

06 FEB - 1 11 5:05

Analytical Report

Fluorochemical Characterization of Aqueous Samples

Fayetteville C8 Sampling 11/05

Exygen Research Laboratory Report No. L0006767 (Amended Final Report)

Testing Laboratory

Exygen Research
3058 Research Drive
State College, PA 16801

Requester

Michael D. Aucoin
URS Corporation
Barley Mill Plaza
Bldg 19 Room 2118
Lancaster Pike and Rt 141
Wilmington, DE 19805

1 Introduction

Results are reported for the analysis of water samples received by Centre Analytical Laboratories, Inc. (dba Exygen Research) (Exygen) from DuPont Corporation. This report presents results assigned to Exygen Research study number L0006767. This report is an amended report of Exygen Research study number L0006767. This amended report utilizes a different technique for determining signal to noise ratios for the determination of limit of detection and limit of quantification than in the original report.

Specific fluorochemical characterization by liquid chromatography / tandem mass spectrometry (LC/MS/MS) was requested for all samples. A total of 3 samples were received for analysis.

The sample was prepared and analyzed by LC/MS/MS for the following list of fluorochemicals:

- Table 1: Target Analysis

Compound Name	Acronym
Ammonium Perfluorooctanoate	APFO
Perfluorooctanoic Acid	PFOA

2 Sample Receipt

The samples were submitted in plastic containers, cooled with wet ice. The samples were collected on 11/16/05 and were received on 11/17/05. Chain-of-custody information is presented in Attachment C.

3 Holding Times

The analytical method used was validated against a maximum holding time of 14 days in water samples. All samples were analyzed within the required holding time.

4 Methods - Analytical and Preparatory

4.1 LC/MS/MS

4.1.1 Sample Preparation for LC/MS/MS Analysis

Water samples were initially treated with 200 μ L of 250 mg/mL sodium thiosulfate solution to remove residual chlorine. Solid phase extraction (SPE) was used to prepare the samples for LC/MS/MS analysis. A forty-milliliter portion of sample was transferred to a C₁₈ SPE cartridge. The cartridge was eluted with 40% methanol: water. The eluate was discarded. The cartridge was then eluted with 100% methanol. A 5 mL portion of methanol was collected for analysis by LC/MS/MS. This treatment resulted in an eight-fold concentration of the samples prior to analysis.

4.1.2 Sample Analysis by LC/MS/MS

A Hewlett-Packard HP1100 HPLC system coupled to a Micromass Ultima MS/MS was used to analyze the sample extracts. Analysis was performed using selected reaction monitoring (SRM). Samples were extracted and analyzed on 11/18/05. Raw analytical data can be found in Attachment D.

5 Analysis

5.1 Calibration

An 8-point calibration curve was analyzed at the beginning of the analytical sequence and throughout the run for the compounds of interest. The calibration points were prepared at 5, 10, 25, 50, 100, 250, 500, and 1000 ng/L (ppt), as perfluorooctanoic acid (PFOA). The instrument response versus the concentration was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and correlation coefficient (r) and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r \geq 0.992$ ($r^2 \geq 0.985$).

Calibration standards for MS/MS analysis were prepared using the same SPE procedure used for samples.

For the results reported here, calibration criteria were met.

5.2 Blanks

Extraction blanks were prepared and analyzed with every extraction batch of samples. The extraction blanks should not have any target analytes present at or above the limit of detection. For these samples, the extraction blanks were compliant.

Instrument blanks in the form of clean methanol solvent were also analyzed after every high-level calibration standard, and after known high-level samples. Again, the blanks should not have any target analytes present at or above the limit of detection. For the samples presented here the instrument blanks are compliant.

5.3 Surrogates

Surrogate spikes are not a component of the LC/MS/MS analytical method.

5.4 Matrix Spikes

A matrix spike was prepared for each field sample at 5000 ng/L. The matrix spike recovery was within the acceptable range of 70 – 130%. Matrix spike recoveries are given in Attachment B.

5.5 Duplicates

Each field sample was analyzed in duplicate. Results are given along with the sample results in Attachment A.

5.6 Laboratory Control Samples

Milliq water was spiked with PFOA at 50 and 500 ng/L. with each extraction set. All laboratory control samples showed recoveries between 70 and 130%.

5.7 Sample Related Comments

A single analytical run was performed to complete the sample analysis. The following is a brief quality control narrative for the run.

Analytical Run Number	Samples Analyzed	Comments
111805 A	FAY-D-4328MLR, FAY-D-6999PED, FAY-K-FBLK-1	All quality control results were acceptable and all data are useable.

6 Data Summary

Please see Attachment A for a detailed listing of the analytical results. All results are reported in (ng/L) parts per trillion as APFO and PFOA.

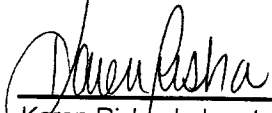
7 Data/Sample Retention

Samples are disposed of one month after the report is issued unless otherwise specified. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by Exygen.

8 Attachments

- 8.1 Attachment A: Results
- 8.2 Attachment B: Matrix Spike Recoveries
- 8.3 Attachment C: Chain of Custody
- 8.4 Attachment D: LC/MS/MS Raw Analytical Data
- 8.5 Attachment E: Standards Data

9 Signatures



Karen Risha, Laboratory Supervisor

01/10/06

Date



John M Flaherty, Vice President

1/10/06

Date

Analytical Results Fayetteville C8 Sampling 11/05**DuPont Sample Identification****APFO (ng/L)****PFOA (ng/L)**

FAY-D-4328MLR	NQ	NQ
FAY-D-4328MLR (laboratory duplicate)	NQ	NQ
FAY-D-6999PED	ND	ND
FAY-D-6999PED (laboratory duplicate)	ND	ND
FAY-K-FBLK-1	ND	ND

Limit of Detection (LOD) for the procedure is approximately 4.5 ng/L

Limit of Quantitation (LOQ) for the procedure is 22 ng/L

ND - Compound not detected

NQ - Compound detected at a level between the LOD and LOQ. Result is not quantifiable.

ND < LOD < NQ < LOQ

0006

Analytical Results Fayetteville C8 Sampling 11/05**DuPont Sample Identification****APFO (ng/L)****PFOA (ng/L)**

FAY-D-4328MLR
FAY-D-6999PED
FAY-K-FBLK-1

NQ	NQ
ND	ND
ND	ND

Limit of Detection (LOD) for the procedure is approximately 4.5 ng/L

Limit of Quantitation (LOQ) for the procedure is 22 ng/L

ND - Compound not detected

NQ - Compound detected at a level between the LOD and LOQ. Result is not quantifiable.

ND < LOD < NQ < LOQ

Results are calculated according to the following criteria

If the sample and laboratory duplicate are greater than 5X LOQ, and the relative percent difference (RPD) is less than 20, the average value is reported. If the RPD is greater than 20, the higher value is reported.

If the sample and laboratory duplicate are less than 5X LOQ, and the absolute difference is less than LOQ, the average value is reported. If the absolute difference is greater than LOQ, the higher value is reported.

0007

Attachment B: LC/MS/MS Laboratory Spike RecoverySample ID: Spiked Amount (ng/L):

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Recovery (%)	Criteria (Pass / Fail)
PFOA	9.95	5870	117.2	PASS

Lower Recovery Limit: Upper Recovery Limit:

Concentrations represent amount present as the PFOA anion

Note: Sample results less than 13 ng/L are reported as NQ in the results section as they are below the limit of quantitation. Results are given in this table for recovery calculations only.

Attachment B: LC/MS/MS Laboratory Spike RecoverySample ID: Spiked Amount (ng/L):

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Recovery (%)	Criteria (Pass / Fail)
PFOA	0	5250	105.0	PASS

Lower Recovery Limit: Upper Recovery Limit:

Concentrations represent amount present as the PFOA anion

.

1

Login

Login Group: L0006767

Login #:	6878	Conform COC Sample:	True
Project:	P0000418	Conform COC:	True
Company Name:	DuPont	Conform Sample:	True
Submitted By:	Michael D. Aucoin	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	11/17/2005		
Due Date:	11/27/2005		
Received Date:	11/17/2005		
Received By:	Ammerman, Mark		
Spread Sample:			
Label:			
Exygen SD/PI:	Risha, Karen		
Project Title/Type:	Analysis of Fluorochemicals in Water / ROUTINE		
Login Notes:			

Packages / Containers

Package	Carton	Date / Condition		Shipper / ID	Temp. Control/Temp.	Direction / Handled By
*K0007848		Received Date: 11/17/05 11:10 Package & Contents Uncompromised		FEDEX 72742 6564 6567	Wet Ice 0.8	RECEIVED Ammerman, Mark
Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0119853	562.80 g	8.40	500 ml Clear Plastic Narrow	NONE		
C0119854	551.20 g	8.20	500 ml Clear Plastic Narrow	NONE		
C0119855	540.90 g	4.70	500 ml Clear Plastic Narrow	NONE		
C0119856	549.00 g	4.40	500 ml Clear Plastic Narrow	NONE		
C0119857	574.40 g	9.70	500 ml Clear Plastic Narrow	NONE		
C0119858	585.80 g	9.80	500 ml Clear Plastic Narrow	NONE		

Samples

Sample ID	Container	Matrix	Fraction	Sample	Date Sampled	Date Due
L0006767-0001	C0119853 C0119854	LIQUID	Water	FAY-D-4328MLR	11/16/2005	11/27/2005
L0006767-0002	C0119855 C0119856	LIQUID	Water	FAY-D-6999PED	11/16/2005	11/27/2005
L0006767-0003	C0119857 C0119858	LIQUID	Water	FAY-K-FBLK-1	11/16/2005	11/27/2005

0010



Login

Login Reviewed By: Bon S. McCune Date/Time: 11/21/05 845

0011

CHAIN OF CUSTODY/ANALYSIS REQUEST FORM

Exygen Research Sample Receiving • 3048 Research Drive • State College, PA 16801, USA
T: 814.231.8032 • F: 814.231.1580 • exygenresearch.com

Page 7 of 7

Client (name & address):

Atch. At Service Dept

Project Manager (Name & E-mail Address):

Milk Arcs

Project Name:

PO #. _____

Quotation #:

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

SAMPLE ANALYSIS

Ex/LIMS#	Client Sample Identification	Collection Date	Collection Time	Grab	Comp	Number Contained	Specify Matrix	Comments						
	FA4-D-4328 M/LR	11.16.05	1316	X		2	6.46	9933512, 9933513	X					8.482
	FA4-D-6999PE1D	11.16.05	1413	X		2	"	9933513, 9933514	X					4.744
	FA4-D-6999PE1D					2	"		X					
	FA4-D-6999PE1D					2	"		X					
	FA4-D-6999PE1D					2	"		X					
	FA4-D-6999PE1D					2	"		X					
	FA4-D-6999PE1D					2	"		X					
	FA4-K-F66K-1	11.16.05	1520	X		2	"	9933509, 9933508	X	X				9.794

CHAIN OF CUSTODY

Relinquished by	Date	Time
B. H. H. H.	11.16.05	1530

Cooler ID #

Glenn?

