPE bottles) in a refrigerator at 2°C to 6°C for a maximum period of 6 months from the date of preparation.

### 3.5.3. Calibration Standards

Prepare seven LC/MS/MS mixed calibration standards in methanol via dilution of the 1.0 µg/mL fortification solution.

This is a typical example; additional concentrations may be prepared as needed.

Initial Conc. (µg/mL) <sup>1</sup>	Volume (mL)	Diluted to (mL)	Final Conc. (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	10.0	100	0.005
0.025	10.0	100	0.0025
0.01	10.0	100	0.001
0.005	10.0	100	0.0005

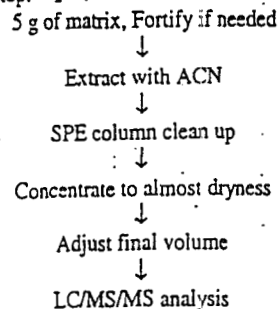
<sup>1</sup> each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA

Store all fortification standard solutions (in 125-mL LDPE bottles) in a refrigerator at 2°C to 6°C for a maximum period of 6 months from the date of preparation.

## 4. METHOD

### 4.1. FLOW DIAGRAM

The flow diagram of the method is given below, followed by a detailed description of each step.



#### 4.2. SAMPLE PROCESSING

For apple, green beans, bread, hot dog, pork, chicken and catfish, place frozen samples in a Hobart food processor and homogenize with dry ice. Then place samples in polyethylene containers and leave open in frozen storage overnight to allow for CO<sub>2</sub> sublimation. Seal and place the samples in frozen storage below -10°C until time of analysis. For milk, shake sample container, transfer to a polyethylene container, seal and place in frozen storage below -10°C until time of analysis. For egg, place samples in a blender and homogenize. Transfer samples to polyethylene containers, seal and place in frozen storage below -10°C until time of analysis.

#### 4.3. EXTRACTION

Note: A. Preparation/conditioning of SPE columns is described in Section 4.3.4.  
B. Silanization of pear-shaped flasks is described in Section 4.3.5.  
C. Evaluation/standardization of SPE columns (if poor recoveries are obtained or when a different supplier is used etc.) is described in Section 4.3.6.

##### 4.3.1. *Apple, Green Beans, Bread, Hot Dog, and Pork*

- a. Weigh 5 g of frozen sample into 50 mL disposable polypropylene centrifuge tube (untreated control samples must be fortified at this point for determination of method recovery).
- b. Add 30 mL of ACN, replace lid tightly, and shake on a wrist-action shaker for 15 minutes. Let the tubes sit for ~ 5 minutes, carefully decant, and transfer the extract on to a conditioned SPE column (see Section 4.3.4.). Collect the eluate in a 125 mL silanized pear-shaped flask (see Section 4.3.5.).

Note: The 20-mL SPE column fits well inside the mouth (sleeve) of the pear-shaped flask. Make sure that the pear-shaped flask is well supported and will not topple. No vacuum is needed for this step.

- c. Add 20 mL of ACN to the sample, replace lid tightly, and shake for 10 minutes on a wrist-action shaker. Let the tubes sit for ~ 5 minutes, carefully decant and transfer the extract to the same SPE column. Collect the eluate into the same pear shaped flask and combine.
- d. Repeat Step c one more time. This time, centrifuge the tubes at ~ 2000 rpm for ~ 5 minutes before decanting the extract. Combine the eluate in the same pear-shaped flask as the original extract.

- e. 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  $<40^{\circ}\text{C}$ ).
- f. Make the final volume, by adding 2 mL of 2% ascorbic acid in methanol to the pear-shaped flask and swirl to mix/dissolve. Transfer the extracts to HPLC vials using disposable pipets.

#### 4.3.2. Chicken and Milk

- a. Weigh 5 g of frozen sample (allow milk to thaw and shake well before weighing) into 50 mL disposable polypropylene centrifuge tube (untreated control samples must be fortified at this point for determination of method recovery).
- b. Add 30 mL of ACN, replace lid tightly and shake on a wrist-action shaker for 15 minutes. Place tubes in freezer ( $< -10^{\circ}\text{C}$ ) for ~2 hours. If a stopping point is needed, samples may be stored overnight in the freezer. Centrifuge the tubes at ~ 10,000 rpm for ~ 10 min. Carefully decant, and transfer the extract onto a conditioned SPE column (see Section 4.3.4.). Collect the eluate in a 125 mL silanized pear-shaped flask (see Section 4.3.5.).  
  
Note: The 20-mL SPE column fits well inside the mouth (sleeve) of the pear-shaped flask. Make sure that the pear-shaped flask is well supported and will not topple. No vacuum is needed for this step.
- c. Add 20 mL of ACN to the sample, replace lid tightly, and shake the sample for 10 minutes on a wrist-action shaker. Place tubes in freezer ( $< -10^{\circ}\text{C}$ ) for ~1 hour. Centrifuge tubes at ~ 10,000 rpm for ~ 10 minutes and carefully decant and transfer the extract to the same SPE column. Collect the eluate into the same pear shaped flask and combine.
- d. Pass 20 mL ACN through the SPE column and combine eluate in the same pear-shaped flask.
- e. 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  $<40^{\circ}\text{C}$ ).
- f. Make the final volume, by adding 2 mL of 2% ascorbic acid in methanol to the pear-shaped flask and swirl to mix/dissolve. Transfer the extracts to HPLC vials using disposable pipets.

#### 4.3.3. Egg and Catfish

- a. Weigh 5 g of frozen sample (allow egg to thaw and shake well before weighing) into 50 mL disposable polypropylene centrifuge tube (untreated control samples must be fortified at this point for determination of method recovery).
- b. Add 30 mL of ACN, replace lid tightly and shake on a wrist-action shaker for 15 minutes. Place tubes in freezer ( $< -10^{\circ}\text{C}$ ) for ~ 1 hour. Centrifuge tubes at ~ 2,000 rpm for ~ 10 minutes. Carefully decant, and transfer the extract on to a conditioned SPE column (see Section 4.3.4.). Collect the eluate in a 125 mL silanized pear-shaped flask (see Section 4.3.5.).

Note: The 20-mL SPE column fits well inside the mouth (sleeve) of the pear-shaped flask. Make sure that the pear-shaped flask is well supported and will not topple. No vacuum is needed for this step.

- c. Add 10 mL of ACN to the sample, homogenize the frozen fat phase using a Tissumizer for ~ 30 seconds and rinse the Tissumizer with 10 mL ACN into the tube. Replace lid tightly, and shake the sample for 10 minutes on a wrist-action shaker. Place tubes in freezer ( $< -10^{\circ}\text{C}$ ) for ~ 1 hour. Centrifuge tubes at ~ 2,000 rpm for ~ 10 minutes and carefully decant, extract to the same SPE column. Collect the eluate into the same pear shaped flask and combine with eluent from initial food extraction.
- d. Pass 20 mL ACN through the SPE column and combine eluate in the same pear-shaped flask.
- e. 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  $< 40^{\circ}\text{C}$ ).
- f. Make the final volume, by adding 2 mL of 2% ascorbic acid in methanol to the pear-shaped flask and swirl to mix/dissolve. Transfer the extracts to HPLC vials using disposable pipets.

Note: The extraction procedures described in Sections 4.3.1, 4.3.2. and 4.3.3. are given as a general guideline for each matrix. The procedures in Sections 4.3.2. and 4.3.3. were developed as a result of poor recoveries (for Et-FOSE-OH and Me-FOSE-OH) and broad peak shapes (for Et-FOSE-OH) when using the procedure described in Section 4.3.1. If problems are encountered with any matrix, it is recommended to try the other two procedures.

Centre Protocol No. 00P-023-057

#### 4.3.4. SPE Column Preparation

- a. Pack 20-mL SPE tubes with (in sequence) 2 g florisil, 2 g silica gel, 2 g carbon, and 1 g LC-NH<sub>2</sub> (Note: the florisil should be activated at ~ 160°C for at least 24 hours before use).
- b. Wash the columns with 20 mL MeOH, then 20 mL ACN. Discard all washes. Do not allow the column to dry.

Note: The SPE columns may be packed and conditioned at any point. Use the vacuum manifold to condition the columns. Do not let the SPE columns run dry at any time.

#### 4.3.5. Silanization of Pear-Shaped Flasks

Pear-shaped flasks are silanized before use, by rinsing with a 30% dimethyl-dichlorosilane in toluene solution. Then rinse with toluene (1x) followed by methanol (3x). The flasks are dried completely before use, either by allowing to air dry or with a stream of nitrogen.

#### 4.3.6. Standardization of SPE Columns

Standardize the SPE columns before analyzing samples in the following manner, especially when using carbon from a different supplier than recommended:-

- a. Using a mixed standard with a concentration ~ 50 ng/mL (each of the five analytes) follow the elution schemes as outlined in Steps b to d in Section 4.3.1. Collect all eluting fractions.
- b. Collect a post-elution fraction, after Step d, eluting with 20 mL of 50/50 MeOH/ACN.
- c. Adjust the final volume of all the fractions to 2 mL with methanol.
- d. Analyze all the fractions by LC/MS/MS.
- e. If the target fraction contains a minimum of 85% of the respective analytes, it may be considered acceptable.
- f. If the "post-elution" fraction contains a significant amount (> 10%) of analytes, the target elution volume may be increased.

### 4.4. ANALYSIS BY LC/MS/MS

#### 4.4.1. LC/MS/MS System and Operating Conditions (Electrospray)

Mass Spec: Micromass Quattro Ultima (Micromass)  
Interface: Electrospray (Micromass)  
Harvard infusion pump  
Computer: COMPAQ Professional Workstation AP200  
Software: Windows NT, MassLynx 3.3

Centre Protocol No. 00P-023-057

HPLC: Hewlett Packard (HP) Series 1100  
 HP Quat Pump  
 HP Vacuum Degasser  
 HP Autosampler  
 HP Column Oven

**Note:** A 4 x 10 mm hypercarb drop-in-guard cartridge (Keystone, part # 844017-400) is attached on-line after the purge valve and before the sample injector port to trap any residue contaminants that may be in the mobile phase and/or HPLC system.

HPLC Column: Genesis C<sub>8</sub> (Jones Chromatography), 2.1 mm x 50 mm, 4µ  
 Column Temp.: 35° C  
 Injection Vol.: 10 µL  
 Mobile Phase (A): 2 mM Ammonium Acetate in Type I water  
 Mobile Phase (B): Methanol

Time	% A	% B	Flow Rate (mL/min)
0	60	40	0.300
0.4	60	40	0.300
1.0	10	90	0.300
7.0	10	90	0.300
7.5	0	100	0.300
9.0	0	100	0.400
9.5	60	40	0.400
13.5	60	40	0.400
14.0	60	40	0.300

It may be necessary to adjust the HPLC gradient in order to optimize instrument performance. Columns with different dimensions (e.g. 2.1 mm x 30 mm) and also columns from different manufacturers (Keystone Betasil C<sub>18</sub> etc.) can be used provided equivalent chromatography is obtained.

Ions monitored:

Analyte	Mode	Transition Monitored	Approximate Retention Time (min)
Et-FOSE-OH	negative	630 → 59	6.7
Me-FOSE-OH	negative	616 → 59	6.7
PFOS	negative	499 → 99	5.3
POAA	negative	413 → 369	5.1
PFOSA	negative	498 → 78	6.0

On a day-to-day basis, the retention times may vary slightly depending on the batch of mobile phase, etc.



**4.4.2. Example Tune File Parameters**

The following values are provided as an example. Actual values may vary from instrument to instrument. Also these values may be changed from time to time in order to optimize for greatest sensitivity.

The mass spectrometer is tuned using a solution of each analyte at ~ 0.5 µg/mL, prepared via dilution of the stock solution in methanol. The solution is infused (using a "T" connector) at 10 µL/min into a 0.2 mL/min stream of mobile phase consisting of 40% methanol and 60% 2 mM ammonium acetate. The analytes are initially tuned for the parent ion and then tuned for the product ion. Once the instrument is tuned, the optimized parameters are saved as a "tune file". This tune file is then used during routine analysis.

<u>Analyte</u>	<u>Dwell (s)</u>	<u>Collision Energy (eV)</u>	<u>Cone (V)</u>
Et-FOSE-OH	0.2	31	30
Me-FOSE-OH	0.2	31	30
PFOS	0.2	43	76
POAA	0.2	11	25
PFOSA	0.2	28	34

<u>Source</u>	<u>Set</u>
Capillary	2.56 kV
Hexapole 1	0.5 V
Aperture 1	0.2 V
Hexapole 2	0.8 V
Source Block Temp.	100°C
Desolvation Temp.	400°C

<u>Analyzer</u>	<u>Set</u>
LM Res 1	13.0 V
HM Res 1	13.0 V
IEnergy 1	0.7 V
Entrance	-2 V
Exit	1 V
LM Res 2	11.0 V
HM Res 2	11.0 V
IEnergy 2	1.0 V
Multiplier	650 V

<u>Gas Flows</u>	<u>Set</u>
Cone Gas	~ 150 L/hr
Desolvation	~ 700 L/hr

<u>Pressures</u>	<u>Read back</u>
Gas Cell	~ 3.0e-3 mbar

Note: An alternative LC/MS/MS system may be used once demonstrated to be equivalent.

#### 4.4.3. Calibration Procedures

- a. Inject the same aliquot (between 10 to 25  $\mu$ L) of each calibration standard into the LC/MS/MS.
- b. Use linear standard curves for quantification. Linear standard curves are generated for each analyte by linear regression using 1/x fit weighting of peak area versus calibration standard concentration using the Windows NT, MassLynx 3.4 (or equivalent) software system. Any calibration standards falling outside  $\pm 30\%$ , based on of its calculated concentration, must be excluded from the calibration curve. However, the total number of calibration standards that could be excluded must not exceed 30% of the total number of standards injected.
- c. The correlation coefficient (R) for calibration curves generated must be  $\geq 0.9925$  ( $R^2 \geq 0.985$ ). If calibration results fall outside these limits, then appropriate steps should be taken to adjust instrument operation, and the relevant set of samples must be reanalyzed.

#### 4.4.4. Sample Analysis

- a. Inject the same aliquot (between 10 to 25  $\mu$ L) of each sample, fortification, control etc. into the LC/MS/MS.
- b. Standards corresponding to at least six concentrations must be included in an analytical set.
- c. Include an entire set of extracted calibration standards at the beginning of a sample set. Continuing calibration verification standards (CCVs) can be interspersed between at least every 8-10 samples. As an alternative, an entire set of standards interspersed every 3-4 samples can be included to account for a second set of standards. In either case, a standard or CCV should be the last injection in a sample set (see Section 4.5).
- d. Each set of samples analyzed for investigation purposes should include at least one reagent control (method blank) at least one matrix control and two matrix control samples fortified at known concentrations and carried through the procedure to verify recovery.

Note: The analysis performed during method development included fortifications at 0.5, 1.0, 2.0, 5.0 and 10 ng/g (ppb) for each analyte.

Centre Protocol No. 00P-023-057

- e. The concentration of each sample, fortification, control etc. is determined from the standard curve, based on the peak area of each analyte. The standard responses should bracket responses of the residue found in each sample set. If necessary, dilute the samples and re-analyze to give a response within the standard curve range.
- f. Fortifications that bracket the highest residue expected in each treated matrix will be included with each sample set. If residues are found outside these limits, additional fortifications will be included in a subsequent sample set to establish that method recoveries are available for all analytes of interest at concentrations exceeding those in treated samples.
- g. Fortification recoveries falling within 50 to 140% are considered acceptable for fortifications at the LOQ level and 60 to 130% for fortifications at levels greater than the LOQ.
- h. Samples or controls in which no peaks are detected (i.e. signal : noise ratio < 3:1) at the corresponding analyte retention times will be reported as ND (not detected). Samples or controls in which peaks are detected at the corresponding analyte retention times that have a quantification concentration of zero or a negative number will be reported as NQ (not quantifiable). Otherwise, all positive numbers will be reported.
- i. If samples are not loaded on the instrument to be analyzed the day they are extracted, samples will be stored refrigerated at approximately 4°C until analysis, and analyzed preferably within a week.

Recoveries from method development for all matrices can be found in Tables 1-9. Lower recoveries for Me-FOSE-OH and broad peak shapes for Et-FOSE-OH have been observed in some animal matrices (chicken, pork and chicken egg).

#### 4.5. PERFORMANCE CRITERIA

The following two criteria should be met before the initial analysis of samples, especially when using different instrumentation set-ups than those cited in this method.

First Criterion - Inject a standard solution on LC/MS/MS corresponding to the estimated LOQ (1.25 ng/mL) and obtain a signal to noise ratio of at least 9:1 relative to the reagent blank. If this criterion cannot be met, optimize and change instrument operating parameters.