Cooler Temp. (°C) 0.8

Received by	Date	Time
<i>Black</i>	11.16.05	1130
<i>Black</i>	11/17/07	1110

OTHER INFORMATION

PROJECT REQUIREMENTS

Results Deadline:

Laboratory Report Options:

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

0012

ORIGIN ID: FAYA (910) 678-1146
 KEN LEADBETTER
 E I DUPONT/SITE SUPPORT
 22828 NC HIGHWAY 87 W

FAYETTEVILLE, NC 28306
 UNITED STATES US

Ship Date: 16NOV05
 Actual Wgt: 38.0 LB MAN
 System#: 385790/CAFE2264
 Account: S 028300174
 Dimmed: 13x14x24 IN

TO ATTN: SAMPLE RECEIVING
 EXYGEN RESEARCH
 3048 RESEARCH DRIVE

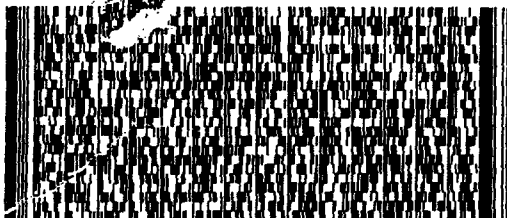
(814) 231-8032

FedEx
 Express

STATE COLLEGE, PA 16801



REF:



Delivery Address
 Barcode

BILL THIRD PARTY

PRIORITY OVERNIGHT

THU

Deliver By:

17NOV05

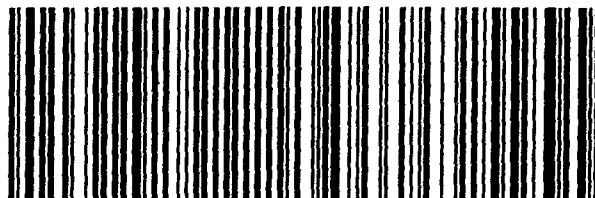
TRK# 7242 6564 6567 Form 0201

PIT AA

16801 -PA-US

NV SCEA

Part # 156148-434 NRIT 10-04



RAW DATA REPORT

Sponsor Study No: NA
 Exygen Study No: L6767
 Analyte: APFO
 Ions Monitored: 413 → 369
 Site: Fayetteville

Limit of Quantitation (LOQ): 22.4 ng/L
 Limit of Detection (LOD): 4.47 ng/L
 Injection Volume: 15 µL
 Matrix: Water
 Sample Volume: 40.0 mL
 Final Volume: 5.0 mL

Set No: 111805A
 Analyst: Karen Risha
 Instrument Type: LC/MS/MS Unit # 7
 Extraction Date: 11/18/05
 Analyzed on: 11/18/05

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC110705-0	-	C	111805A-1001	0	-	0	-	-	-	-
XC110705-1	-	CS	111805A-1002	5	-	494	-	-	-	-
XC110705-2	-	CS	111805A-1003	10	-	826	-	-	-	-
XC110705-3	-	CS	111805A-1004	25	-	2065	-	-	-	-
XC110705-4	-	CS	111805A-1005	50	-	3584	-	-	-	-
XC110705-5	-	CS	111805A-1006	100	-	7752	-	-	-	-
XC110705-6	-	CS	111805A-1007	250	-	16331	-	-	-	-
XC110705-7	-	CS	111805A-1008	500	-	35308	-	-	-	-
XC110705-8	-	CS	111805A-1009	1000	-	69493	-	-	-	-
XC110705-9	-	CK	111805A-1010	250	-	16694	-	-	-	-
Methanol Wash	-	C	111805A-1011	-	-	0	-	-	-	-
Reagent Control L6767-3	NA	C	111805A-1012	-	1	73	ND	ND	-	-
Reagent Spk A	FAY-K-FBLK-1	S	111805A-1013	-	1	0	ND	ND	-	-
Reagent Spk B	NA	LCS	111805A-1014	-	1	4065	55.5	57.8	50	111
L6767-1 Spk C	NA	LCS	111805A-1015	-	1	39560	555	578	500	111
L6767-2 Spk D	FAY-D-4328MLR	LF	111805A-1016	-	10	41817	5870	6110	5000	117
Methanol Wash	FAY-D-6999PED	LF	111805A-1017	-	10	37432	5250	5470	5000	105
XC110705-1	-	C	111805A-1018	-	-	0	-	-	-	-
XC110705-2	-	CS	111805A-1019	5	-	410	-	-	-	-
XC110705-3	-	CS	111805A-1020	10	-	807	-	-	-	-
XC110705-4	-	CS	111805A-1021	25	-	1937	-	-	-	-
L6767-1	-	CS	111805A-1022	50	-	3814	-	-	-	-
L6767-1 Rep	FAY-D-4328MLR	S	111805A-1023	-	1	832	9.95	NQ	-	-
L6767-2	FAY-D-4328MLR	S	111805A-1024	-	1	917	11.1	NQ	-	-
L6767-2 Rep	FAY-D-6999PED	S	111805A-1025	-	1	0	ND	ND	-	-
XC110705-5	FAY-D-6999PED	S	111805A-1026	-	1	0	ND	ND	-	-
XC110705-6	-	CS	111805A-1027	100	-	7939	-	-	-	-
XC110705-7	-	CS	111805A-1028	250	-	17931	-	-	-	-
XC110705-8	-	CS	111805A-1029	500	-	35873	-	-	-	-
XC110705-9	-	CS	111805A-1030	1000	-	72903	-	-	-	-

PFOA Found (ng/L) = (peak area - intercept) / slope x DF

Recovery (%) = $\frac{[\text{PFOA found (ng/L)} - \text{PFOA found in control (ng/L)}]}{\text{amount PFOA added (ng/L)}} \times 100$

APFO Found (ng/L) = PFOA found (ng/L) x (MW APFO (431) / MW PFOA (414))

CS = Calibration standard
 C = Control sample
 S = Sample

LF = Lab fortified sample
 FF = Field fortified sample
 LCS = Laboratory Control Spike

CK = Check Standard
 ND = Not detected = Response between 0 and LOD
 NQ = Not quantifiable = Response between LOD and LOQ

Avg Resp. of the 250 ppt Standards (area): 17131
 Response of the Check Standard (area): 16694
 Percent Difference (%): 3
 % difference acceptable if within ±15%

Spreadsheet prepared by:

RF 01/06/06

Internal Chain of Custody/Fortification Sheet

Oxygen Study Number: L6767

Matrix: Water

Sponsor Study/Protocol No: NA

The samples listed below were removed from refrigerator No. 34

Time 8:05 AM

Date 11/18/2005

Initials EPD

[illegible][illegible]

All samples were measured:

Time 10:45 AM

Date 11/18/2005

Initials *EDD*

After measuring samples were returned to refrigerator No. 32

Time 3:40 PM

Date 11/18/2005

Initials EDD 11/18/2005

Comments: 200 μ L of 250 mg/mL sodium thiosulfate was added to all samples before spiking. Initials/Date: CDN 11/18/05

Analysis Summary:

Data Set: 111805A

Initials/Date: be / 11/18/05

Data Set:

Initials/Date: DE / 11/10/15
Initials/Date: DE / -

Data Set: _____

Initials/Date:

Set extraction/analysis data verified by: DMF

0015

Date: ~~1/19~~ 11/21/05

July 26, 2001/6

3058 Research Drive
State College, PA 16801

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

METHOD: 01M-008-046^{SS}
ANALYTES: PFOA

PROTOCOL NUMBER: NA

[illegible]

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 1.5 mL polypropylene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

^Initials and date under each step indicates the personnel that performed this step.

\$\$\$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Water

COMMENTS:

Final extracts stored in refrigerator 32 Initials: SD Date: 11/18/2005

July 12, 2005/5



3058 Research Drive
State College, PA 16801

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

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Protocol No.: None
Method No.: 01M-008-046*

Exygen Study No.: NA
Analytes: PFOA

Matrix: Water
Sample Vol: 40 mL
Exygen Study No.: 66767

Sponsor Sample ID	Exygen Solution ID	Sample Description	Fort. Solution ID	Fort. Soln. Conc. (ng/mL)	Fort. Volume (μL)	Micropipet used (μL)	Fort. Level (ppt)	Final Solution ID **	Reagents/ Materials	Lot #
NA	SL0016609	Water^	-	-	-	-	-	XC110705-0	Methanol	45227
NA	SL0016609	Water^	F091305-5	10	20	20	5	XC110705-1	C18 SPE	0045352314
NA	SL0016609	Water^	F091305-5	10	40	200	10	XC110705-2	-	-
NA	SL0016609	Water^	F091305-5	10	100	200	25	XC110705-3	-	-
NA	SL0016609	Water^	F091305-5	10	200	200	50	XC110705-4	-	-
NA	SL0016609	Water^	F091305-5	10	400	200	100	XC110705-5	-	-
NA	SL0016609	Water^	F091305-4	100	100	200	250	XC110705-6	-	-
NA	SL0016609	Water^	F091305-4	100	200	200	500	XC110705-7	-	-
NA	SL0016609	Water^	F091305-4	100	400	200	1000	XC110705-8	-	-
NA	SL0016609	Water^	F091305-7	100	100	200	250	XC110705-9	-	-

Vertical arrows in a column indicate identical values.

**This must be a unique number. Use this system: Extracted Calibration Soln ID #: XCMMDYY-0,1,2,3, etc.

Samples removed from refrigerator/ freezer # 34 Time: 9:00 AM

40 mL of each sample measured using a 50 mL graduated cylinder.

After measuring, samples returned to refrigerator/ freezer # 32 Time: 3:30 PM

Samples fortified: Initials/Date: AMB / 11/07/05

SPE clean-up: Initials/Date: EDD / 11/07/05

Final volume adjusted to 5 mL: Initials/Date: EDD / 11/07/05

Extracts placed in refrigerator # 32 Initials/Date: EDD / 11/07/05

Comments:

^This water has been filtered through a hypercarb filter

* Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Water.