Second Criterion - Inject a set of standards of six or more concentration levels ranging from at or below the LOQ, up to the highest concentration level to be included in the analysis. Generate a calibration curve for each analyte and obtain a 1/x weighted linear regression with a coefficient of determination of at least 0.985 for each analyte.

#### 4.6. TIME REQUIRED FOR ANALYSIS

A set of 14 samples can be taken through the extraction procedure in approximately six hours by one person. The LC/MS/MS analysis (8-10 standards and 14 samples) will take approximately 6 hours.

### 5. CALCULATIONS

Calculate the amount of analyte found (in ng/mL, based on peak area) using the standard curve generated by the Mass Lynx software program using Equation 1.

Equation 1:

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

#### 5.1. COMPONENT RESIDUE CONCENTRATION

Determine the component residue concentration using Equation 2.

Equation 2:

$$\text{Residue found (ng/g)} = \frac{(\text{analyte found (ng/mL)} \times \text{DF} \times \text{FV (mL)})}{\text{sample weight (g)}}$$

Where DF = dilution factor and FV = final volume

Note: Residues found in the samples should be corrected to reflect the average amount of residue (if any) found in the matrix blank. If the amount of residue found in the reagent blanks is similar to the amount found in the matrix blanks then the average of the reagent and matrix blanks will be taken to achieve a more accurate number. If however, the matrix blanks have residues higher than the reagent blanks then correct only for the matrix blanks. Also, when taking averages, consider anything reported as not quantifiable (NQ) as equal to zero.

## 5.2. FORTIFICATION RECOVERY

For samples fortified with known amounts of analytes prior to extraction, calculate the percent recovery from Equation 3.

Equation 3:

$$\text{Recovery (\%)} = \frac{(\text{ng/g found} - (\text{average ng/g found in control}))}{\text{ng/g added}} \times 100$$

or

$$\frac{(\text{analyte found (ng/mL)} - (\text{average analyte found in control (ng/mL)/DF}) \times \text{DF} \times \text{FV})}{\text{sample weight (g)} \times \text{amount analyte added (ng/g)}} \times 100$$

## 6. SAFETY

There are no unusual hazards associated with this method. The analyst should read the material safety data sheets for all reagents before performing this method. Normal laboratory precautions should be taken.

Centre Protocol No. 00P-023-057

TABLES

Centre Analytical Laboratories, Inc. Study # 023-001

Page 23 of 69

Centre Analytical Laboratories, Inc.

Page 35 of 81

Table 1: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Whole Apple

Description	Et-FOSE-OH				Me-FOSE-OH				PFOS				POAA				PFOSA			
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2	
	11/11/99	11/17/99	11/11/99	11/17/99	11/11/99	11/17/99	11/11/99	11/17/99	11/11/99	11/17/99	11/11/99	11/17/99	11/11/99	11/17/99	11/11/99	11/17/99	11/11/99	11/17/99	11/11/99	11/17/99
	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec
Reagent Blank A	NQ	-	ND	-	NQ	-	NQ	-	0.301	-	0.345	-	NQ	-	NQ	-	ND	-	ND	-
Reagent Blank B	NQ	-	ND	-	NQ	-	NQ	-	0.615	-	0.362	-	NQ	-	ND	-	ND	-	NQ	-
Matrix Blank A	NQ	-	ND	-	NQ	-	NQ	-	0.295	-	0.297	-	NQ	-	NQ	-	ND	-	ND	-
Matrix Blank B	NQ	-	ND	-	NQ	-	NQ	-	0.302	-	0.335	-	NQ	-	NQ	-	ND	-	ND	-
0.5 ppb Spk A	0.419	84	0.437	87	0.433	87	0.512	102	0.714	83	0.780	93	0.466	93	0.485	99	0.379	76	0.432	86
0.5 ppb Spk B	0.413	83	0.427	85	0.435	87	0.453	91	0.622	85	0.784	94	0.465	93	0.465	93	0.371	74	0.411	82
1.0 ppb Spk C	0.860	88	0.861	88	0.888	89	0.951	95	1.005	71	1.308	99	0.912	91	0.928	93	0.788	79	0.892	89
1.0 ppb Spk D	0.798	80	0.768	77	0.821	82	0.851	85	0.939	64	1.078	78	0.804	80	0.824	82	0.724	72	0.840	84
2.0 ppb Spk E	1.878	94	1.872	84	1.905	95	1.884	84	2.312	101	2.130	91	1.783	89	1.642	82	1.682	84	1.787	89
2.0 ppb Spk F	1.779	89	1.716	88	1.889	94	1.807	90	2.090	90	2.118	90	1.793	90	1.685	84	1.711	88	1.781	89
5.0 ppb Spk G	4.481	90	3.794	76	4.451	89	3.983	79	4.627	87	4.184	77	4.427	89	3.811	72	4.370	87	4.514	90
5.0 ppb Spk H	4.629	93	4.391	88	4.878	98	4.238	85	4.776	90	4.432	82	4.598	92	4.259	85	4.410	88	4.767	95
10.0 ppb Spk I	9.036	90	7.714	77	8.818	88	7.388	74	9.000	87	7.716	74	9.178	92	7.672	77	8.900	90	9.100	91
10.0 ppb Spk J	9.438	84	7.290	73	9.884	99	7.322	73	10.216	99	8.058	77	9.924	99	7.022	70	8.900	99	9.134	91
AVERAGE:		88		82		91		86		84		85		91		84		84		89
STD DEVIATION:		5		6		6		9		13		9		5		9		8		4

Table 2: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Green Beans

Description	Et-FOSE-OH				Me-FOSE-OH				PFOS				POAA				PFOSA			
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2	
	11/12/99		11/18/99		11/12/99		11/18/99		11/12/99		11/18/99		11/12/99		11/18/99		11/12/99		11/18/99	
	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec
Reagent Blank A	ND	-	ND	-	NQ	-	ND	-	0.290	-	0.190	-	ND	-	ND	-	ND	-	ND	-
Reagent Blank B	ND	-	ND	-	ND	-	ND	-	0.244	-	0.162	-	ND	-	ND	-	ND	-	ND	-
Matrix Blank A	ND	-	ND	-	NQ	-	NQ	-	0.433	-	0.204	-	NQ	-	NQ	-	NQ	-	ND	-
Matrix Blank B	NQ	-	NQ	-	NQ	-	NQ	-	0.308	-	0.139	-	NQ	-	NQ	-	ND	-	ND	-
0.5 ppb Spk A	0.397	79	0.450	90	0.454	91	0.456	91	0.668	59	0.659	97	0.517	103	0.435	87	0.412	82	0.427	85
0.5 ppb Spk B	0.363	73	0.388	77	0.395	79	0.378	76	0.578	41	0.324	31	0.430	86	0.415	83	0.351	70	0.360	72
1.0 ppb Spk C	0.887	87	0.930	93	0.881	88	0.931	93	1.149	78	0.921	75	0.920	92	0.994	99	0.828	83	0.944	94
1.0 ppb Spk D	1.027	103	0.952	95	0.977	98	1.005	101	1.288	92	0.967	82	0.974	97	1.019	102	0.930	93	0.934	93
2.0 ppb Spk E	1.865	93	1.878	94	1.735	87	1.808	90	2.245	94	1.797	81	1.634	82	1.860	93	1.878	84	1.818	91
2.0 ppb Spk F	1.728	88	1.813	91	1.669	83	1.749	87	2.044	84	1.764	80	1.618	81	1.843	92	1.586	79	1.783	89
5.0 ppb Spk G	4.770	95	4.652	93	4.582	91	4.583	92	4.746	88	4.365	84	4.588	92	4.334	87	4.305	86	4.574	91
5.0 ppb Spk H	4.638	91	4.648	93	4.422	88	4.570	91	4.487	82	4.599	89	4.477	90	4.572	91	4.320	86	4.798	96
10.0 ppb Spk I	9.854	99	8.460	85	10.000	100	9.264	93	9.802	92	8.986	88	9.224	92	8.180	82	8.074	91	8.908	90
10.0 ppb Spk J	10.370	104	9.022	90	9.794	88	8.828	86	10.080	97	8.598	84	9.540	95	8.648	86	8.790	90	9.358	94
AVERAGE:		91		90		90		90		81		79		91		90		85		90
STD DEVIATION:		10		5		7		6		18		18		7		7		8		7



Table 3: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Chicken Eggs

Description	Et-FOSE-OH				Me-FOSE-OH				PFOS				POAA				PFOSA					
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2			
	12/29/99	1/4/00	12/29/99	1/4/00	12/29/99	1/4/00	12/29/99	1/4/00	12/29/99	1/4/00	12/29/99	1/4/00	12/29/99	1/4/00	12/29/99	1/4/00	12/29/99	1/4/00	12/29/99	1/4/00		
Reagent Blank A	ND	-	ND	-	ND	-	ND	-	0.208	-	ND	-	NQ	-	ND	-	ND	-	ND	-	ND	-
Reagent Blank B	ND	-	ND	-	ND	-	NQ	-	0.289	-	ND	-	NQ	-	ND	-	ND	-	ND	-	ND	-
Matrix Blank A	ND	-	NQ	-	ND	-	NQ	-	0.762	-	0.550	-	NQ	-	NQ	-	NQ	-	NQ	-	NQ	-
Matrix Blank B	ND	-	NQ	-	ND	-	NQ	-	0.654	-	0.530	-	NQ	-	NQ	-	NQ	-	NQ	-	NQ	-
0.5 ppb Spk A	0.308	62	0.389	78	0.314	63	0.289	58	1.430	145	1.182	128	0.603	121	0.465	93	0.392	78	0.435	87		
0.5 ppb Spk B	0.458	91	0.341	68	0.492	98	0.352	70	1.293	117	0.977	87	0.548	110	0.489	98	0.399	80	0.456	91		
1.0 ppb Spk C	0.751	75	1.071	107	0.815	81	0.820	92	1.894	129	1.872	133	1.208	121	1.111	111	0.899	90	1.082	109		
1.0 ppb Spk D	1.032	103	1.135	114	0.931	93	0.848	85	1.673	97	1.757	122	1.222	122	1.153	115	0.942	94	1.120	112		
2.0 ppb Spk E	1.121	56	2.088	104	1.180	59	2.066	103	2.047	112	2.675	107	2.112	108	2.080	103	1.583	79	2.138	107		
2.0 ppb Spk F	1.415	71	2.040	102	1.208	60	1.478	74	2.808	109	2.733	110	1.973	99	1.981	99	1.728	86	2.145	107		
5.0 ppb Spk G	3.575	71	5.574	111	3.542	71	4.990	100	6.212	110	5.812	105	5.482	110	4.523	90	4.884	97	5.203	104		
5.0 ppb Spk H	4.333	57	5.440	109	4.409	88	5.548	111	6.349	113	5.898	103	5.718	114	4.807	98	5.039	101	5.418	108		
10.0 ppb Spk I	5.917	59	10.588	108	5.950	59	8.300	83	10.008	93	10.009	95	9.888	99	9.129	91	8.587	88	10.408	104		
10.0 ppb Spk J	9.252	83	10.838	108	8.708	87	7.789	78	10.815	101	10.171	98	10.207	102	10.148	101	9.228	92	11.314	113		
AVERAGE:		77		101		76		85		113		109		110		100		88		104		
STD DEVIATION:		16		15		15		16		15		15		9		8		8		9		

Table 4: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Chicken Muscle

Description	Et-FOSE-OH				Me-FOSE-OH				PFOS				POAA				PFOSA					
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2			
	11/20/99	11/22/99	11/20/99	11/22/99	11/20/99	11/22/99	11/20/99	11/22/99	11/20/99	11/22/99	11/20/99	11/22/99	11/20/99	11/22/99	11/20/99	11/22/99	11/20/99	11/22/99	11/20/99	11/22/99		
ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	
Reagent Blank A	ND	-	ND	-	ND	-	NQ	-	NQ	-	0.109	-	ND	-	ND	-	ND	-	ND	-	ND	-
Reagent Blank B	ND	-	ND	-	ND	-	NQ	-	NQ	-	0.103	-	ND	-	ND	-	ND	-	ND	-	ND	-
Matrix Blank A	ND	-	NQ	-	NQ	-	NQ	-	0.055	-	0.093	-	NQ	-	NQ	-	NQ	-	NQ	-	NQ	-
Matrix Blank B	ND	-	NQ	-	NQ	-	NQ	-	0.048	-	0.143	-	NQ	-	NQ	-	NQ	-	NQ	-	NQ	-
0.5 ppb Spk A	0.421	84	1.041	208	0.302	60	0.209	42	0.480	88	0.630	103	0.468	94	0.453	91	0.417	83	0.418	83		
0.5 ppb Spk B	0.473	95	0.391	78	0.348	70	0.287	53	0.508	92	0.602	97	0.470	94	0.430	86	0.418	84	0.426	85		
1.0 ppb Spk C	0.911	91	0.878	88	0.649	65	0.596	60	1.058	101	1.162	104	0.981	98	0.993	99	0.903	90	0.953	95		
1.0 ppb Spk D	0.992	99	1.026	103	0.894	89	0.562	56	1.055	100	1.074	96	0.969	97	0.997	97	0.987	99	0.958	98		
2.0 ppb Spk E	2.038	102	1.342	87	1.683	79	1.152	58	2.057	100	2.014	95	1.921	96	1.869	93	1.986	99	1.894	95		
2.0 ppb Spk F	1.885	84	1.498	75	1.480	74	1.044	52	1.792	87	2.147	101	1.849	82	1.779	89	1.783	90	1.809	95		
5.0 ppb Spk G	4.764	95	4.710	94	3.484	70	4.249	85	4.884	97	4.730	92	4.751	95	4.479	90	4.908	100	4.815	98		
5.0 ppb Spk H	4.757	95	4.842	93	3.875	74	3.835	77	4.746	94	4.757	93	4.528	91	4.648	93	4.944	99	4.842	99		
10.0 ppb Spk I	10.218	102	9.736	97	9.650	97	8.034	80	10.834	108	9.392	93	10.186	102	9.326	93	10.950	109	10.386	104		
10.0 ppb Spk J	10.058	101	11.146	111	9.524	95	9.862	100	10.016	100	11.614	115	9.920	99	10.868	109	11.128	111	11.978	120		
AVERAGE:		95		101		77		66		97		99		95		94		96		97		
STD DEVIATION:		7		40		13		18		7		7		5		6		10		10		

Table 5: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Cow Milk.