THIS IS NOT A VALID COPY
THE ORIGINAL COPY IS IN THE
BY PL 11/18/05

STANDARD EXPIRATION DATE: 11/21/2005

Masslynx - Sample List

Sample List: C:\MASSLYNX\1008 APFQ PRO\SampleDB\111805A.SPL
 Printed: Fri Nov 18 12:55:12 2005

2008060712 16767

Page 1

Page Position: (1, 1)

Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/L)	Conc B	Conc C	Test ID	DF	MS Method
1	111805A-1001	---	---	XC110705-0, 0 ng/L standard	---	Blank	0	---	---	0	1	008 PFOA
2	111805A-1002	---	---	XC110705-1, 5 ng/L standard	---	Standard	5	---	---	0	1	008 PFOA
3	111805A-1003	---	---	XC110705-2, 10 ng/L standard	---	Standard	10	---	---	0	1	008 PFOA
4	111805A-1004	---	---	XC110705-3, 25 ng/L standard	---	Standard	25	---	---	0	1	008 PFOA
5	111805A-1005	---	---	XC110705-4, 50 ng/L standard	---	Standard	50	---	---	0	1	008 PFOA
6	111805A-1006	---	---	XC110705-5, 100 ng/L standard	---	Standard	100	---	---	0	1	008 PFOA
7	111805A-1007	---	---	XC110705-6, 250 ng/L standard	---	Standard	250	---	---	0	1	008 PFOA
8	111805A-1008	---	---	XC110705-7, 500 ng/L standard	---	Standard	500	---	---	0	1	008 PFOA
9	111805A-1009	---	---	XC110705-8, 1000 ng/L standard	---	Standard	1000	---	---	0	1	008 PFOA
10	111805A-1010	---	---	XC110705-9, 250 ng/L check standard	---	QC	250	---	---	0	1	008 PFOA
11	111805A-1011	---	---	Methanol Wash	---	Blank	---	---	---	0	1	008 PFOA
12	111805A-1012	---	---	Reagent Control	---	Blank	---	---	---	0	1	008 PFOA
13	111805A-1013	---	---	L6767-3	---	Analyte	---	---	---	0	1	008 PFOA
14	111805A-1014	---	---	Reagent Spk A, 50 ng/L	---	QC	50	---	---	0	1	008 PFOA
15	111805A-1015	---	---	Reagent Spk B, 500 ng/L	---	QC	500	---	---	0	1	008 PFOA
16	111805A-1016	---	---	L6767-1 Spk C, 5000 ng/L, DF=10	---	QC	5000	---	---	0	10	008 PFOA
17	111805A-1017	---	---	L6767-2 Spk D, 5000 ng/L, DF=10	---	QC	5000	---	---	0	10	008 PFOA
18	111805A-1018	---	---	Methanol Wash	---	Blank	---	---	---	0	1	008 PFOA
19	111805A-1019	---	---	XC110705-1, 5 ng/L standard	---	Standard	5	---	---	0	1	008 PFOA
20	111805A-1020	---	---	XC110705-2, 10 ng/L standard	---	Standard	10	---	---	0	1	008 PFOA
21	111805A-1021	---	---	XC110705-3, 25 ng/L standard	---	Standard	25	---	---	0	1	008 PFOA
22	111805A-1022	---	---	XC110705-4, 50 ng/L standard	---	Standard	50	---	---	0	1	008 PFOA
23	111805A-1023	---	---	L6767-1	---	Analyte	---	---	---	0	1	008 PFOA
24	111805A-1024	---	---	L6767-1 Rep	---	Analyte	---	---	---	0	1	008 PFOA
25	111805A-1025	---	---	L6767-2	---	Analyte	---	---	---	0	1	008 PFOA
26	111805A-1026	---	---	L6767-2 Rep	---	Analyte	---	---	---	0	1	008 PFOA
27	111805A-1027	---	---	XC110705-5, 100 ng/L standard	---	Standard	100	---	---	0	1	008 PFOA
28	111805A-1028	---	---	XC110705-6, 250 ng/L standard	---	Standard	250	---	---	0	1	008 PFOA
29	111805A-1029	---	---	XC110705-7, 500 ng/L standard	---	Standard	500	---	---	0	1	008 PFOA
30	111805A-1030	---	---	XC110705-8, 1000 ng/L standard	---	Standard	1000	---	---	0	1	008 PFOA

Masslynx - Sample List

Sample List: C:\MASSLYNX\008 APFO.PRO\SampleDB\111805A.SPL
Printed: Fri Nov 18 12:55:12 2005

Exogen Study No. 16767

Page 2

Page Position: (2, 1)

HPLC Method MS Tune File Inj Volume (µL)

1	water	PFOA Tune	15
2	water	PFOA Tune	15
3	water	PFOA Tune	15
4	water	PFOA Tune	15
5	water	PFOA Tune	15
6	water	PFOA Tune	15
7	water	PFOA Tune	15
8	water	PFOA Tune	15
9	water	PFOA Tune	15
10	water	PFOA Tune	15
11	water	PFOA Tune	15
12	water	PFOA Tune	15
13	water	PFOA Tune	15
14	water	PFOA Tune	15
15	water	PFOA Tune	15
16	water	PFOA Tune	15
17	water	PFOA Tune	15
18	water	PFOA Tune	15
19	water	PFOA Tune	15
20	water	PFOA Tune	15
21	water	PFOA Tune	15
22	water	PFOA Tune	15
23	water	PFOA Tune	15
24	water	PFOA Tune	15
25	water	PFOA Tune	15
26	water	PFOA Tune	15
27	water	PFOA Tune	15
28	water	PFOA Tune	15
29	water	PFOA Tune	15
30	water	PFOA Tune	15

0019

Scanning Method Report

Page 1

Method: C:\MASSLYNX\008 APFO.PRO\ACQUDB\008 PFOA
Last Modified: Wed Mar 20 15:07:50 2002

Printed: Fri Nov 18 13:17:55 2005

11/18/05

Solvent Delay (mins) : 0.00

Function : 1 MRM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 8.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
---------	--------	----------	--------------	------------------	------------

1	413.00	369.00	0.20	10	10
---	--------	--------	------	----	----

Method Report

Page 1

Method File: c:\masslynx\008 apfo.pro\acqddb\water
 Last Modified: Friday, November 18, 2005 13:18:43

Printed: Friday, November 18, 2005 13:18:48

HP1100 LC Pump Initial Conditions

Solvents

A%	60.0
B%	40.0
C%	0.0
D%	0.0

Flow (ml/min)	0.300
Stop Time (mins)	15.0
Min Pressure (bar)	0
Max Pressure (bar)	400
Oven Temperature Left(°C)	35.0
Oven Temperature Right(°C)	35.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 10 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	60.0	40.0	0.0	0.0	0.300	400
0.40	60.0	40.0	0.0	0.0	0.300	400
1.00	10.0	90.0	0.0	0.0	0.300	400
7.00	10.0	90.0	0.0	0.0	0.300	400
7.50	0.0	100.0	0.0	0.0	0.300	400
9.00	0.0	100.0	0.0	0.0	0.400	400
9.50	60.0	40.0	0.0	0.0	0.400	400
13.50	60.0	40.0	0.0	0.0	0.400	400
14.00	60.0	40.0	0.0	0.0	0.300	400
15.00	60.0	40.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 4 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	Off	Off	Off
0.10	Off	Off	On	Off	Off
8.00	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed	200.0
Eject Speed (µl/min)	200
Draw Position (mm)	0.10
Stop Time (mins)	15.00
Injection Volume (µl)	15.0
Vial Number	9

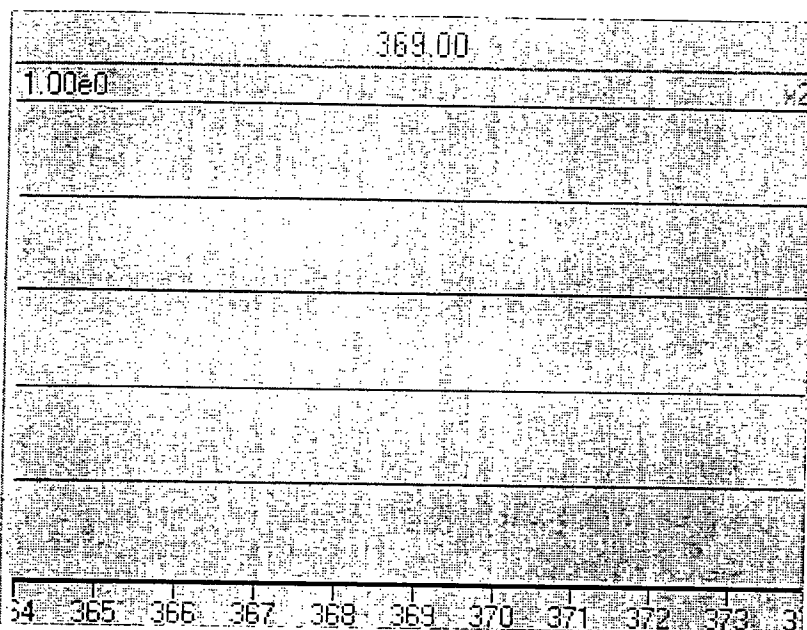
Tuning Method Report

Page 1

Method: C:\MASSLYNX\008 APFO.PRO\ACQUDB\PFOA TUNE

Printed: Fri Nov 18 13:20:17 2005

11/18/05



Dau 413.00

SOURCE (ESP-)	Set	Rdbk	Analyser	Set	Rdbk
Capillary	3.00	-2.93	LM Res 1	14.0	
Cone	10	-10	HM Res 1	14.0	
Hexapole 1	0.0		IEnergy 1	1.0	
Aperture 1	0.0		Entrance	-2	8
Hexapole 2	0.0		Collision	10	9
Source Block Temp.	100	101	Exit	2	11
Desolvation Temp.	300	300	LM Res 2	14.0	
			HM Res 2	14.0	
			IEnergy 2	2.0	
			Multiplier	700	-695

Pressures	Rdbk	Gas Flows	Rdbk
Analyser Vacuum	OFF	Cone Gas	84.9
Gas Cell	3.0e-3	Desolvation	682.4

In order to verify mass resolution, a calibration check is performed on the mass spectrometer using a NaI/Rb/Cs tuning solution. The tuning solution is infused onto the mass spec using an infusion pump set to a flow rate of 10 μ L/min. An MS scan from 80 to 1380 amu is performed on both MS1 and MS2. The calibration is considered acceptable if the following 11 peaks are found within ± 0.2 amu of their known masses on both MS1 and MS2: 84.9, 132.9, 172.8, 322.7, 472.6, 622.5, 772.4, 922.3, 1072.2, 1222.1 and 1372.0. If any of the masses are outside the ± 0.2 amu, then the instrument will be recalibrated and another calibration check performed.

The printouts from the calibration check can be found on the following 2 pages.

20
12/18/05

Calibration Check on MS using NaIRbCs Tuning Solution

CALCHK111605-1 1 (0.087) Cn (Cen,2, 80.00, Ht); Sm (SG, 2x0.60); Sb (1,40.00); Cm (1:16)

100- 132.8 172.7

363.1

292.8

232.7

472.6

772.4

532.6

322.7

442.8

682.6

832.5

582.5

882.4

732.4

982.4

1032.3

1182.2

1332.3

m/z

LC/MS/MS #7

16-Nov-2005 06:49:33

Scan ES+

1.32e8

AMB

11/10/05

EXPERIMENT STUDY NO. 66767

"THIS IS AUTO FILE"

THE ORIGINAL COPY FILE

BY

DATE 11/10/05

1072.3

1222.2

1372.1

0025

Calibration Check on MS2 using NaIRbCs Tuning Solution

CALCHK111605-2 15 (0.933) Cn (Cen, 2, 80.00, Ht); Sm (SG, 2x0.60); Sb (1, 40.00); Cm (1:15)

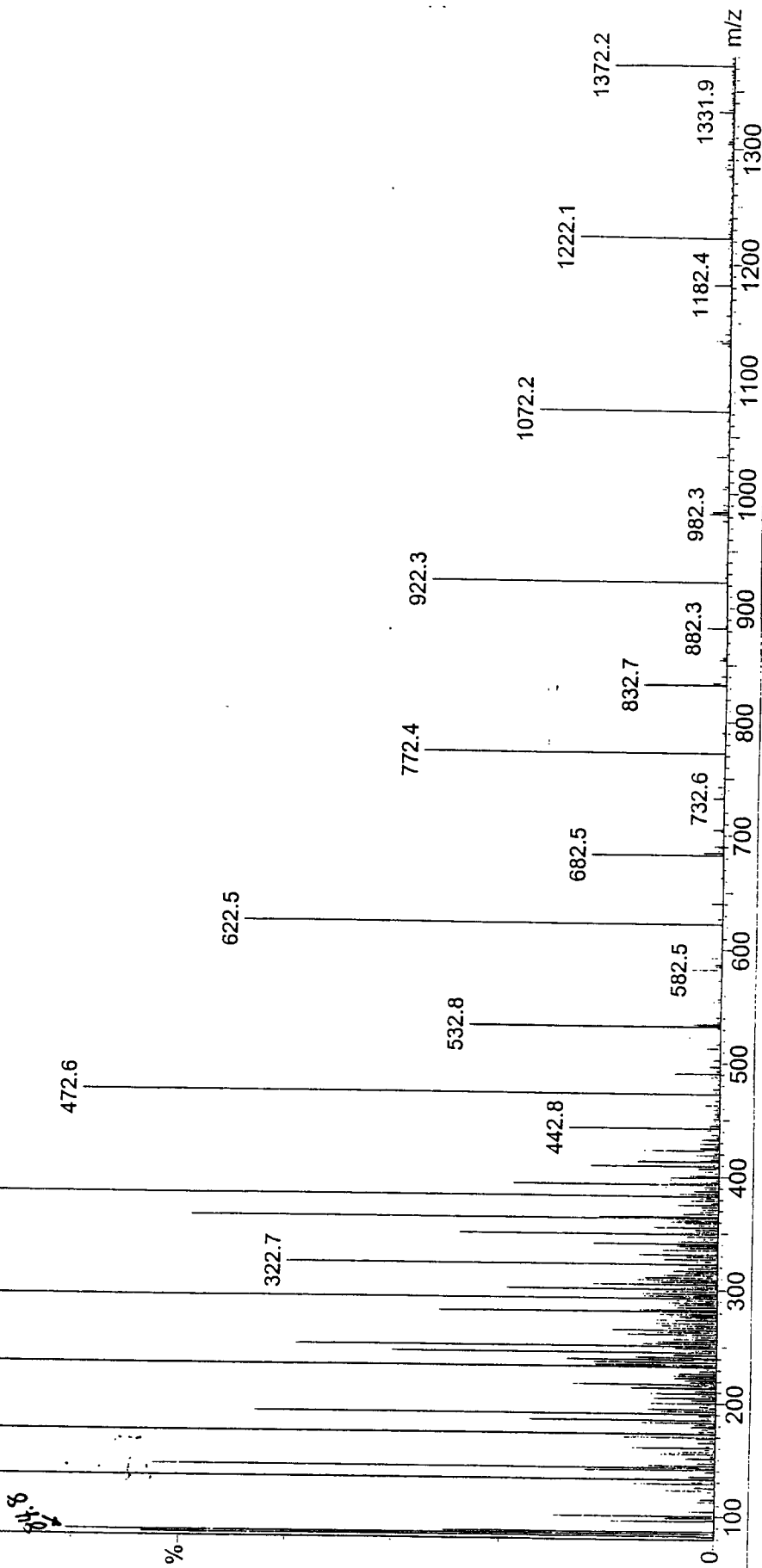
LC/MS/MS #7
16-Nov-2005 06:58:12
MS2 ES+
1.37e8

AMB
11/10/05

EXPERIMENT NO. 16767

"THIS IS AN EXACT COPY OF
THE ORIGINAL DOCUMENT"

BY pf DATE 11/10/05



Initial Calibration Curve

Oxygen Study No.: L6767
 Set No: 111805A
 Extraction Date: 11/18/05
 Analyzed on: 11/18/05

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first eight standards of the analytical run. The coefficient of determination (R Square) must be 0.985 or greater. If the set fails to meet this requirement, the run must be rejected and the samples reanalyzed.

Oxygen ID	Standard Concentration (ng/L)	Peak Area
XC110705-1	5	494
XC110705-2	10	826
XC110705-3	25	2065
XC110705-4	50	3584
XC110705-5	100	7752
XC110705-6	250	16331
XC110705-7	500	35308
XC110705-8	1000	69493

Coefficient of Determination: 0.999502113

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999751025
R Square	0.999502113
Adjusted R Square	0.999419132
Standard Error	584.264172
Observations	8

ANOVA

	df	SS	MS	F	Significance F
Regression	1	4111709682	4111709682	12044.92032	3.85766E-11
Residual	6	2048187.736	341364.6227		
Total	7	4113757870			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%	Lower 95.0%	Upper 95.0%
Intercept	167.9081974	257.1792017	0.652884045	0.538027981	-461.3870993	797.2034941	-461.3870993	797.2034941
X Variable 1	69.33491465	0.631756937	109.7493523	3.85766E-11	67.78905998	70.88076932	67.78905998	70.88076932

LOD and LOQ Determination

Oxygen Study No.: L6767
Set No: 111805A
Extraction Date: 11/18/05
Analyzed on: 11/18/05

The height of the first 5 ng/L standard of the analytical run is measured in centimeters and recorded. The height of the noise in a one minute range before the PFOA retention time in the reagent control is measured in centimeters and recorded. The noise height is then divided by the standard height. The result is converted to ng/L by multiplying by the concentration of the standard measured (5 ng/L). The LOD is calculated by taking 3 times the signal to noise ratio in ng/L. The LOQ is calculated by taking 5 times the LOD.

Oxygen ID	Concentration (ng/L)	Height (cm)
XC110705-1	5	15.1
Reagent Control	0	4.5

Signal to Noise Ratio = [height of the noise in the reagent control (cm) / height of the 5 ng/L standard(cm)] x 5 ng/mL

Signal to Noise Ratio (ng/L) = 1.490 ng/L
LOD = 4.47 ng/L
LOQ = 22.4 ng/L

Handwritten signature
01/06/06

XC110705-1, 5 ng/L standard

18-Nov-2005 13:50:58

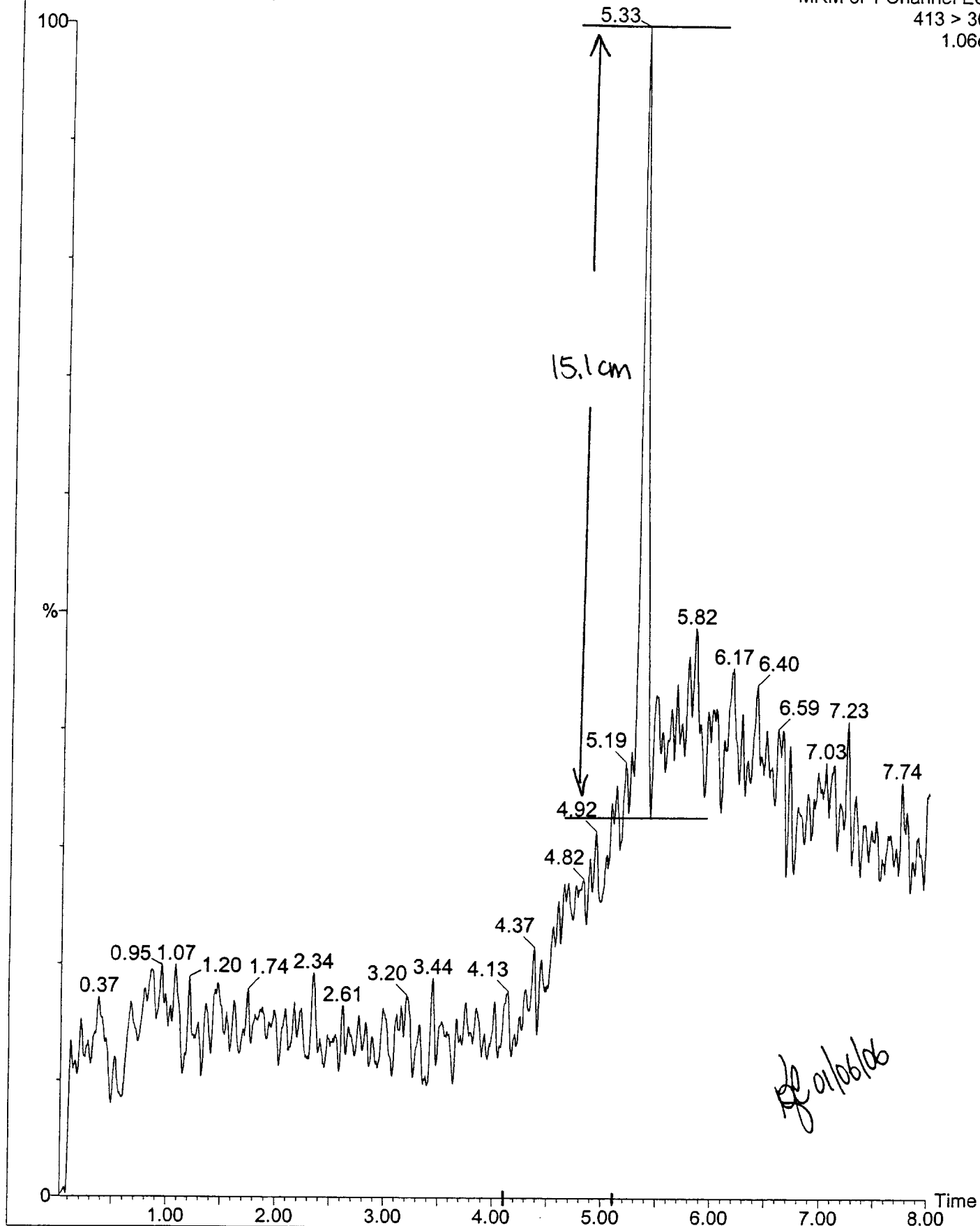
111805A-1002 Sm (Mn, 2x3)