Description	Et-FOSE-OH				Me-FOSE-OH				PFOS				POAA				PFOSA			
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2	
	11/9/99	11/18/99	11/9/99	11/18/99	11/9/99	11/18/99	11/9/99	11/18/99	11/9/99	11/18/99	11/9/99	11/18/99	11/9/99	11/18/99	11/9/99	11/18/99	11/9/99	11/18/99	11/9/99	11/18/99
Reagent Blank A	ND	-	ND	-	NQ	-	NQ	-	NQ	-	0.453	-	NQ	-	ND	-	NQ	-	ND	-
Reagent Blank B	ND	-	ND	-	NQ	-	ND	-	0.097	-	0.269	-	NQ	-	ND	-	NQ	-	ND	-
Matrix Blank A	NQ	-	ND	-	NQ	-	NQ	-	0.170	-	0.335	-	NQ	-	NQ	-	NQ	-	ND	-
Matrix Blank B	ND	-	ND	-	NQ	-	ND	-	0.174	-	0.332	-	NQ	-	NQ	-	ND	-	ND	-
0.5 ppb Spk A	0.418	84	0.478	98	0.438	87	0.449	90	0.729	111	0.552	44	0.504	101	0.534	107	0.399	80	0.423	85
0.5 ppb Spk B	0.394	79	0.459	92	0.440	88	0.489	98	0.670	100	0.530	39	0.532	108	0.551	110	0.342	68	0.458	92
1.0 ppb Spk C	0.825	83	1.082	108	0.848	85	1.030	103	1.027	88	1.217	88	0.951	95	1.223	122	0.747	75	1.019	102
1.0 ppb Spk D	0.901	90	1.038	104	0.845	84	0.998	100	1.001	92	1.018	68	0.987	97	1.082	108	0.841	84	0.924	92
2.0 ppb Spk E	1.498	76	1.807	90	1.508	75	1.788	89	1.868	75	1.844	68	1.578	78	1.854	93	1.408	70	1.688	84
2.0 ppb Spk F	1.831	82	2.058	103	1.664	83	1.971	99	1.939	88	1.938	80	1.767	88	2.019	101	1.834	82	1.847	92
5.0 ppb Spk G	4.519	90	5.430	109	4.314	86	5.390	108	4.610	89	4.800	89	4.928	99	5.588	112	4.450	89	4.988	99
5.0 ppb Spk H	4.462	89	5.343	107	4.417	88	5.314	108	4.605	80	4.819	90	4.817	98	5.488	110	4.400	88	4.958	99
10.0 ppb Spk I	8.016	80	9.384	94	7.832	78	9.188	92	8.350	82	9.388	91	8.762	88	9.704	97	8.208	82	10.044	100
10.0 ppb Spk J	8.038	80	10.298	103	7.488	75	10.192	102	8.038	79	9.488	91	8.252	83	10.680	107	8.152	82	10.214	102
AVERAGE:	83		100		83		99		89		75		93		107		80		95	
STD DEVIATION:	5		7		5		7		10		20		8		8		7		7	

Table 6: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Bread

Description	Et-FOSE-OH				Me-FOSE-OH				PFOS				POAA				PFOSA					
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2			
	11/15/99	11/19/99	11/15/99	11/19/99	11/15/99	11/19/99	11/15/99	11/19/99	11/15/99	11/19/99	11/15/99	11/19/99	11/15/99	11/19/99	11/15/99	11/19/99	11/15/99	11/19/99	11/15/99	11/19/99		
Reagent Blank A	ND	-	NQ	-	NQ	-	NQ	-	0.248	-	NQ	-	NQ	-	ND	-	ND	-	ND	-	ND	-
Reagent Blank B	ND	-	NQ	-	NQ	-	NQ	-	0.523	-	NQ	-	NQ	-	ND	-	ND	-	ND	-	ND	-
Matrix Blank A	ND	-	NQ	-	NQ	-	NQ	-	0.363	-	0.047	-	NQ	-	NQ	-	ND	-	ND	-	ND	-
Matrix Blank B	ND	-	NQ	-	NQ	-	NQ	-	0.049	-	0.051	-	NQ	-	NQ	-	ND	-	ND	-	ND	-
0.5 ppb Spk A	0.356	71	0.432	88	0.358	72	0.418	84	0.603	97	0.448	80	0.385	73	0.421	84	0.370	74	0.412	82		
0.5 ppb Spk B	0.385	77	0.484	93	0.340	70	0.423	85	0.568	72	0.574	105	0.382	78	0.398	79	0.390	78	0.409	82		
1.0 ppb Spk C	0.858	88	1.037	104	0.832	83	0.938	94	1.284	108	1.074	103	0.881	88	0.984	98	0.878	88	0.978	98		
1.0 ppb Spk D	0.947	95	1.087	109	0.858	88	1.071	107	1.218	101	1.048	100	0.842	84	1.115	111	0.929	93	1.088	107		
2.0 ppb Spk E	1.728	88	2.210	111	1.898	85	2.028	101	2.178	99	2.090	102	1.443	72	1.941	97	1.745	87	1.852	98		
2.0 ppb Spk F	1.700	85	2.229	111	1.872	84	2.091	105	2.097	95	2.013	98	1.458	73	2.141	107	1.705	85	1.848	97		
5.0 ppb Spk G	5.188	103	8.088	102	4.958	99	4.797	98	4.584	92	4.470	88	4.230	85	4.801	92	4.824	92	4.758	95		
5.0 ppb Spk H	4.888	98	8.182	103	4.748	95	4.814	98	4.799	92	4.274	85	4.375	88	4.394	88	4.898	98	4.707	94		
10.0 ppb Spk I	11.020	110	9.710	97	10.054	101	10.038	100	9.820	98	9.088	90	10.010	100	10.002	100	10.184	102	9.842	98		
10.0 ppb Spk J	9.798	97	11.348	113	9.058	91	11.102	111	8.700	88	9.484	94	8.978	90	10.492	105	9.480	95	10.800	108		
AVERAGE:	91		103		87		98		94		95		83		96		89		96			
STD DEVIATION:	12		9		10		9		9		8		9		10		9		8			

Table 7: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Hot Dog

Description	Et-FOSE-OH				Me-FOSE-OH				PFOS				POAA				PFOSA			
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2	
	11/8/99	11/11/99	11/8/99	11/11/99	11/8/99	11/11/99	11/8/99	11/11/99	11/8/99	11/11/99	11/8/99	11/11/99	11/8/99	11/11/99	11/8/99	11/11/99	11/8/99	11/11/99	11/8/99	11/11/99
Reagent Blank A	ND	-	NQ	-	ND	-	NQ	-	0.178	-	0.208	-	NQ	-	NQ	-	ND	-	ND	-
Reagent Blank B	ND	-	NQ	-	NQ	-	NQ	-	NQ	-	0.123	-	NQ	-	NQ	-	ND	-	ND	-
Matrix Blank A	ND	-	NQ	-	0.307	-	NQ	-	0.070	-	0.065	-	NQ	-	NQ	-	NQ	-	NQ	-
Matrix Blank B	ND	-	NQ	-	ND	-	NQ	-	0.173	-	NQ	-	NQ	-	NQ	-	ND	-	NQ	-
0.5 ppb Spk A	0.438	87	0.488	97	0.448	77	0.441	89	0.557	87	0.617	110	0.482	96	0.529	106	0.412	82	0.394	79
0.5 ppb Spk B	0.414	83	0.385	73	0.453	78	0.353	71	0.540	84	0.594	106	0.503	101	0.424	85	0.301	78	0.208	59
1.0 ppb Spk C	0.948	95	1.004	100	1.001	94	0.879	88	1.041	92	1.238	117	1.033	103	0.968	97	0.881	88	0.830	84
1.0 ppb Spk D	0.954	95	1.027	103	0.983	92	0.989	97	1.137	102	1.132	107	1.058	108	1.007	101	0.857	86	0.817	82
2.0 ppb Spk E	1.534	77	1.990	99	1.195	57	1.920	98	1.987	93	2.120	103	1.971	99	1.816	91	1.594	80	1.639	82
2.0 ppb Spk F	1.878	84	1.989	99	1.253	60	2.001	100	2.098	99	2.167	105	2.120	106	1.780	89	1.703	85	1.693	80
5.0 ppb Spk G	5.401	108	4.959	99	5.805	111	4.915	98	5.248	103	4.743	94	5.802	112	4.590	92	4.892	98	4.420	88
5.0 ppb Spk H	5.421	108	5.340	107	5.399	107	5.273	105	5.117	100	4.784	94	5.505	110	4.988	100	4.791	98	4.888	94
10.0 ppb Spk I	11.040	110	10.590	108	11.482	114	10.120	101	10.090	100	9.282	92	11.830	116	10.426	104	9.858	97	9.384	94
10.0 ppb Spk J	10.454	105	11.240	112	10.898	108	10.234	102	9.812	97	10.674	108	10.570	108	10.002	109	9.380	94	9.788	98
AVERAGE:		95		100		90		95		96		103		106		97		88		84
STD DEVIATION:		12		10		21		10		6		8		6		8		7		11

Table 8: Summary of Recoveries for PFOS, POAA and PFOSA in Catfish

Description	PFOS				POAA				PFOSA			
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2	
	1/21/00	1/24/00	1/21/00	1/24/00	1/21/00	1/24/00	1/21/00	1/24/00	1/21/00	1/24/00	1/21/00	1/24/00
	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec	ng/g	% Rec
Reagent Blank A	1.148	-	0.718	-	ND	-	ND	-	ND	-	ND	-
Reagent Blank B	1.836	-	0.737	-	ND	-	ND	-	ND	-	ND	-
Matrix Blank A	1.236	-	1.320	-	NQ	-	NQ	-	NQ	-	NQ	-
Matrix Blank B	1.238	-	1.168	-	NQ	-	NQ	-	NQ	-	NQ	-
0.5 ppb Spk A	1.707	94	1.650	81	0.483	97	0.598	120	0.550	110	0.481	96
0.5 ppb Spk B	2.078	168	1.844	120	0.510	102	0.642	128	0.653	131	0.508	101
1.0 ppb Spk C	2.438	120	2.280	102	1.008	107	1.318	132	1.178	118	1.083	108
1.0 ppb Spk D	2.764	153	2.224	98	1.120	112	1.308	140	1.218	122	1.118	112
2.0 ppb Spk E	3.531	115	3.609	113	2.183	108	2.864	143	2.089	103	2.141	107
2.0 ppb Spk F	3.339	105	3.710	123	2.281	113	2.951	148	2.109	110	1.892	85
5.0 ppb Spk G	6.012	88	6.210	89	5.272	105	6.674	113	5.908	118	4.942	99
5.0 ppb Spk H	6.170	99	6.988	115	5.100	102	6.383	128	5.824	116	5.701	114
10.0 ppb Spk I	11.088	99	12.074	108	11.760	118	12.101	121	11.140	111	11.433	114
10.0 ppb Spk J	11.391	102	12.083	108	11.088	111	12.834	128	12.762	128	11.208	112
AVERAGE:		115		107		108		130		117		106
STD DEVIATION:		26		12		6		11		9		7

Table 9: Summary of Recoveries for Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA in Pork Muscle

Description	Et-FOSE-OH				Me-FOSE-OH				PFOS				POAA				PFOSA			
	Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2		Trial 1		Trial 2	
	12/20/99	12/21/99	12/20/99	12/21/99	12/20/99	12/21/99	12/20/99	12/21/99	12/20/99	12/21/99	12/20/99	12/21/99	12/20/99	12/21/99	12/20/99	12/21/99	12/20/99	12/21/99	12/20/99	12/21/99
Reagent Blank A	NQ	-	ND	-	ND	-	ND	-	ND	-	NQ	-	ND	-	ND	-	ND	-	ND	-
Reagent Blank B	NQ	-	ND	-	NQ	-	ND	-	ND	-	NQ	-	ND	-	ND	-	ND	-	ND	-
Matrix Blank A	ND	-	NQ	-	ND	-	NQ	-	ND	-	NQ	-	ND	-	ND	-	ND	-	ND	-
Matrix Blank B	NQ	-	NQ	-	NQ	-	NQ	-	ND	-	NQ	-	NQ	-	NQ	-	NQ	-	NQ	-
0.5 ppb Spk A	0.622	124	0.492	98	0.357	71	0.419	84	0.498	99	0.534	107	0.612	122	0.582	112	0.488	97	0.478	98
0.5 ppb Spk B	0.483	97	0.530	108	0.306	61	0.455	91	0.500	101	0.552	110	0.570	114	0.598	120	0.474	95	0.504	101
1.0 ppb Spk C	0.989	99	1.044	104	0.714	71	0.975	97	0.948	95	1.227	123	1.102	110	1.163	116	1.019	102	1.076	108
1.0 ppb Spk D	1.430	143	1.120	112	0.772	77	1.033	103	1.128	113	1.189	117	1.207	121	1.201	120	1.183	118	1.094	109
2.0 ppb Spk E	1.802	90	2.242	112	1.122	68	1.908	95	2.028	101	2.171	109	2.144	107	2.185	108	2.155	108	2.143	107
2.0 ppb Spk F	1.852	93	2.142	107	1.139	57	1.857	93	1.900	100	2.234	112	2.137	107	2.201	110	2.058	103	2.148	107
5.0 ppb Spk G	4.855	97	5.352	107	3.980	79	5.028	101	5.195	104	4.982	100	5.341	107	5.092	102	5.304	106	5.236	105
5.0 ppb Spk H	4.395	88	5.422	108	3.832	77	5.455	109	5.484	109	5.183	104	5.435	108	5.388	108	5.458	109	5.360	107
10.0 ppb Spk I	9.403	94	11.870	118	7.802	78	11.132	111	10.022	100	10.903	109	9.987	100	11.421	114	9.889	100	10.985	110
10.0 ppb Spk J	10.556	109	10.908	109	10.166	102	10.568	106	9.850	98	10.311	103	10.259	103	10.691	107	9.940	99	10.838	106
AVERAGE:		103		108		73		99		102		109		110		112		104		108
STD DEVIATION:		17		5		13		9		5		7		7		6		7		4

Centre Protocol No. 00P-023-057

## FIGURES

Centre Analytical Laboratories, Inc. Study # 023-001

Page 33 of 69

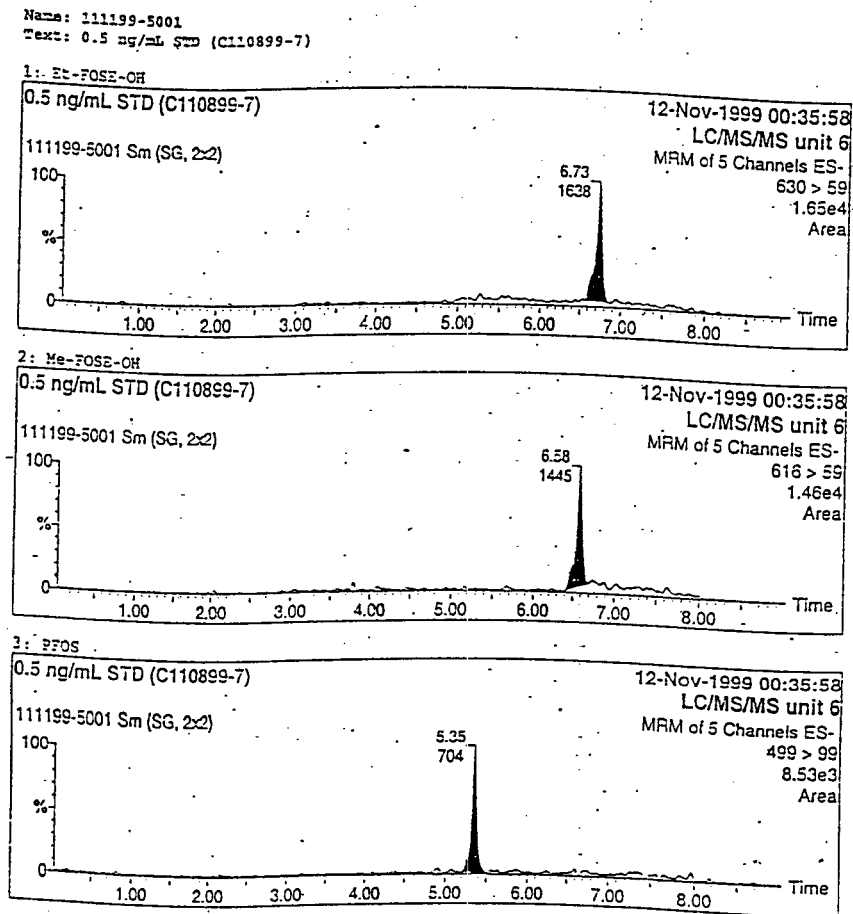
Centre Analytical Laboratories, Inc.

Page 45 of 81



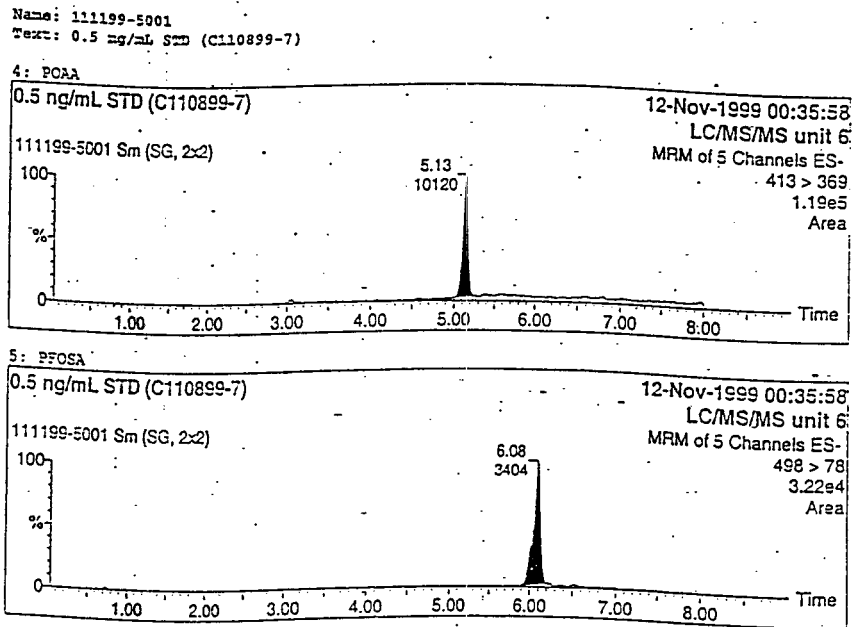
Centre Protocol No. 00P-023-057

Figure 1: Representative Chromatograms of a 0.5 ng/mL Mixed Calibration Standard of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA



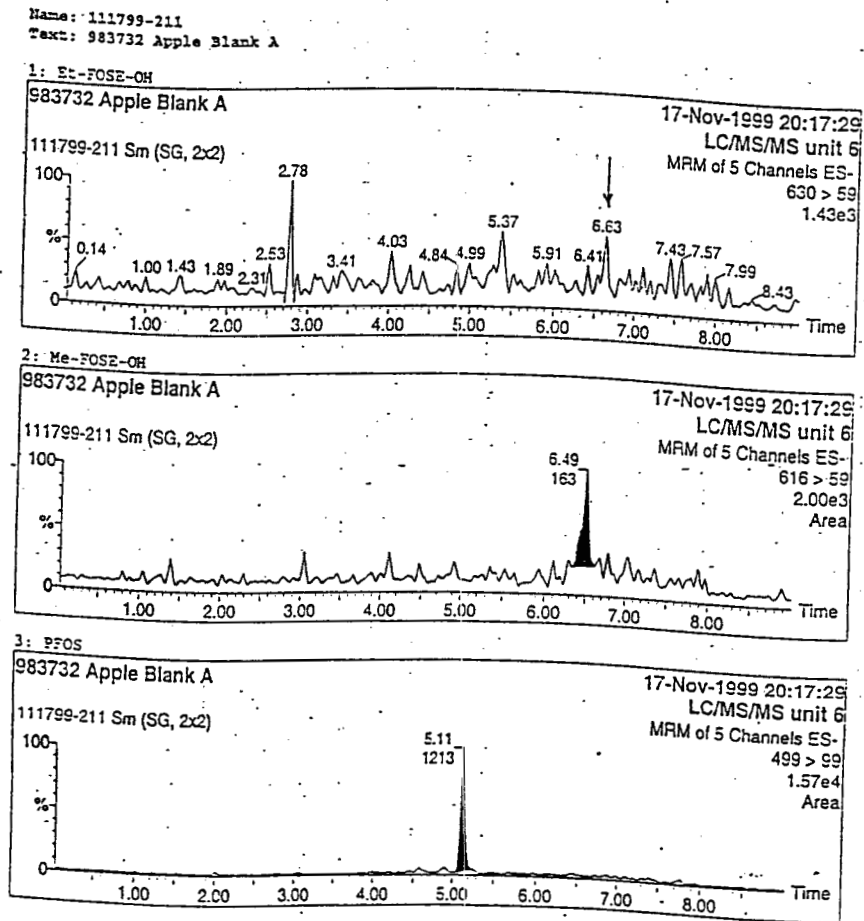
Centre Protocol No. 00P-023-057

Figure 1 (continued): Representative Chromatograms of a 0.5 ng/mL Mixed Calibration Standard of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA and PFOSA



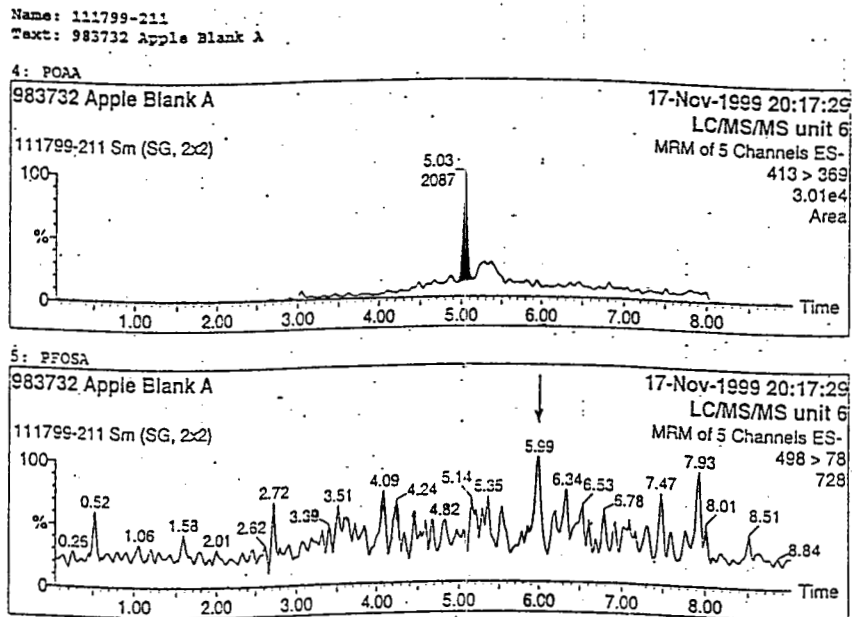
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Figure 2: Representative Chromatograms of a Control Whole Apple Sample (Set 111799 Apple, Centre ID 983732 Apple Blank A)



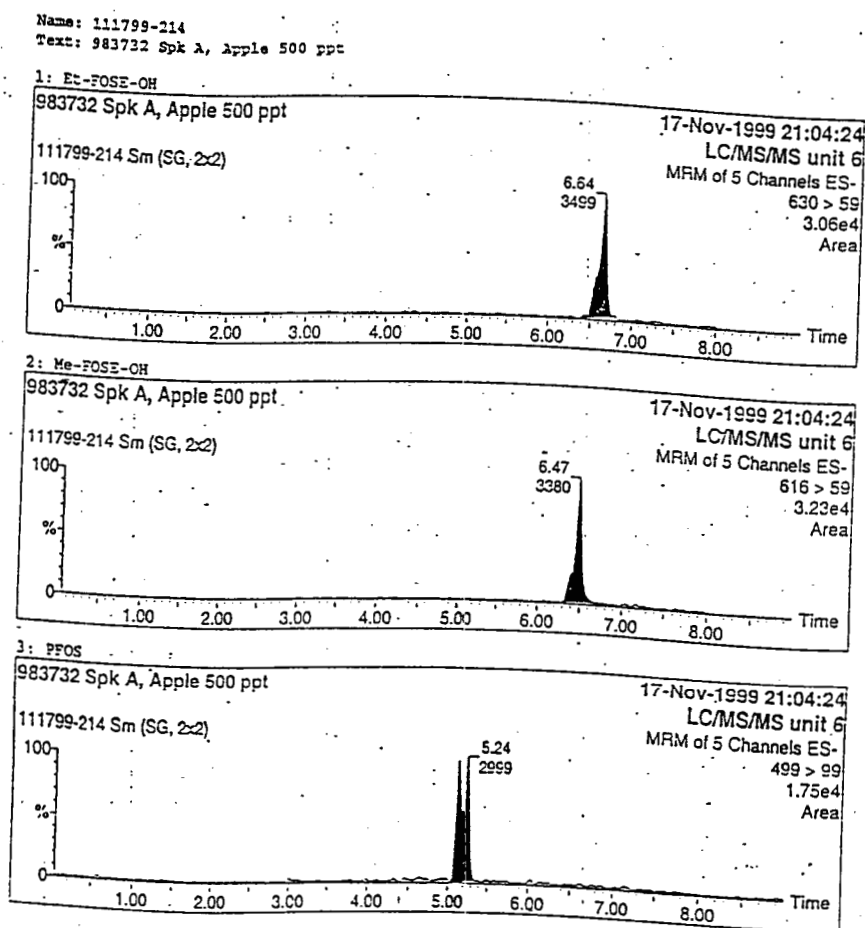
Centre Protocol No. 00P-023-057

Figure 2 (continued): Representative Chromatograms of a Control Whole Apple Sample (Set 111799 Apple, Centre ID 983732 Apple Blank A)



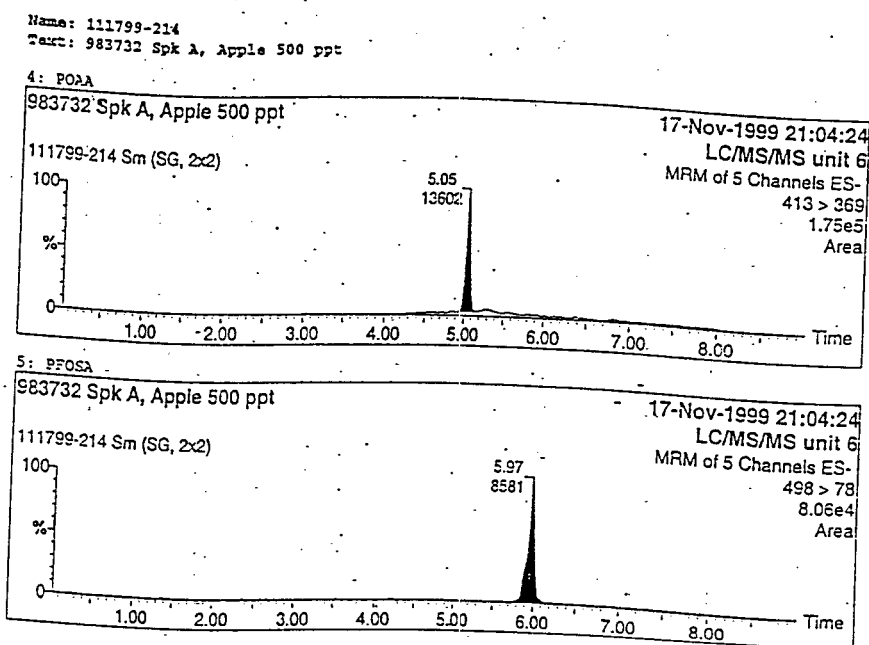
Centre Protocol No. OOP-023-057

Figure 3: Representative Chromatograms from the Injection of Control Whole Apple Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111799 Apple, Centre ID 983732 Spk A)



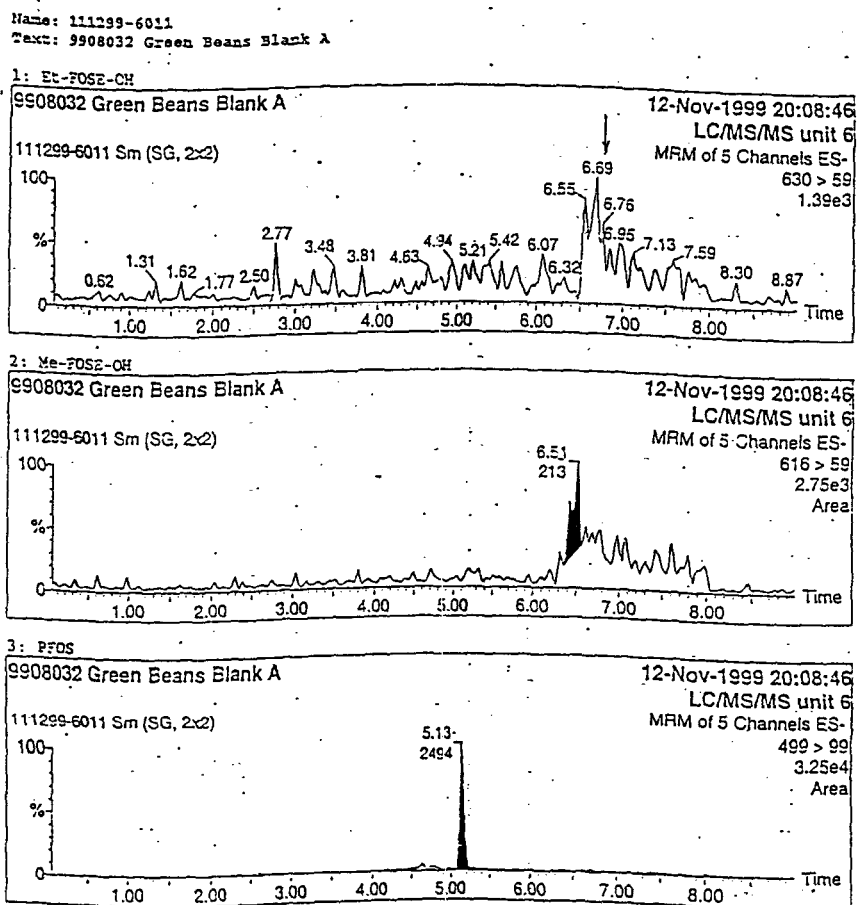
Centre Protocol No. 00P-023-057

Figure 3 (continued): Representative Chromatograms from the Injection of Control Whole Apple Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111799 Apple, Centre ID 983732 Spk A)



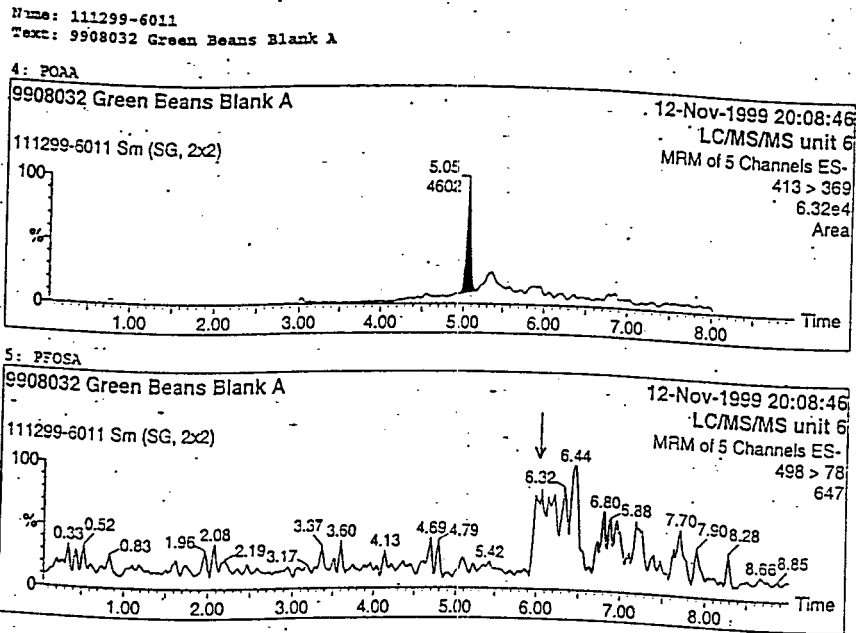
Centre Protocol No. 00P-023-057

Figure 4: Representative Chromatograms of a Control Green Bean Sample (Set 111299 Beans, Centre ID 9908032 Green Beans Blank A)



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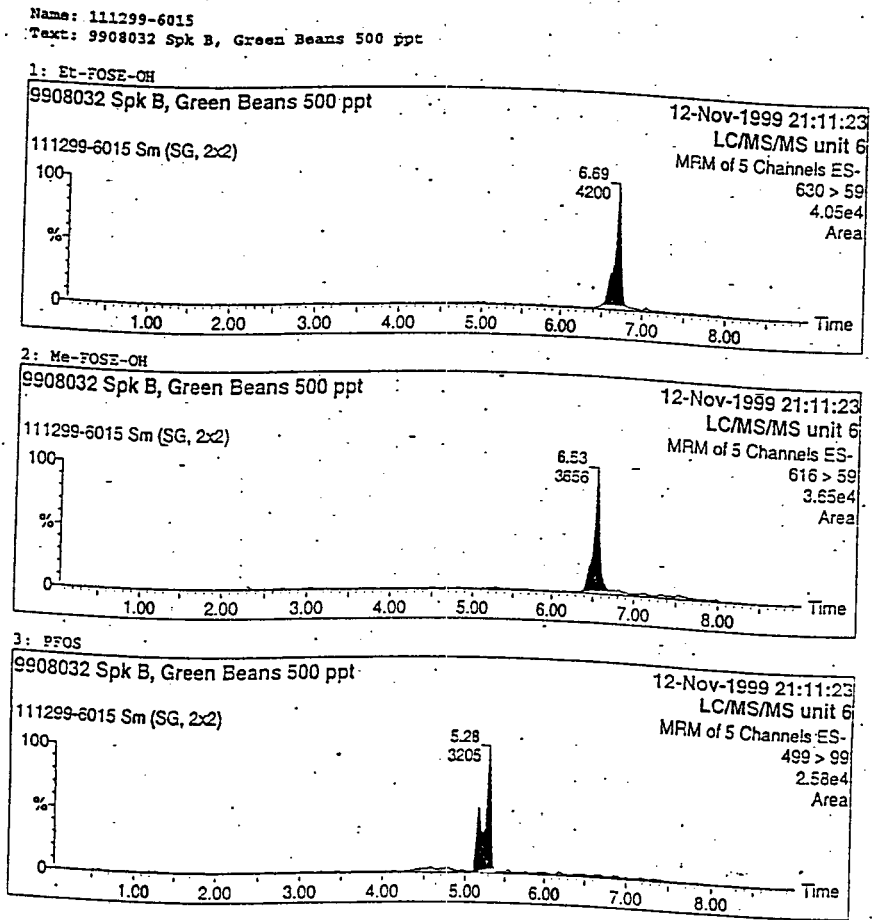
Figure 4 (continued): Representative Chromatograms of a Control Green Bean Sample (Set 111299 Beans, Centre ID 9908032 Green Beans Blank A)





Centre Protocol No. 00P-023-057

Figure 5: Representative Chromatograms from the Injection of Control Green Beans Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111299 Beans, Centre ID 9908032 Spk B)



Centre Protocol No. 00P-023-057

Figure 5 (continued): Representative Chromatograms from the Injection of Control Green Beans Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111299 Beans, Centre ID 9908032 Spk B)