LC/MS/MS #7

MRM of 1 Channel ES-

413 > 369

1.06e4



0028B

Reagent Control

18-Nov-2005 16:37:44

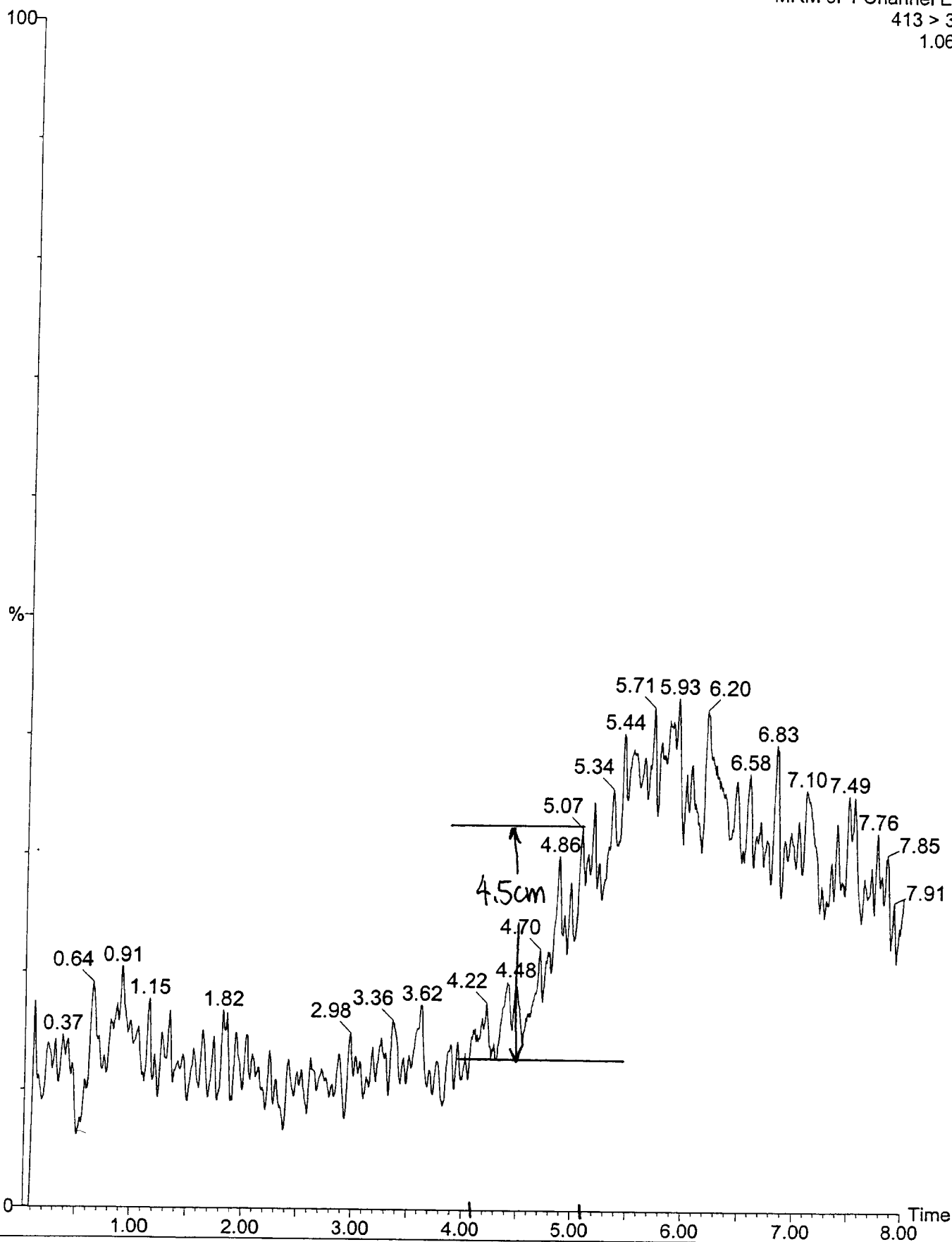
LC/MS/MS #7

MRM of 1 Channel ES-

413 > 369

1.06e4

111805A-1012 Sm (Mn, 2x3)



0028C

Quantify Calibration Report

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Page 1

Calibration: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\CurveDB\111805A

Last modified: Mon Nov 21 06:36:43 2005

Printed: Mon Nov 21 06:44:55 2005

11/21/05

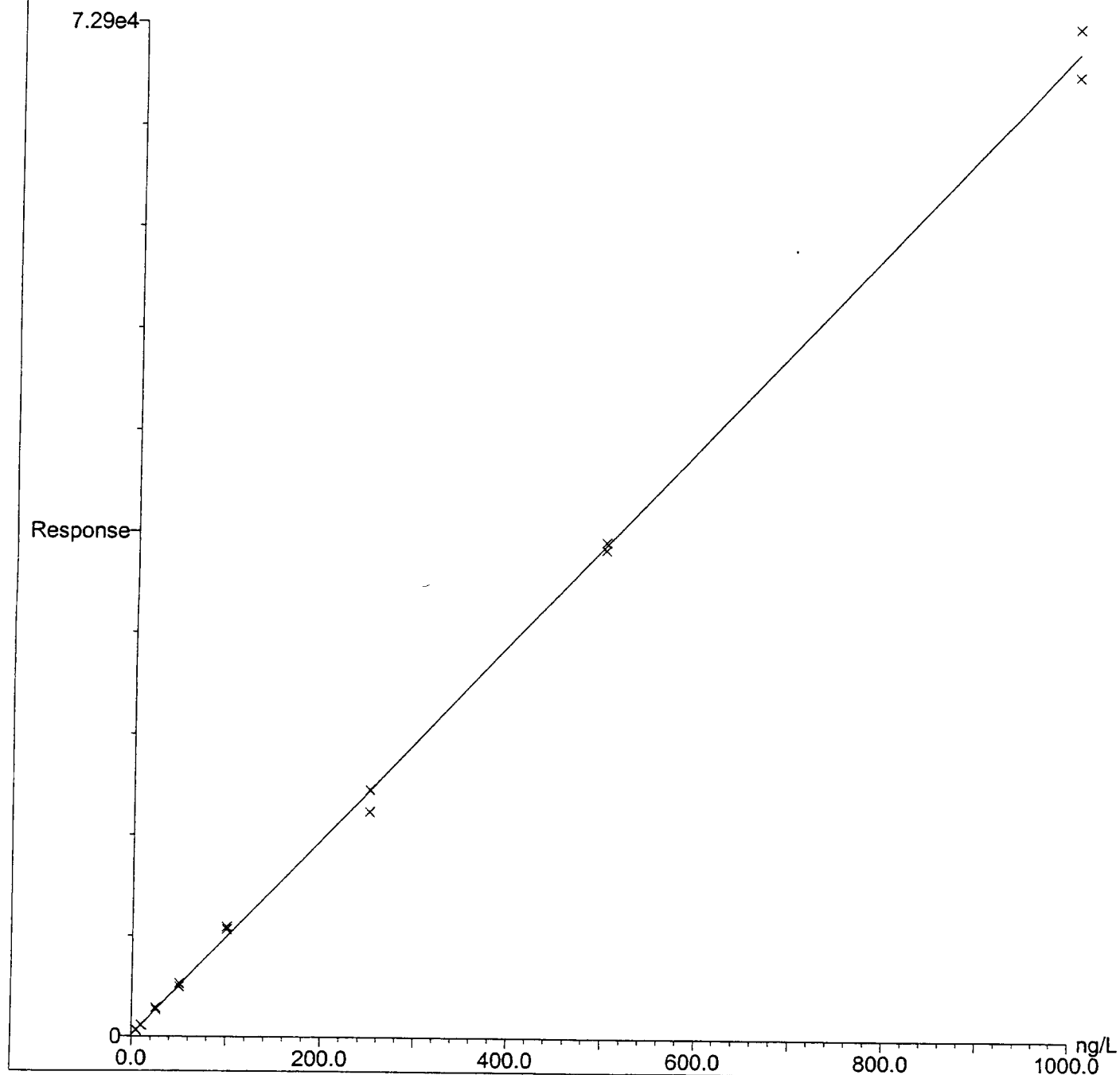
Compound 1 name: PFOA

Coefficient of Determination: 0.998929

Calibration curve: $70.9929 * x + 125.839$

Response type: External Std, Area

Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report

Page 1

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Initials RBDate 11/21/05Run# 111805A-1001 To 111805A-1030

Name: 111805A-1001

Text:

1: PFOA

XC110705-0, 0 ng/L standard

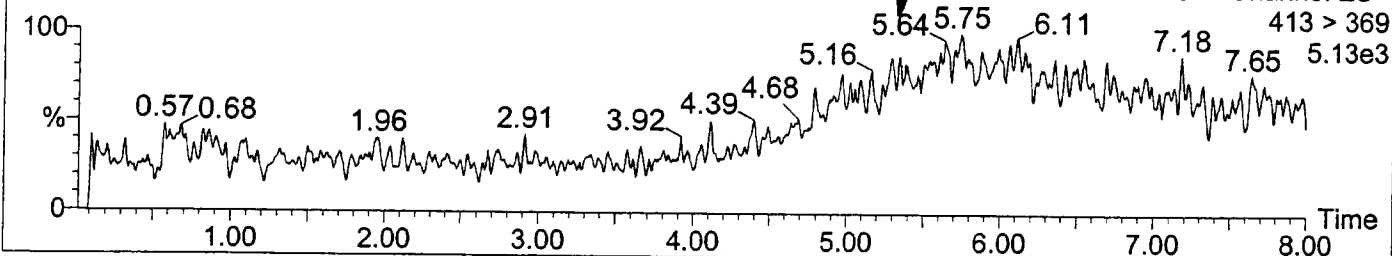
18-Nov-2005 13:34:17

LC/MS/MS #7

111805A-1001 Sm (Mn, 2x2)