Name: 111299-6015  
Text: 9908032 Spk B, Green Beans 500 ppt

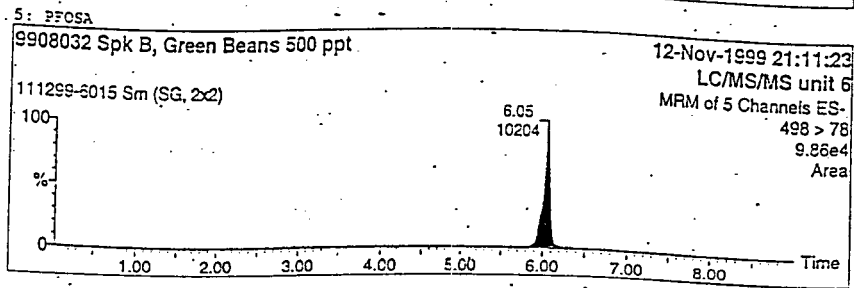
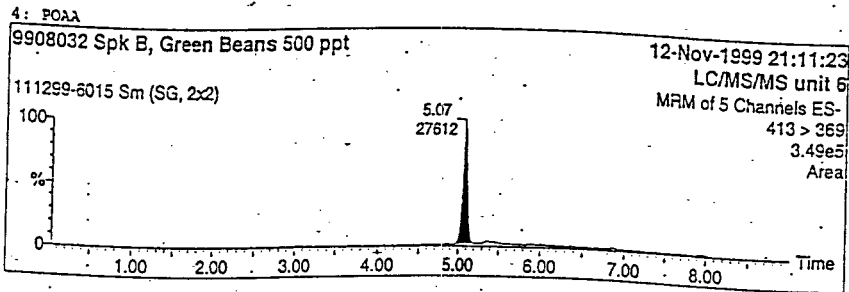
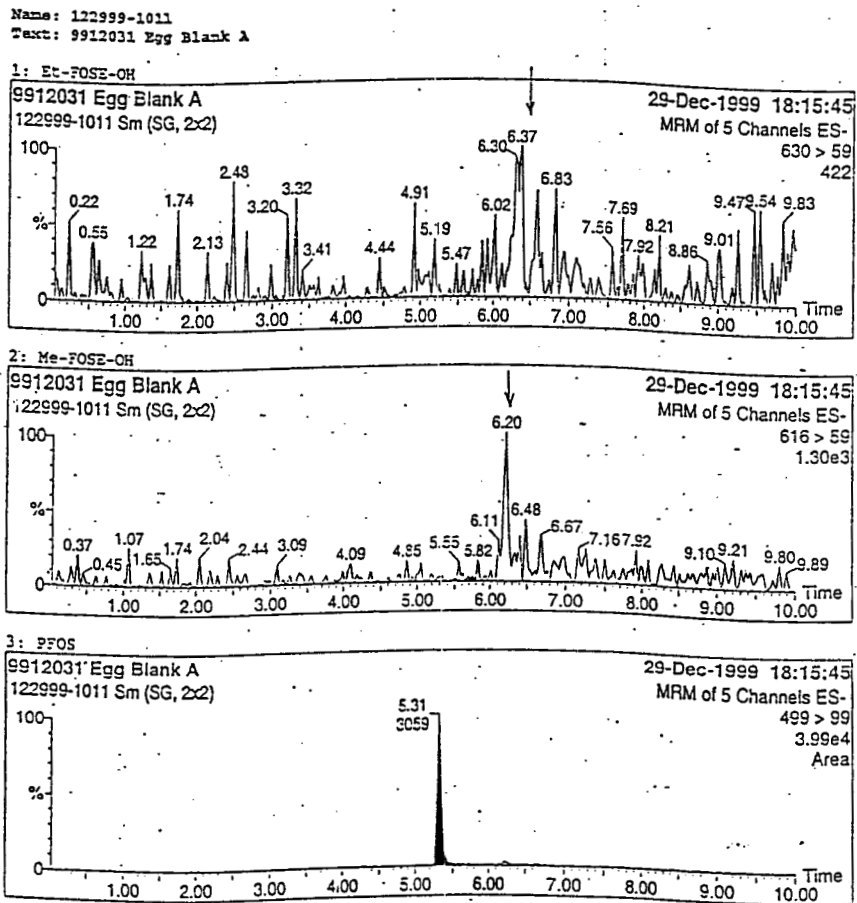
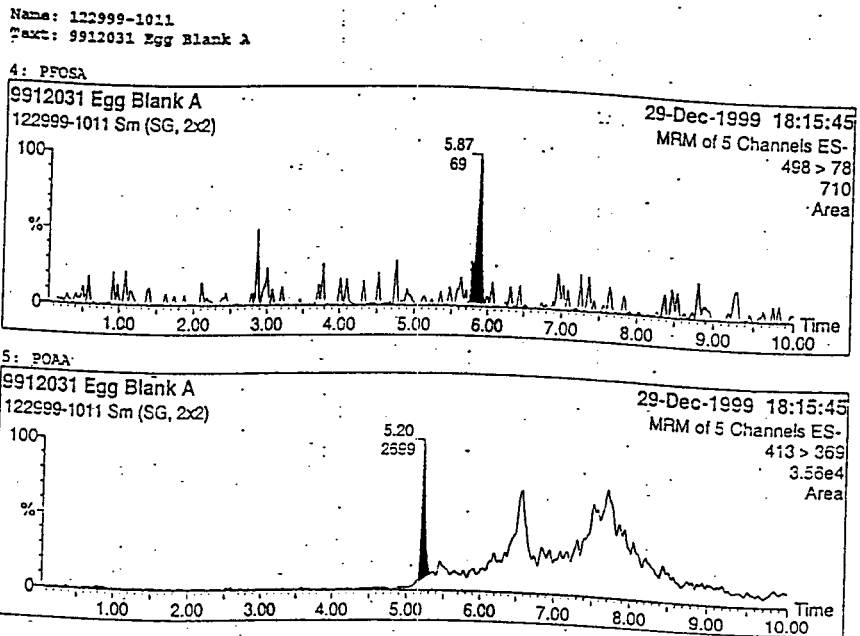


Figure 6: Representative Chromatograms of a Control Chicken Egg Sample (Set 122999 Egg, Centre ID 9912031 Egg Blank A)



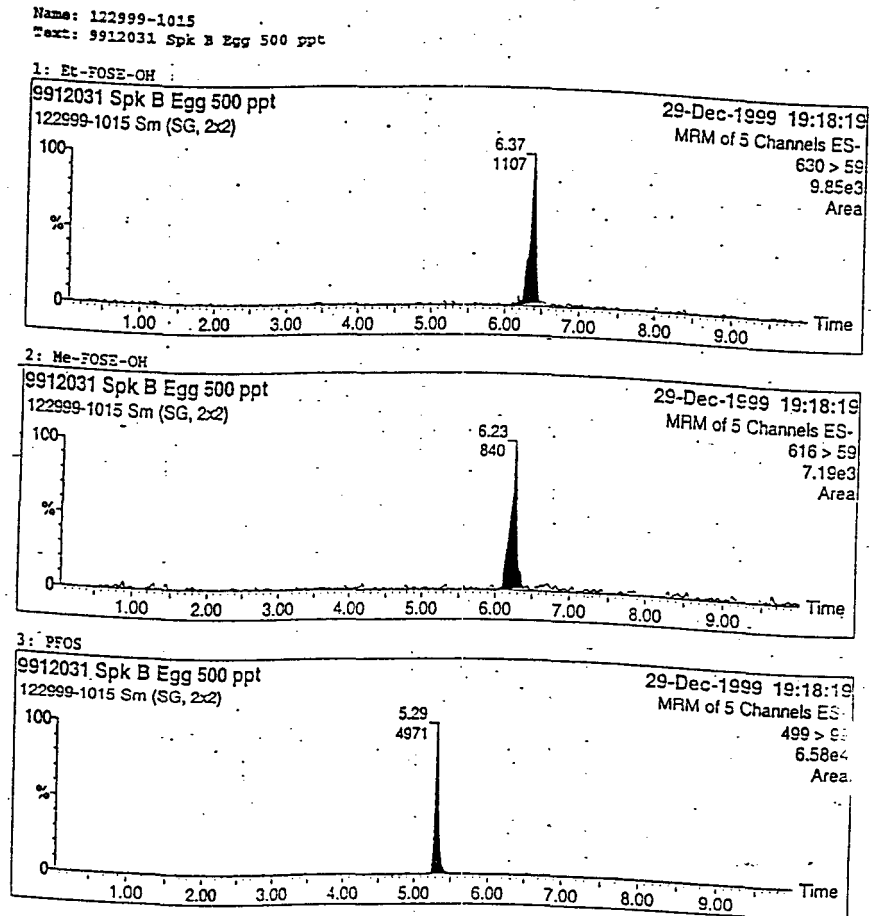
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Figure 6 (continued): Representative Chromatograms of a Control Chicken Egg Sample (Set 122999 Egg, Centre ID 9912031 Egg Blank A)



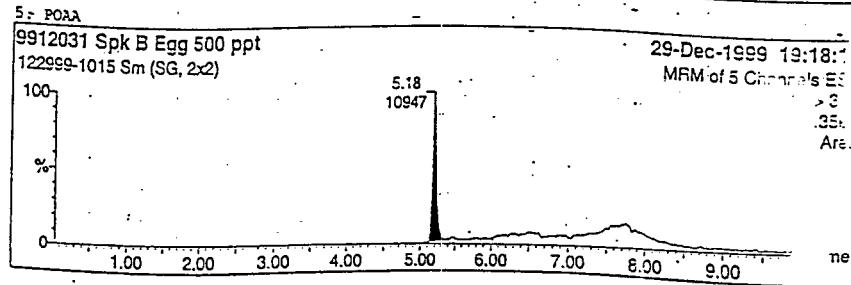
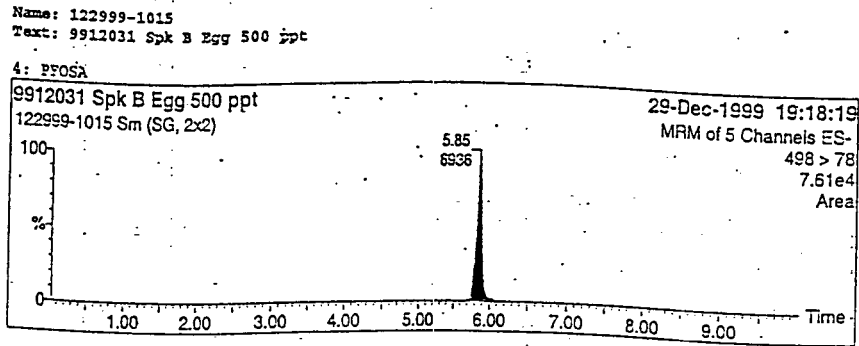
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Figure 7: Representative Chromatograms from the Injection of Control Chicken Egg Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 122999 Egg, Centre ID 9912031 Spk B)



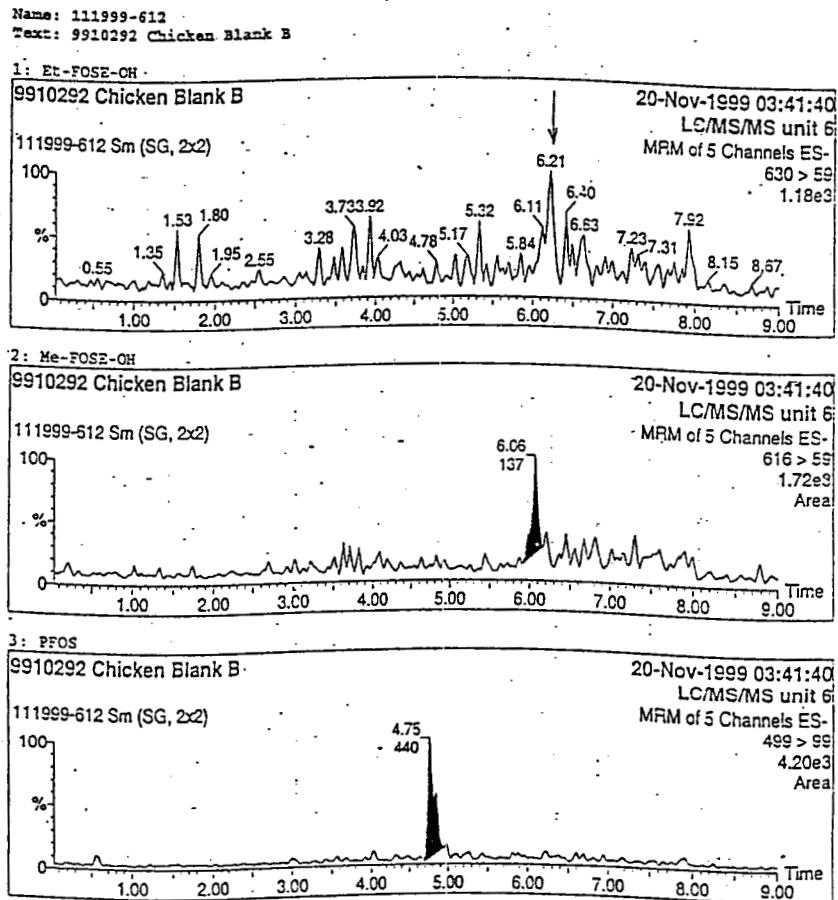
Centre Protocol No. 00P-023-057

Figure 7 (continued): Representative Chromatograms from the Injection of Control Chicken Egg Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 122999 Egg, Centre ID 9912031 Spk B)



Centre Protocol No. 00P-023-057

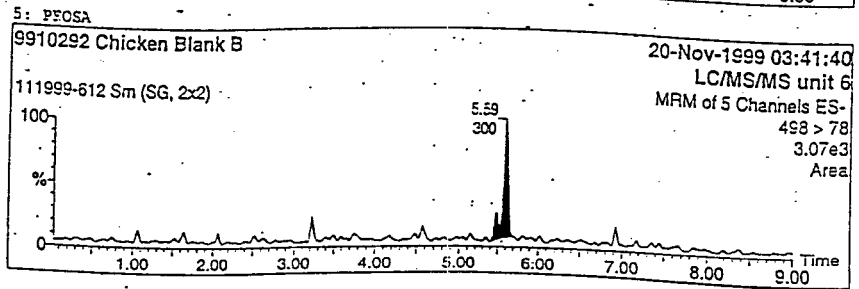
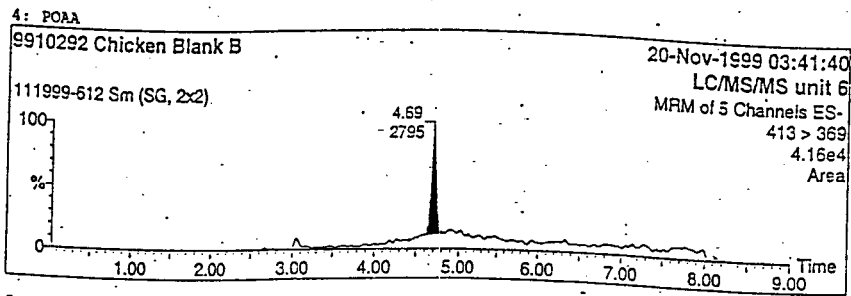
Figure 8: Representative Chromatograms of a Control Chicken Muscle Sample (Set 111999 Chicken, Centre ID 9910292 Chicken Blank B)



Centre Protocol No. 00P-023-057

Figure 8 (continued): Representative Chromatograms of a Control Chicken Muscle Sample (Set 111999 Chicken, Centre ID 9910292 Chicken Blank B)

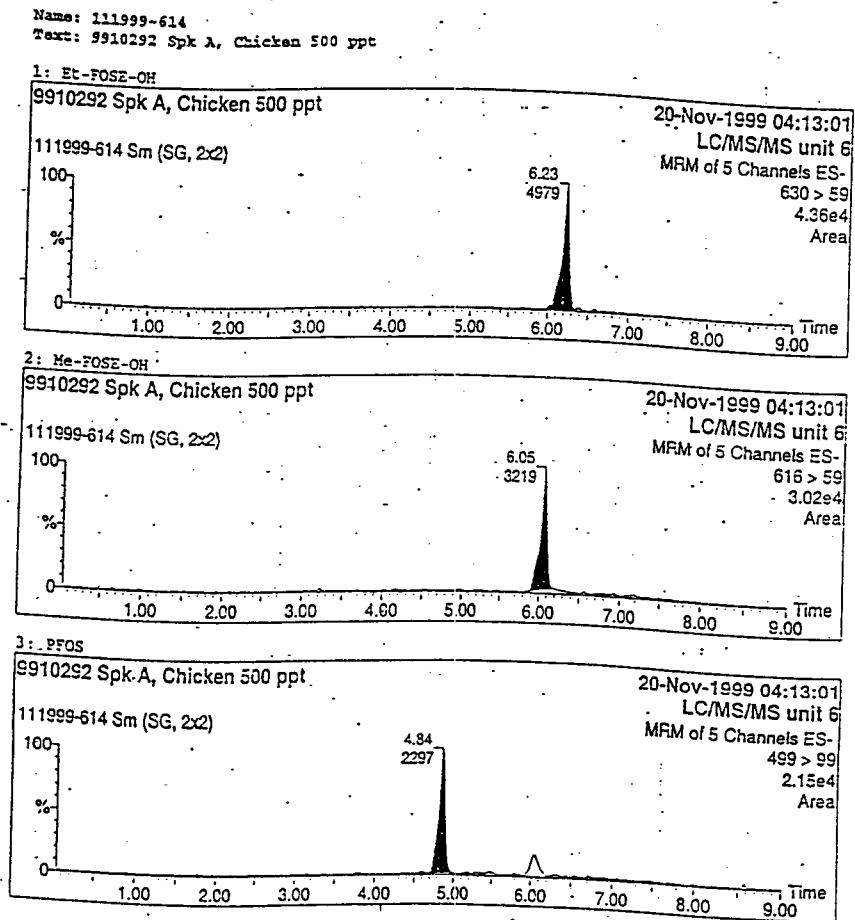
Name: 111999-612  
Text: 9910292 Chicken Blank B





Centre Protocol No. 00P-023-057

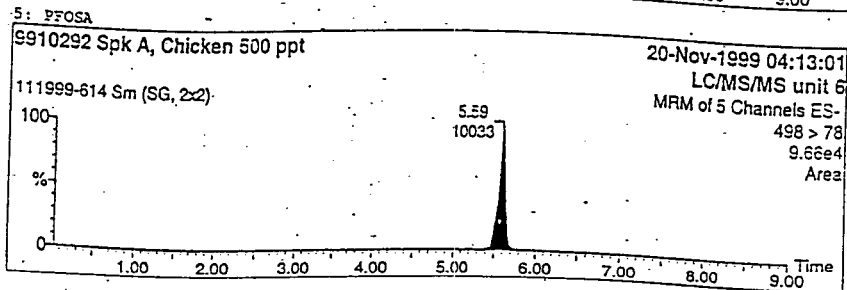
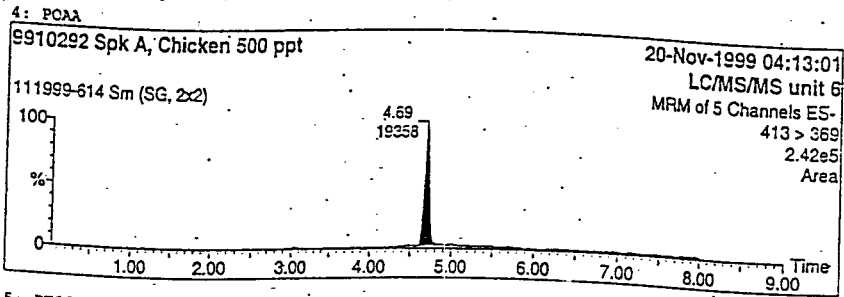
Figure 9: Representative Chromatograms from the Injection of Control Chicken Muscle Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111999 Chicken, Centre ID 9910292 Spk A)



Centre Protocol No. 00P-023-057

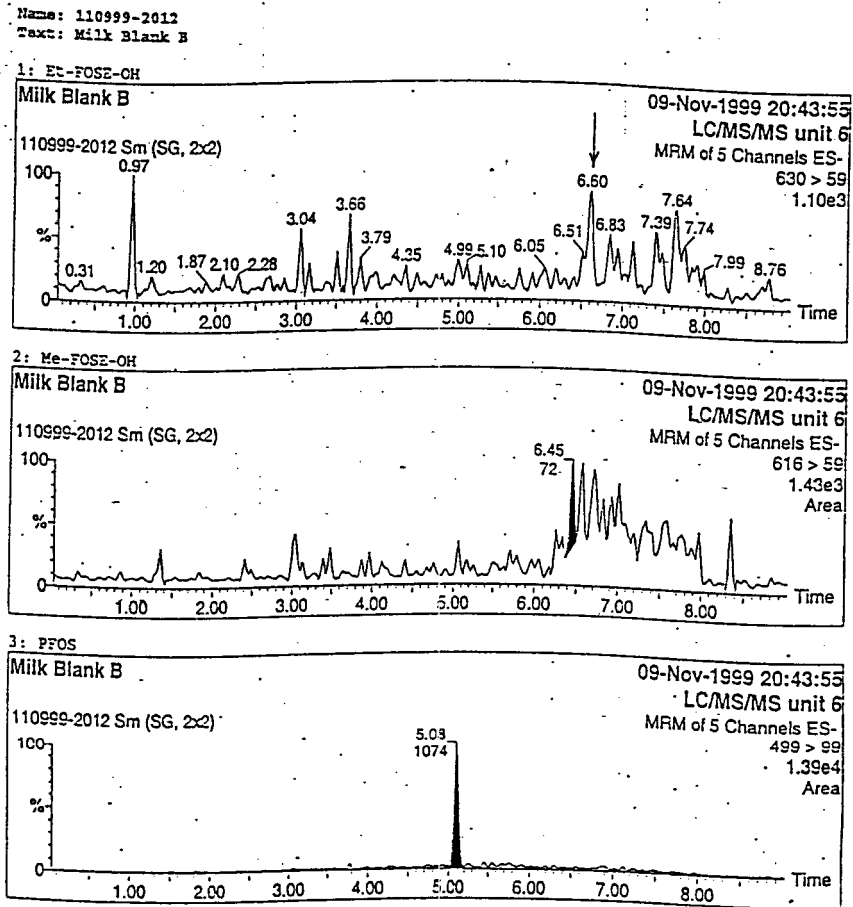
Figure 9 (continued): Representative Chromatograms from the Injection of Control Chicken Muscle Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111999 Chicken, Centre ID 9910292 Spk A)

Name: 111999-614  
Text: 9910292 Spk A, Chicken 500 ppt



Centre Protocol No. 00P-023-057

Figure 10: Representative Chromatograms of a Control Cow Milk Sample (Set 110999 Milk, Centre ID 972627 Milk Blank B)



Centre Protocol No. 00P-023-057

Figure 10 (continued): Representative Chromatograms of a Control Cow Milk Sample (Set 110999 Milk, Centre ID 972627 Milk Blank B)

Name: 110999-2012  
Text: Milk Blank B

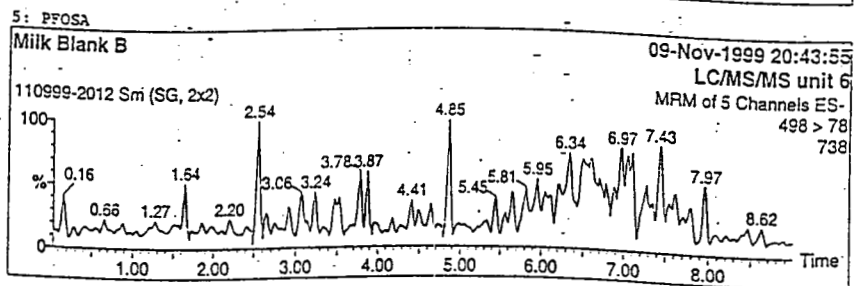
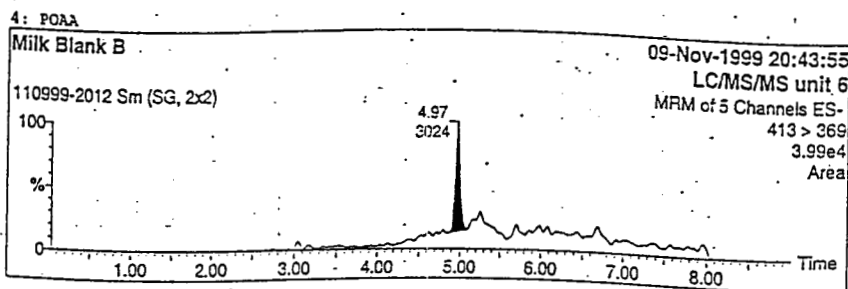
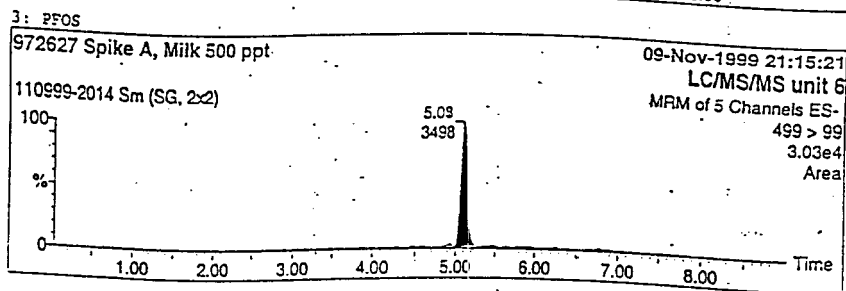
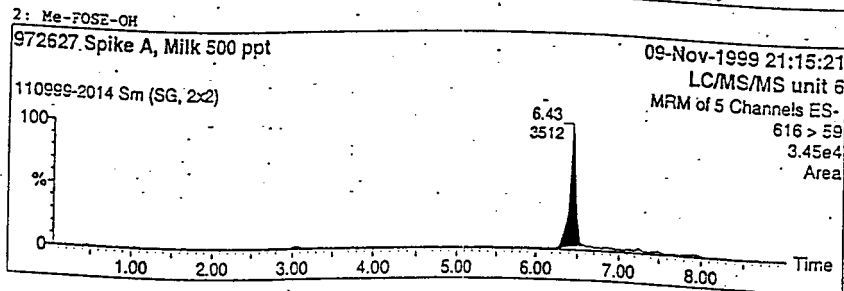
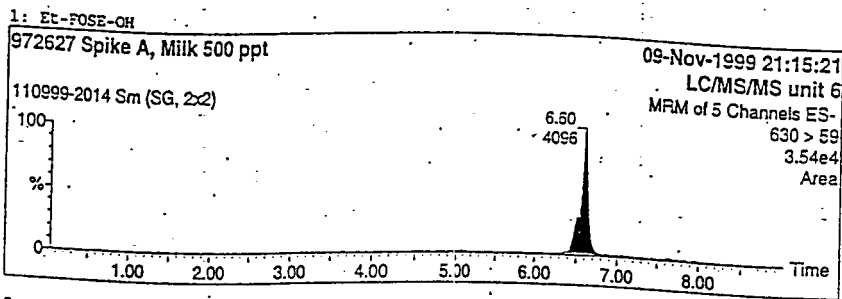


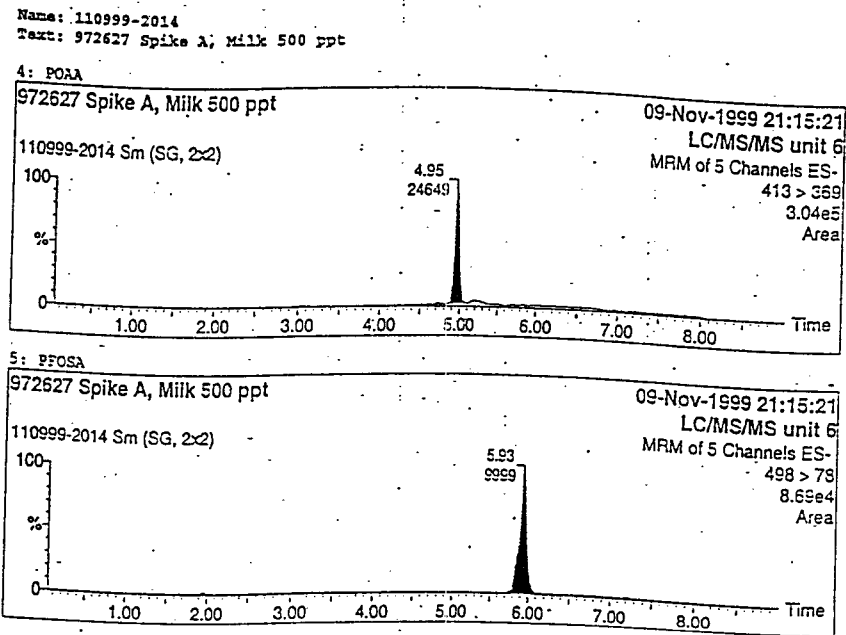
Figure 11: Representative Chromatograms from the Injection of Control Cow Milk Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 110999 Milk, Centre ID 972627 Spk A)

Name: 110999-2014  
Text: 972627 Spike A, Milk 500 ppt



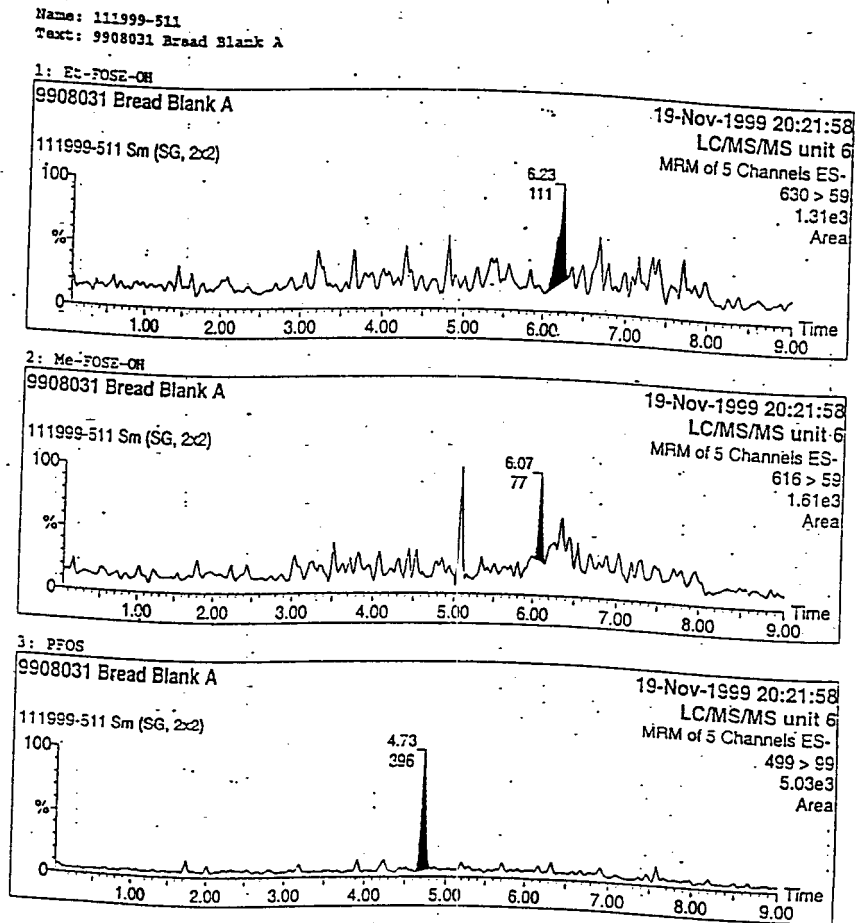
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Figure 11 (continued): Representative Chromatograms from the Injection of Control Cow Milk Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 110999 Milk, Centre ID 972627 Spk A)



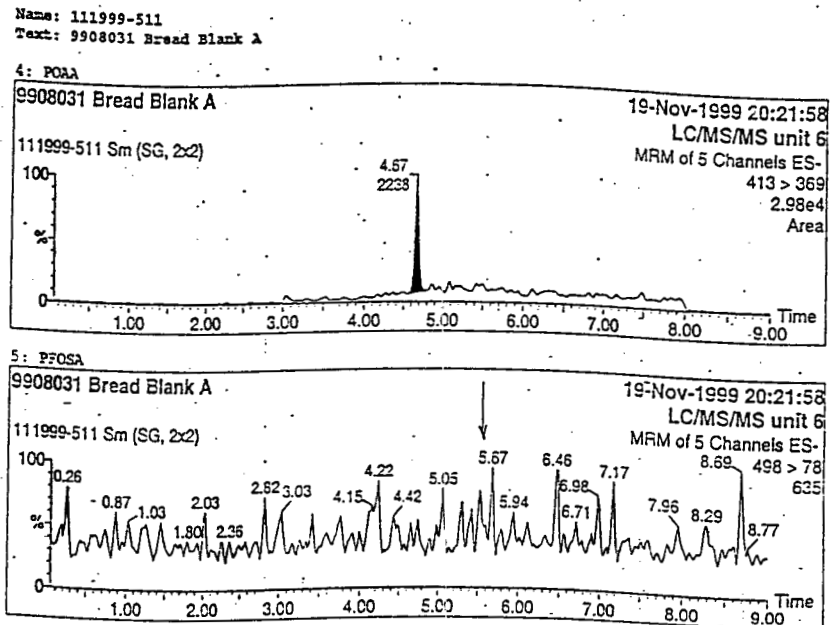
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Figure 12: Representative Chromatograms of a Control Bread Sample (Set 111999 Bread, Centre ID 9908031 Bread Blank A)