MRM of 1 Channel ES-

413 > 369



Quantify Sample Report

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Page 2

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1002
Text:

1: PFOA

XC110705-1, 5 ng/L standard

18-Nov-2005 13:50:58

LC/MS/MS #7

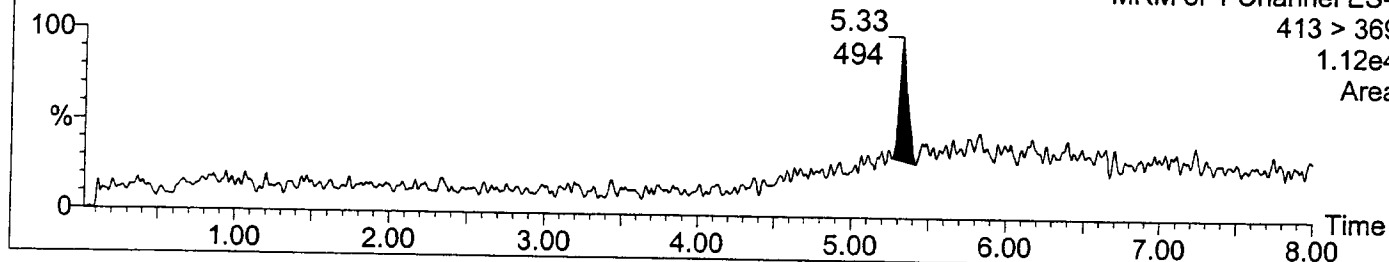
MRM of 1 Channel ES-

413 > 369

1.12e4

Area

111805A-1002 Sm (Mn, 2x2)



Quantify Sample Report

Page 3

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1003
Text:

1: PFOA

XC110705-2, 10 ng/L standard

18-Nov-2005 14:07:36

LC/MS/MS #7

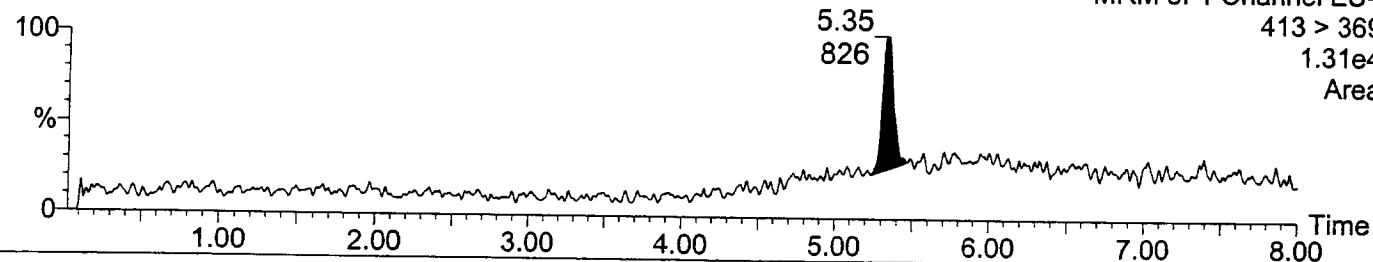
MRM of 1 Channel ES-

413 > 369

1.31e4

Area

111805A-1003 Sm (Mn, 2x2)



Quantify Sample Report

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Page 4

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1004

Text:

1: PFOA

XC110705-3, 25 ng/L standard

18-Nov-2005 14:24:12

LC/MS/MS #7

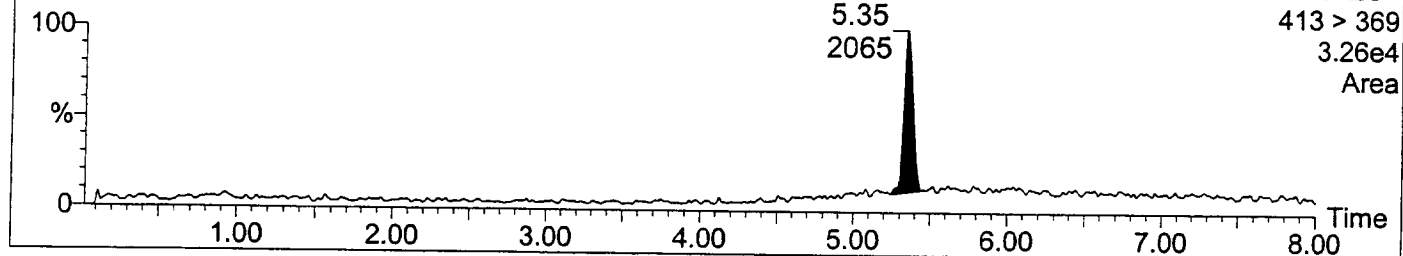
111805A-1004 Sm (Mn, 2x2)

MRM of 1 Channel ES-

413 > 369

3.26e4

Area



Quantify Sample Report

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Page 5

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1005

Text:

1: PFOA

XC110705-4, 50 ng/L standard

18-Nov-2005 14:40:50

LC/MS/MS #7

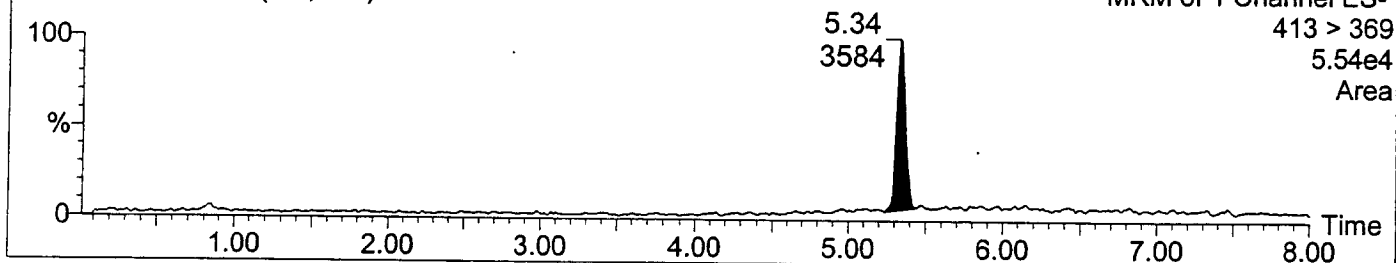
MRM of 1 Channel ES-

413 > 369

5.54e4

Area

111805A-1005 Sm (Mn, 2x2)



Quantify Sample Report

Page 6

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1006

Text:

1: PFOA

XC110705-5, 100 ng/L standard

18-Nov-2005 14:57:29

LC/MS/MS #7

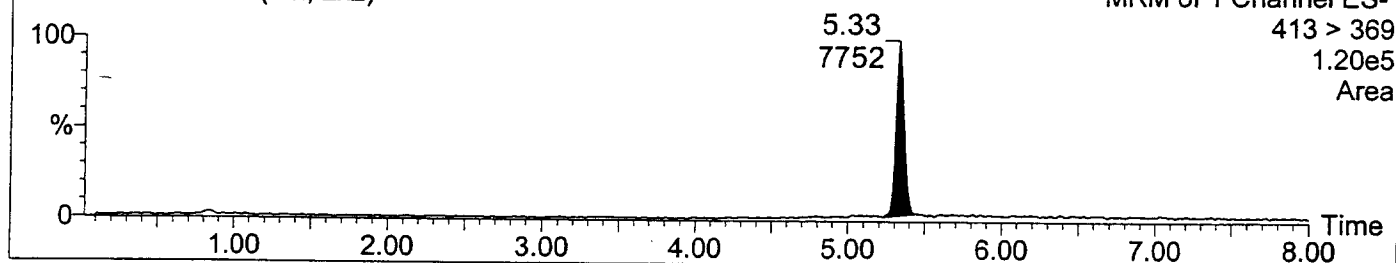
MRM of 1 Channel ES-

413 > 369

1.20e5

Area

111805A-1006 Sm (Mn, 2x2)



Quantify Sample Report

Page 7

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1007

Text:

1: PFOA

XC110705-6, 250 ng/L standard

18-Nov-2005 15:14:08

LC/MS/MS #7

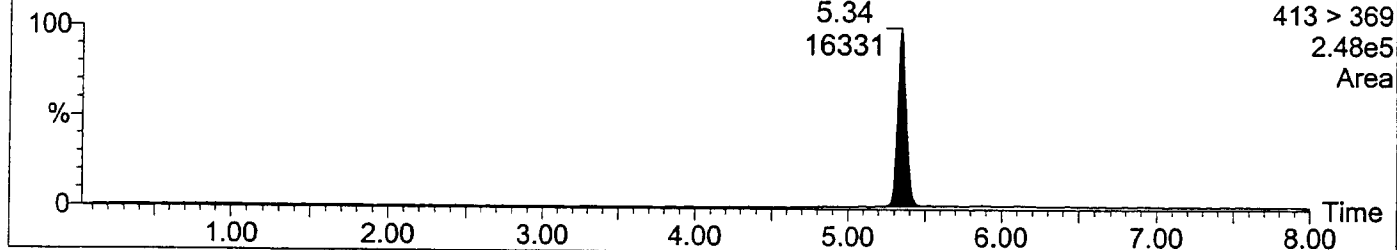
111805A-1007 Sm (Mn, 2x2)

MRM of 1 Channel ES-

413 > 369

2.48e5

Area



Quantify Sample Report

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Page 8

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1008

Text:

1: PFOA

XC110705-7, 500 ng/L standard

18-Nov-2005 15:30:56

LC/MS/MS #7

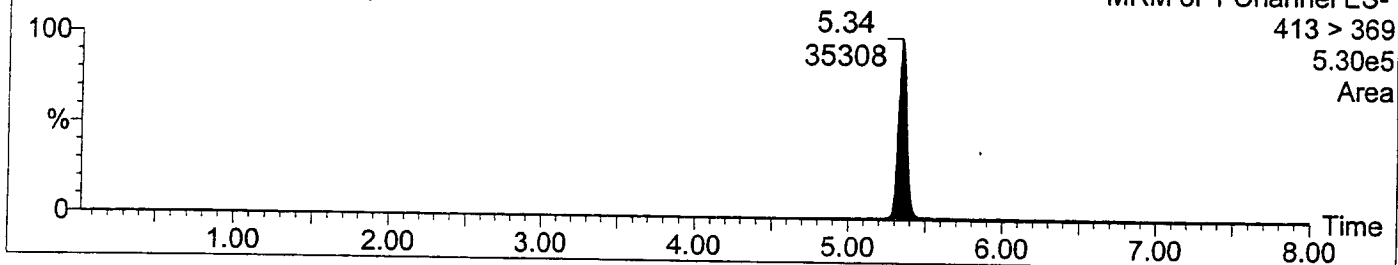
MRM of 1 Channel ES-

413 > 369

5.30e5

Area

111805A-1008 Sm (Mn, 2x2)



Quantify Sample Report

Page 9

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1009

Text:

1: PFOA

XC110705-8, 1000 ng/L standard

18-Nov-2005 15:47:40

LC/MS/MS #7

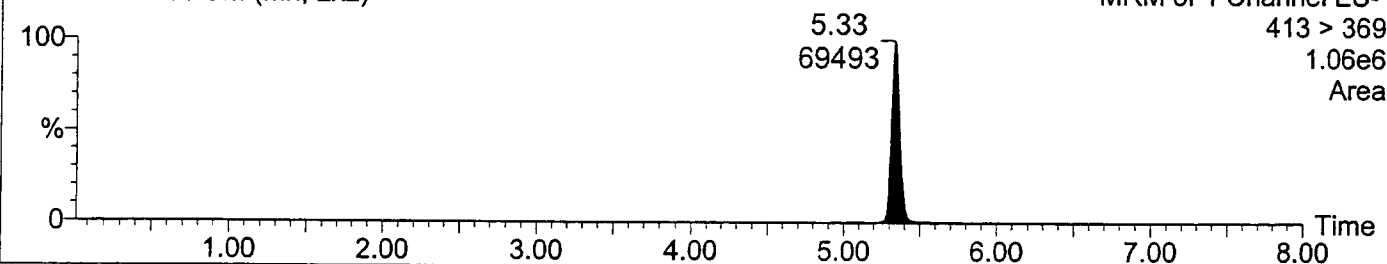
MRM of 1 Channel ES-

413 > 369

1.06e6

Area

111805A-1009 Sm (Mn, 2x2)



Quantify Sample Report

Page 10

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1010

Text:

1: PFOA

XC110705-9, 250 ng/L check standard

18-Nov-2005 16:04:21

LC/MS/MS #7

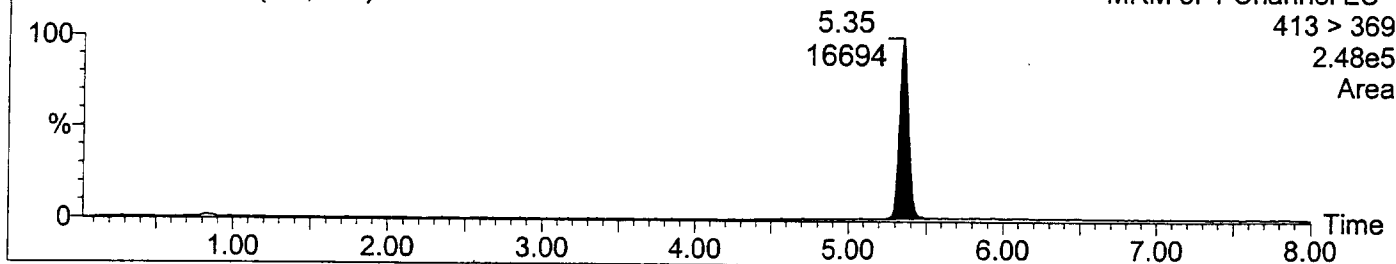
MRM of 1 Channel ES-

413 > 369

2.48e5

Area

111805A-1010 Sm (Mn, 2x2)



Quantify Sample Report

Page 11

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

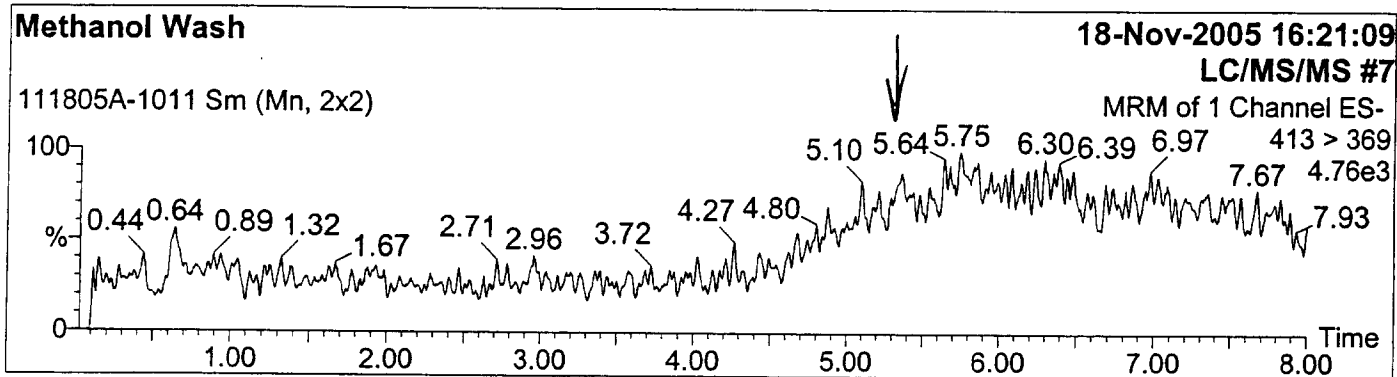
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1011

Text:

1: PFOA



Quantify Sample Report

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Page 12

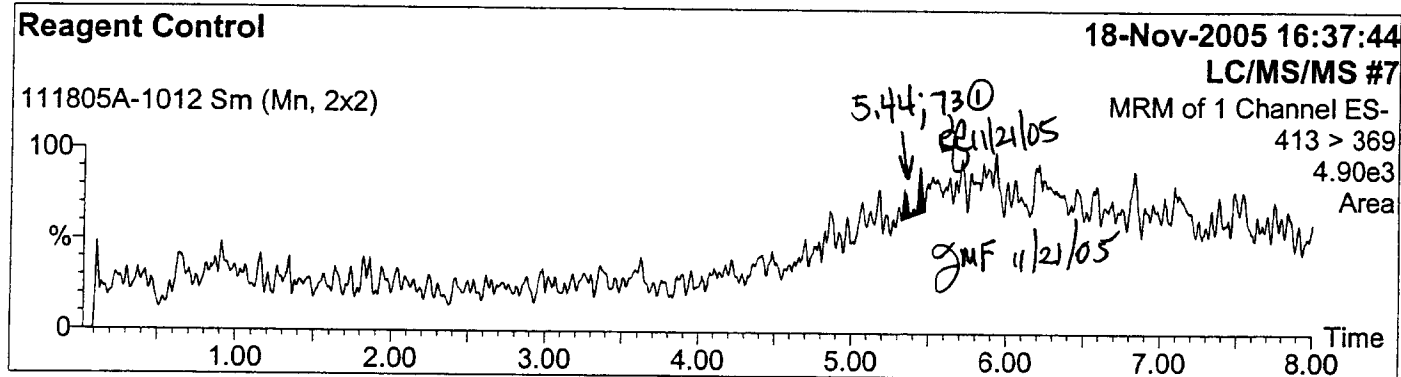
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Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1012

Text:

1: PFOA



Values were transcribed from
Masslynx software. JMF 11/21/05

Quantify Sample Report

Page 13

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

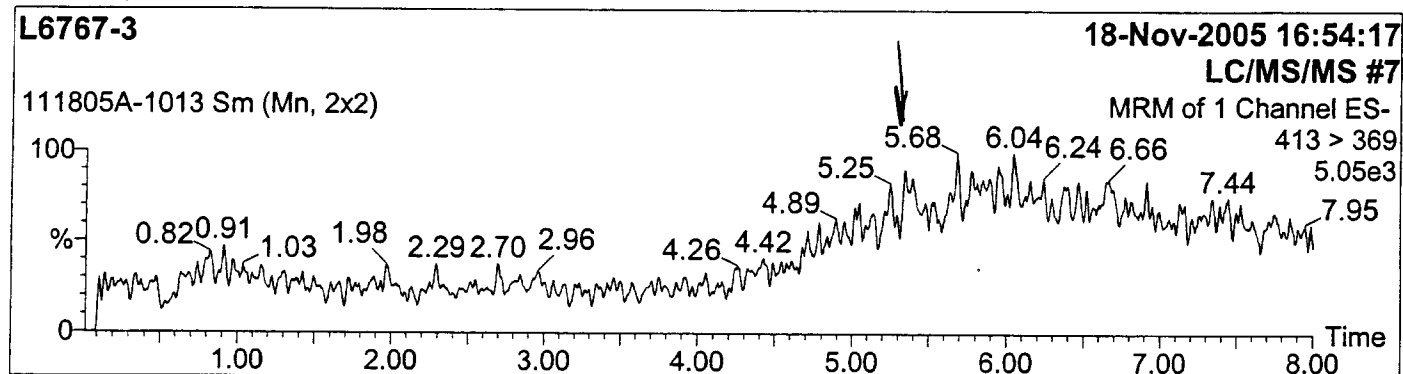
Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1013

Text:

1: PFOA



Quantify Sample Report

Page 14

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

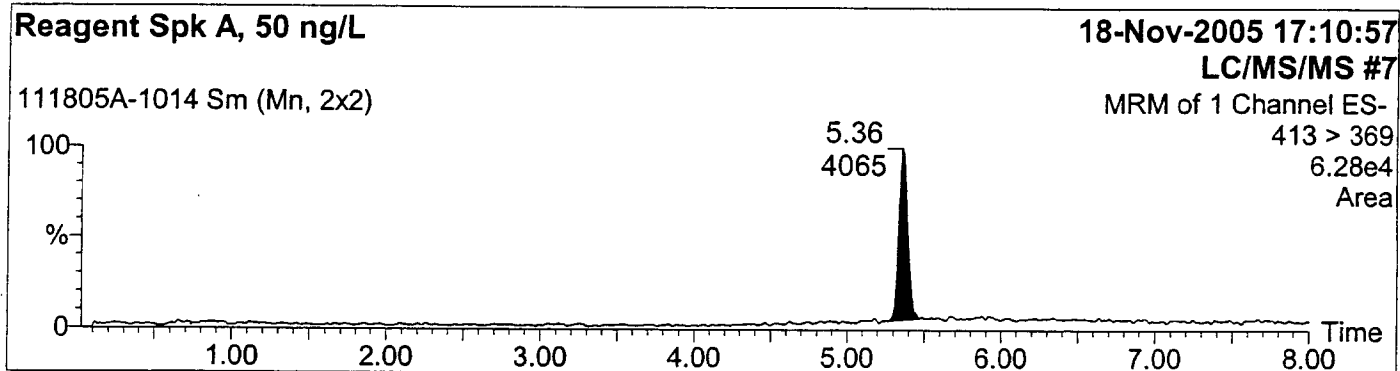
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1014