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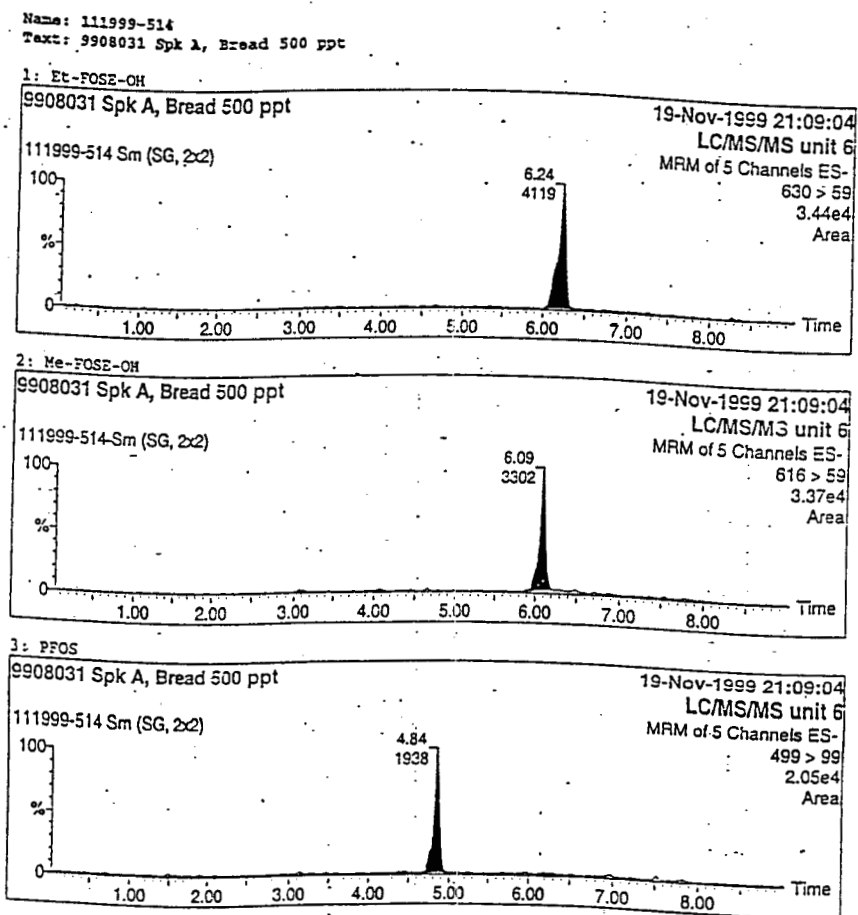
Figure 12 (continued): Representative Chromatograms of a Control Bread Sample  
(Set 111999 Bread, Centre ID 9908031 Bread Blank A)





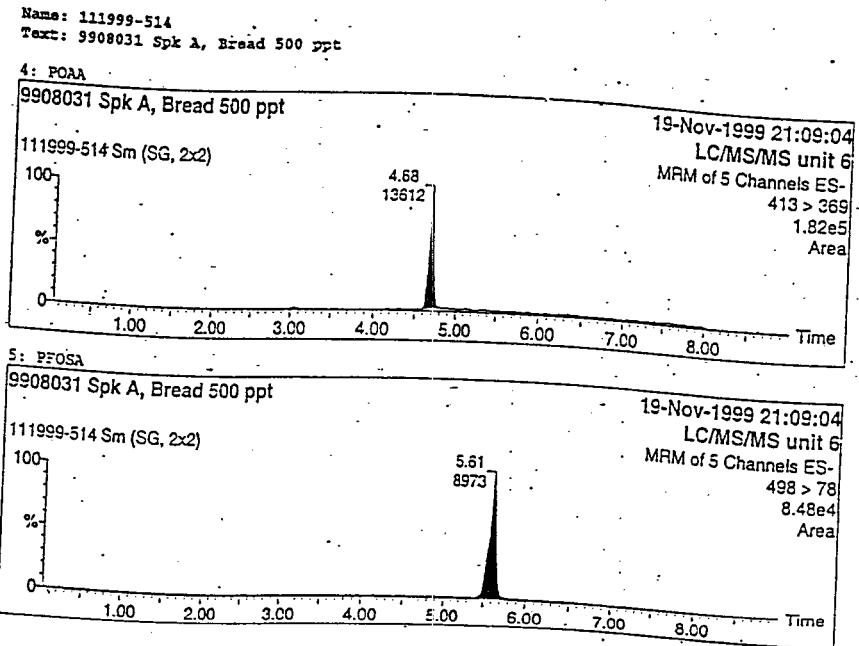
Centre Protocol No. OOP-023-057

Figure 13: Representative Chromatograms from the Injection of Control Bread Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111999 Bread, Centre ID 9908031 Spk A)



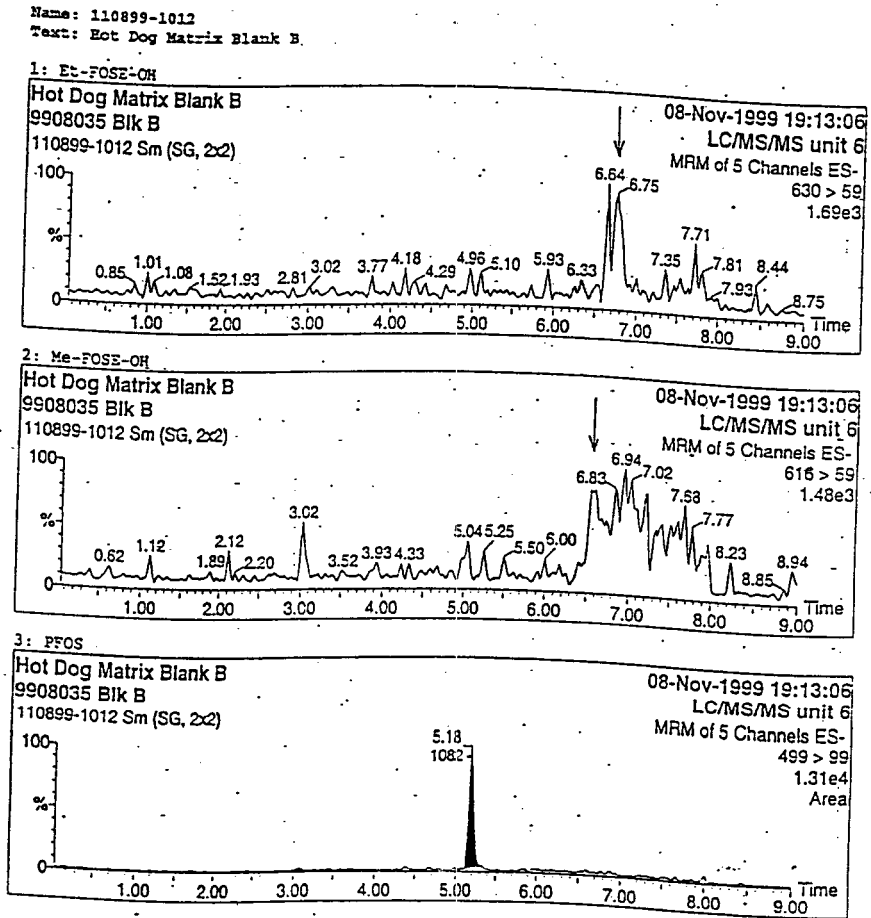
Centre Protocol No. 00P-023-057

Figure 13 (continued): Representative Chromatograms from the Injection of Control Bread Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 111999 Bread, Centre ID 9908031 Spk A)



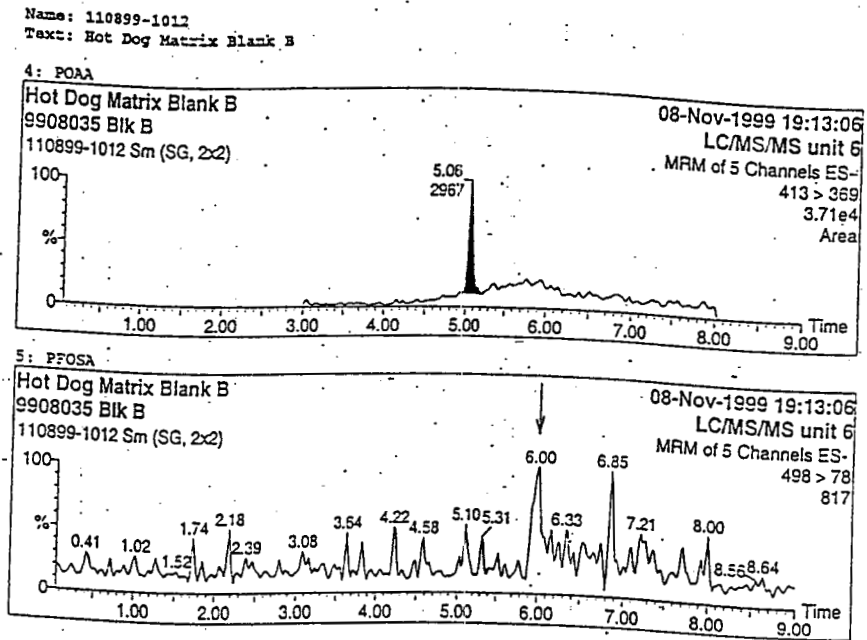
Centre Protocol No: 00P-023-057

Figure 14: Representative Chromatograms of a Control Hot Dog Sample (Set 110899 Hot Dog, Centre ID 9908035 Hot Dog Blank B)



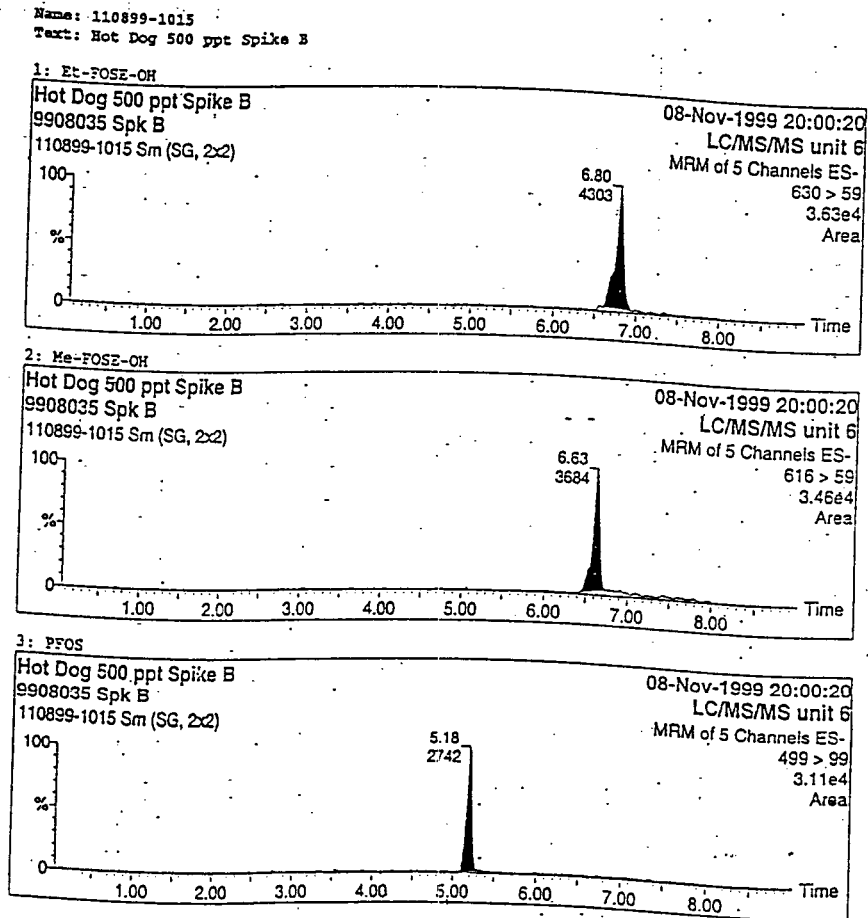
Centre Protocol No. 00P-023-057

Figure 14 (continued): Representative Chromatograms of a Control Hot Dog Sample (Set 110899 Hot Dog, Centre ID 9908035 Hot Dog Blank B)



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Figure 15: Representative Chromatograms from the Injection of Control Hot Dog Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 110899 Hot Dog, Centre ID 9908035 Spk B)



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Figure 15 (continued): Representative Chromatograms from the Injection of Control Hot Dog Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 110899 Hot Dog, Centre ID 9908035 Spk B)

Name: 110899-1015  
Text: Hot Dog 500 ppt Spike B

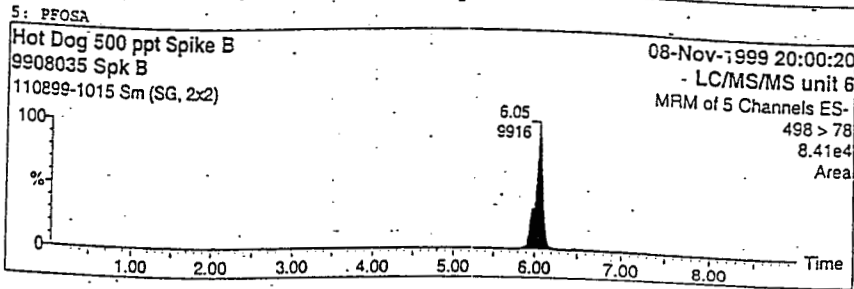
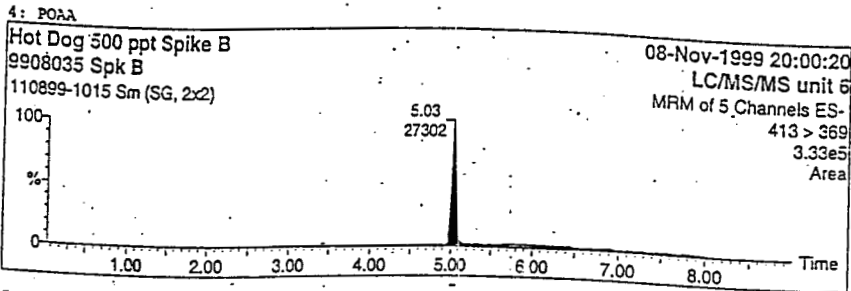
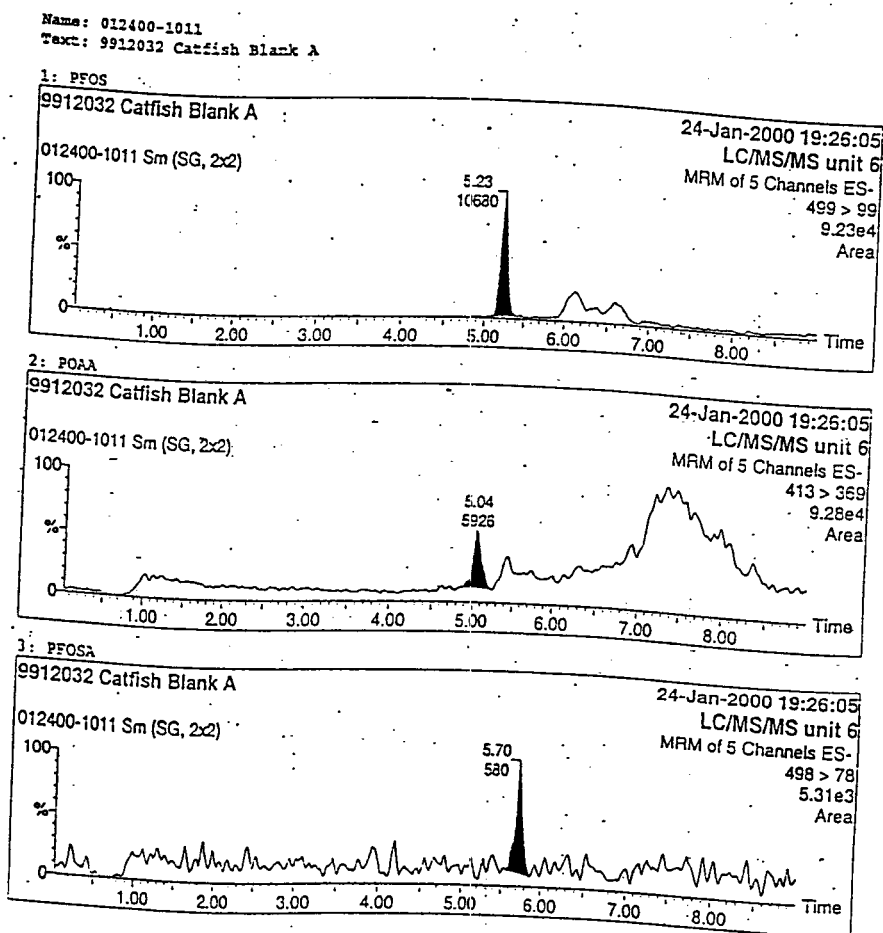


Figure 16: Representative Chromatograms of a Control Catfish Sample (Set 012400 Catfish, Centre ID 9912032 Catfish Blank A)

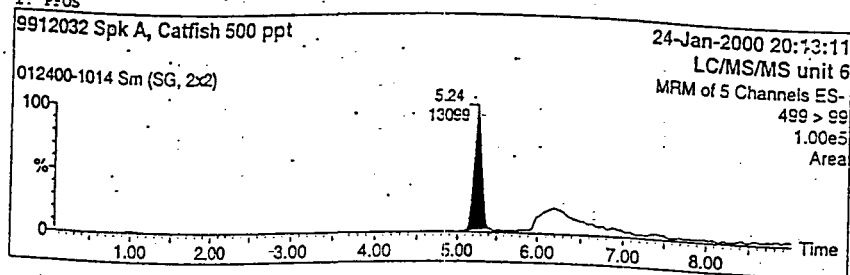


Centre Protocol No. 00P-023-057

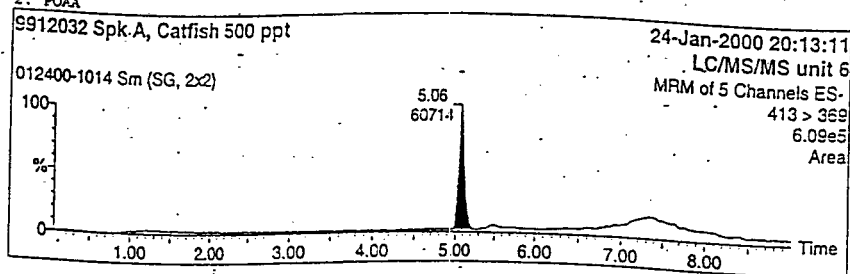
Figure 17: Representative Chromatograms from the Injection of Control Catfish Fortified at 500 ppt each of PFOS, POAA, and PFOSA (Set 012400 Catfish, Centre ID 9912032 Spk A)

Name: 012400-1014  
Text: 9912032 Spk A, Catfish 500 ppt

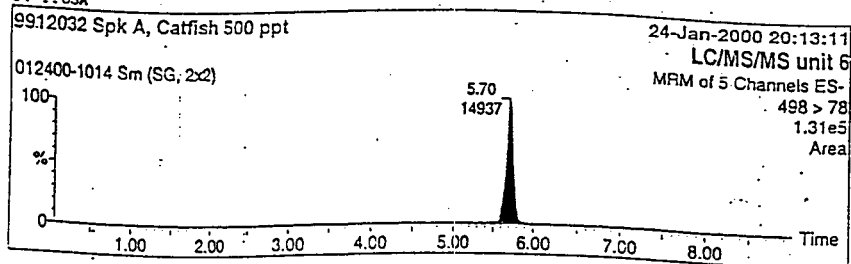
1: PFOS



2: POAA



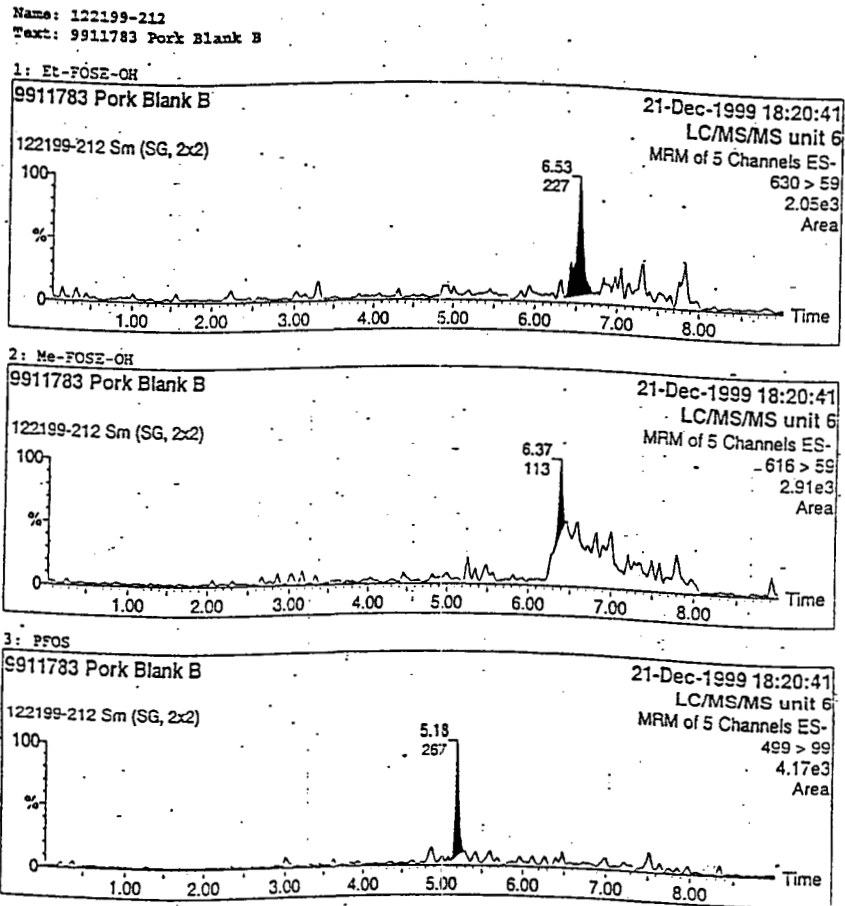
3: PFOSA





Centre Protocol No. OOP-023-057

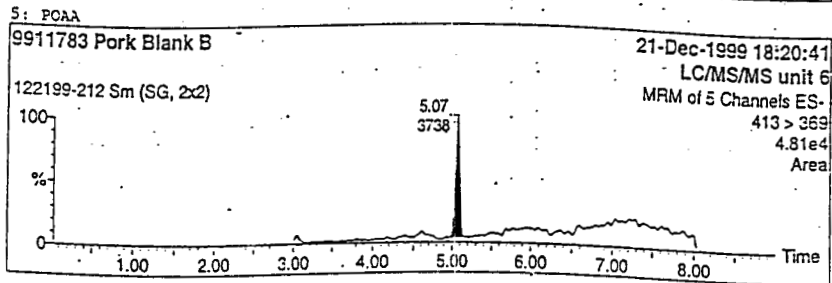
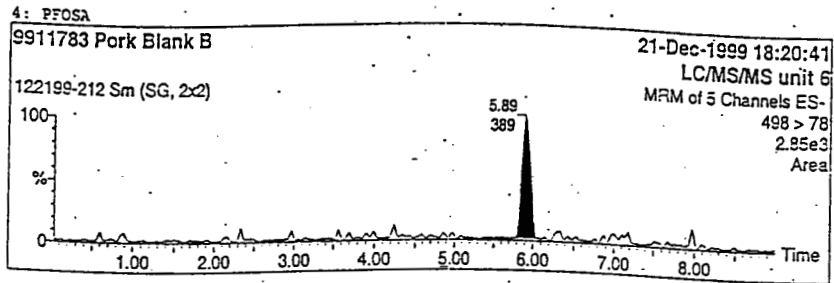
Figure 18: Representative Chromatograms of a Control Pork Muscle Sample (Set 122199 Pork, Centre ID 9911783 Pork Blank B)



Centre Protocol No-00P-023-057

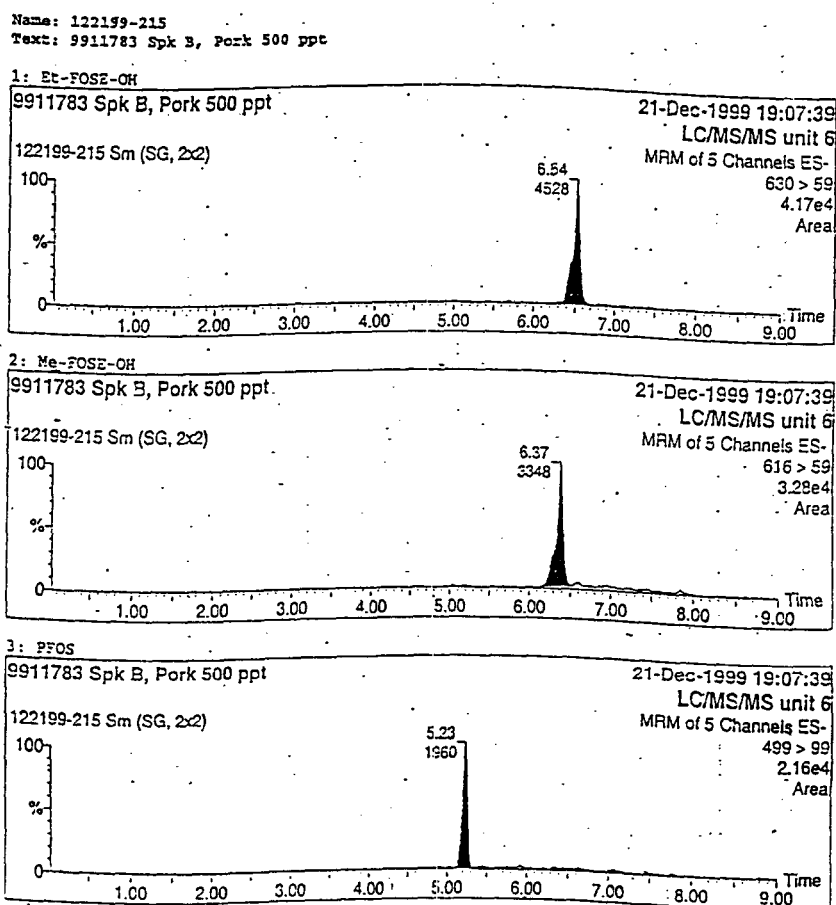
Figure 13 (continued): Representative Chromatograms of a Control Pork Muscle Sample (Set 122199 Pork, Centre ID 9911783 Pork Blank B)

Name: 122199-212  
Text: 9911783 Pork Blank B



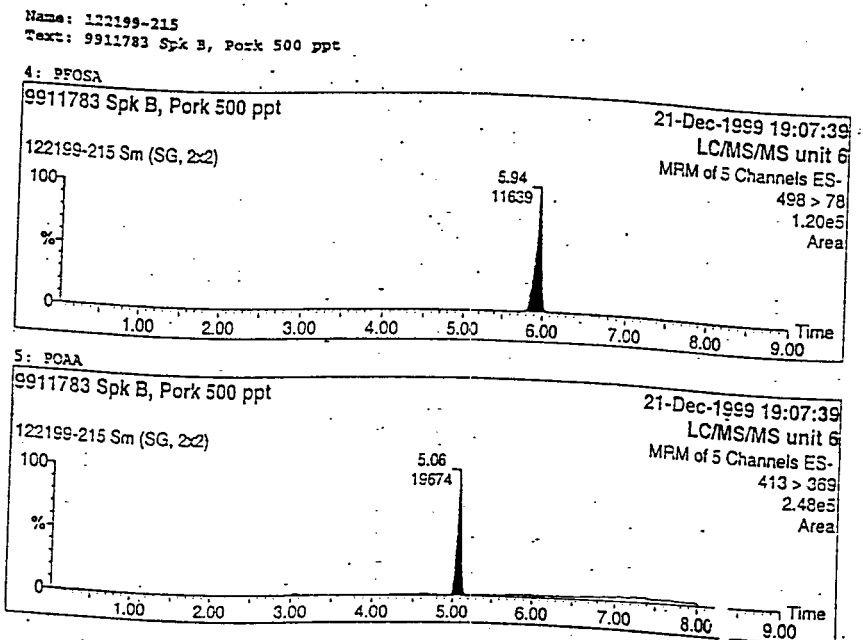
Centre Protocol No. OOP-023-057

Figure 19: Representative Chromatograms from the Injection of Control Pork Muscle Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 122199 Pork, Centre ID 9911783 Spk B)



Centre Protocol No. 00P-023-057

Figure 19 (continued): Representative Chromatograms from the Injection of Control Pork Muscle Fortified at 500 ppt each of Et-FOSE-OH, Me-FOSE-OH, PFOS, POAA, and PFOSA (Set 122199 Pork, Centre ID 9911783 Spk B)





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Page 1 of 1

## PROTOCOL AMENDMENT

Amendment Number: 1

Effective Date: March 26, 2001

Centre Study Number: 023-057

Centre Protocol Number: 00P-023-057

### DESCRIPTION OF AMENDED SECTION

1. Page 5 of Protocol: Assigns Susan Beach as the Study Director at 3M.

### AMENDED TO

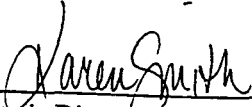
- i. Change the study director to Karen Smith at Centre Analytical Laboratories and make Susan Beach the Sponsor Representative at 3M.

### RATIONALE


1. Reassignment of responsibilities for study at the request of Sponsor.

### IMPACT ON THE STUDY

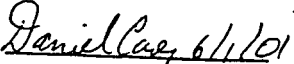
1. There is no negative impact on the study.

  
\_\_\_\_\_  
Study Director Signature

05/01/01  
\_\_\_\_\_  
Date

  
\_\_\_\_\_  
Sponsor Representative Signature

6/6/01  
\_\_\_\_\_  
Date

CAL QAU Review   
February 12, 1998/1



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Page 1 of 1

## PROTOCOL AMENDMENT

Amendment Number: 2  
Effective Date: April 30, 2001

Centre Study Number: 023-057

Centre Protocol Number: 00P-023-057

### DESCRIPTION OF AMENDED SECTION

1. Page 7 of Protocol-Experimental Design: States that each set of samples analyzed will contain at least one matrix control and two fortifications.

### AMENDED TO

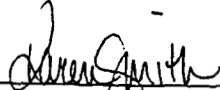
1. Add that each set of samples analyzed will also contain a reagent blank.

### RATIONALE

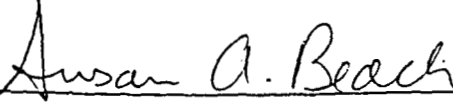
1. Added at request of the sponsor.

### IMPACT ON THE STUDY

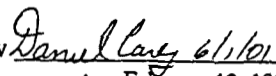
1. There is no negative impact on the study.

  
\_\_\_\_\_  
Study Director Signature

04/30/01  
\_\_\_\_\_  
Date

  
\_\_\_\_\_  
Sponsor Representative Signature

6/6/01  
\_\_\_\_\_  
Date

CAL QAU Review   
February 12, 1998/1



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Page 1 of 1

## PROTOCOL AMENDMENT

Amendment Number: 3

Effective Date: June 15, 2001

Centre Study Number: 023-057

Centre Protocol Number: 00P-023-057

### DESCRIPTION OF AMENDED SECTION

1. Study Title

### AMENDED TO

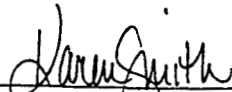
1. Analysis of PFOS, FOSA and PFOA From Various Food Matrices Using HPLC Electropray/Mass Spectrometry.

### RATIONALE

1. Changed for clarity at the request of the sponsor.

### IMPACT ON THE STUDY

1. There is no negative impact on the study.

  
Study Director Signature

06/15/01  
Date

  
Sponsor Representative Signature

6/18/01  
Date

CAL QAU Review NCL 6/15/01

February 12, 1998/1



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Page 1 of 1

## PROTOCOL DEVIATION

Deviation Number: 1

Date of Occurrence: 1) January 19, 2001; 2) February 15, 2001

Centre Study Number: 023-057

Sponsor Study Number: NA

### DESCRIPTION OF DEVIATION

- 1) A recovery of 56% for FOSA is being accepted for the 10.0 ng/g Spk B in data set 011901A Fish.
- 2) A CCV recovery of 143% is being accepted for the 2.5 ng/mL standard for FOSA in data set 021401A Beef.

### ACTIONS TAKEN

i.e., amendment issued, SOP revision, etc...

- 1) Deviation issued.
- 2) Deviation issued.

Recorded By/Date:

*Karen Smith 02/16/01*

### IMPACT ON THE STUDY

- 1) There was no negative impact on the study since the 2.5 ng/mL<sup>30</sup> Spk A for FOSA in data set 011901A Fish was within the 60-130% acceptable range and the levels of residue found in the fish samples were all below 2.5 ng/mL<sup>30</sup> *00804 1/01*
- 2) There was no negative impact on the study since the CCV's before and the CCV run after the standard in question were acceptable.

*Karen Smith*

Principal Investigator Signature

*02/16/01*

Date

NA

Study Director Signature

Date

*[Signature]*

Sponsor Management Signature

*06/11/01*

Date

Centre QAU Review

*Daniel Carey 6/1/01*

January 5, 2001/3