Text:

1: PFOA



Quantify Sample Report

Page 15

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

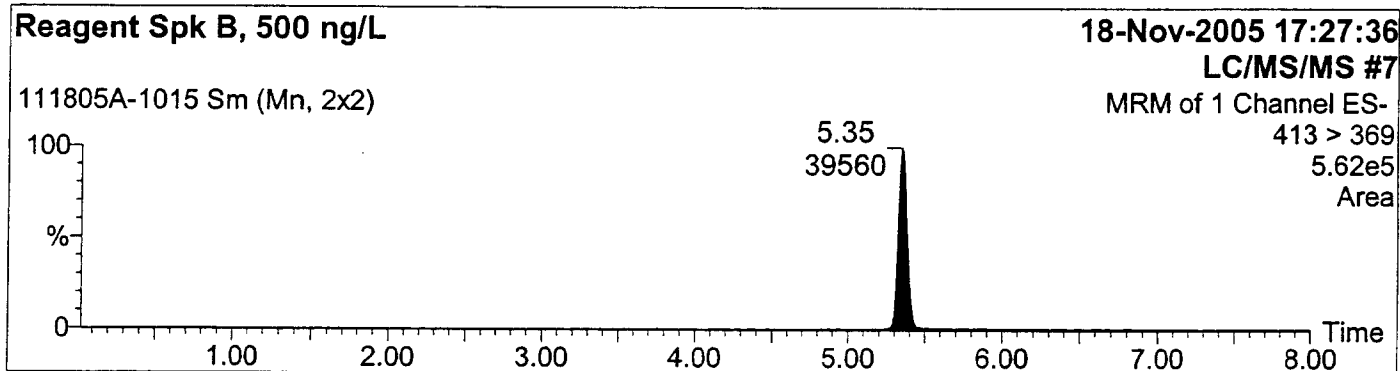
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1015

Text:

1: PFOA



Quantify Sample Report

Page 16

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1016

Text:

1: PFOA

L6767-1 Spk C, 5000 ng/L, DF=10

18-Nov-2005 17:44:19

LC/MS/MS #7

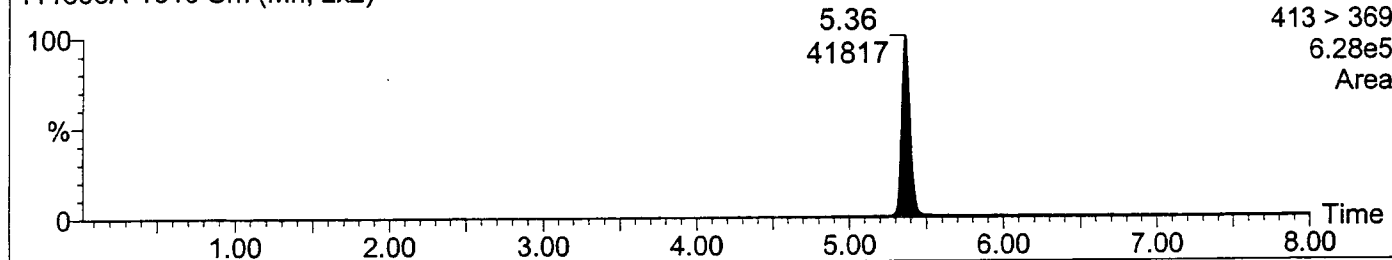
MRM of 1 Channel ES-

413 > 369

6.28e5

Area

111805A-1016 Sm (Mn, 2x2)



Quantify Sample Report

Page 17

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1017

Text:

1: PFOA

L6767-2 Spk D, 5000 ng/L, DF=10

18-Nov-2005 18:01:11

LC/MS/MS #7

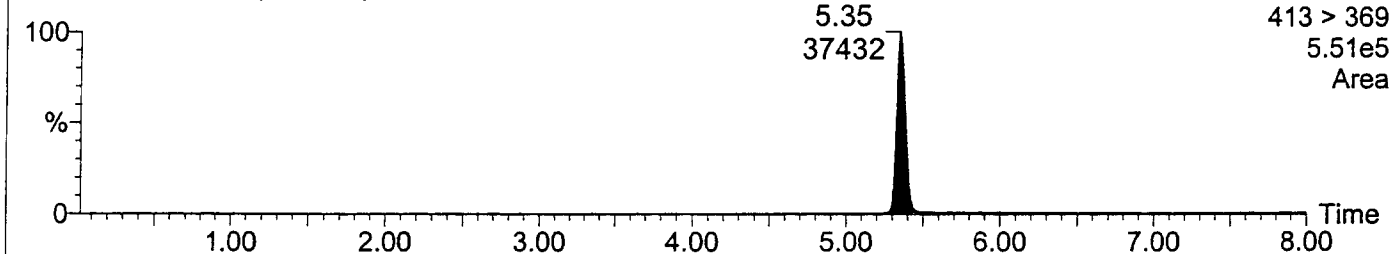
MRM of 1 Channel ES-

413 > 369

5.51e5

Area

111805A-1017 Sm (Mn, 2x2)



Quantify Sample Report

Page 18

Study No.: L6767, Set No.: 111805A, Ext.Date: 11/18/05, Analyst: K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1018

Text:

1: PFOA

Methanol Wash

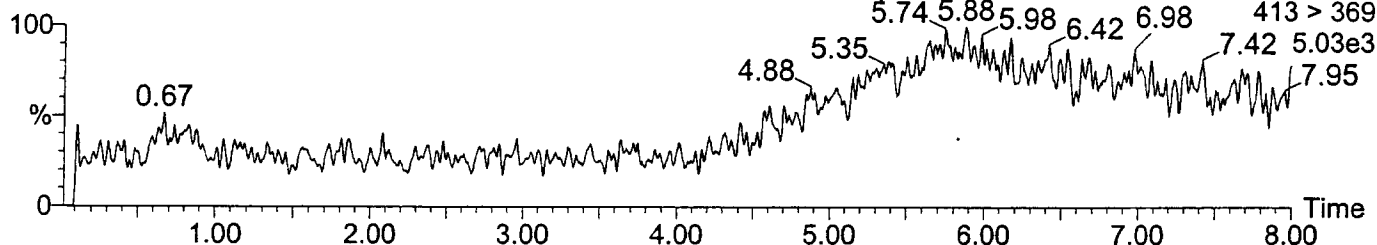
18-Nov-2005 18:17:55

LC/MS/MS #7

111805A-1018 Sm (Mn, 2x2)

MRM of 1 Channel ES-

413 > 369



Quantify Sample Report

Page 19

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1019

Text:

1: PFOA

XC110705-1, 5 ng/L standard

18-Nov-2005 18:34:42

LC/MS/MS #7

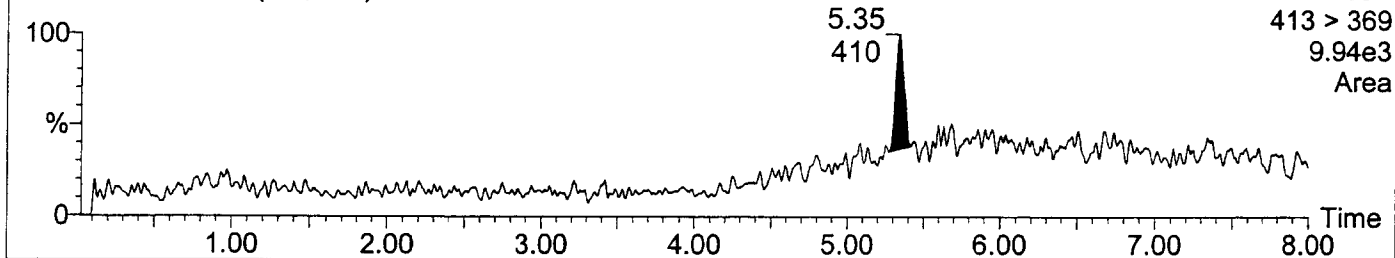
MRM of 1 Channel ES-

413 > 369

9.94e3

Area

111805A-1019 Sm (Mn, 2x2)



Quantify Sample Report

Page 20

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1020

Text:

1: PFOA

XC110705-2, 10 ng/L standard

18-Nov-2005 18:51:21

LC/MS/MS #7

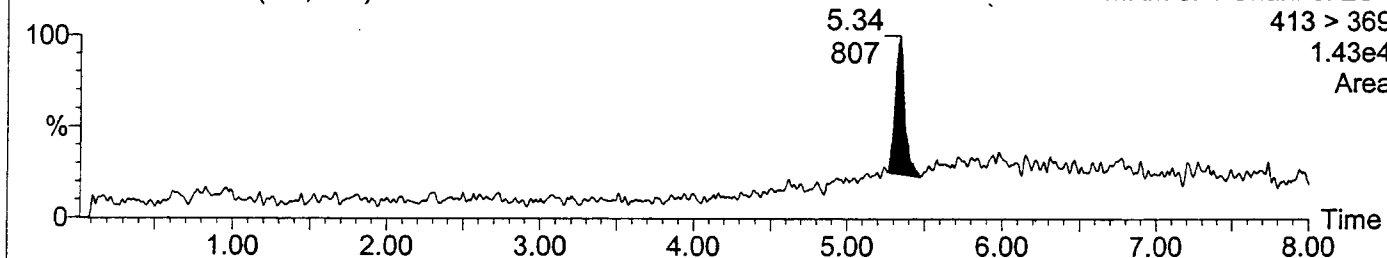
MRM of 1 Channel ES-

413 > 369

1.43e4

Area

111805A-1020 Sm (Mn, 2x2)



Quantify Sample Report

Page 21

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1021

Text:

1: PFOA

XC110705-3, 25 ng/L standard

18-Nov-2005 19:08:04

LC/MS/MS #7

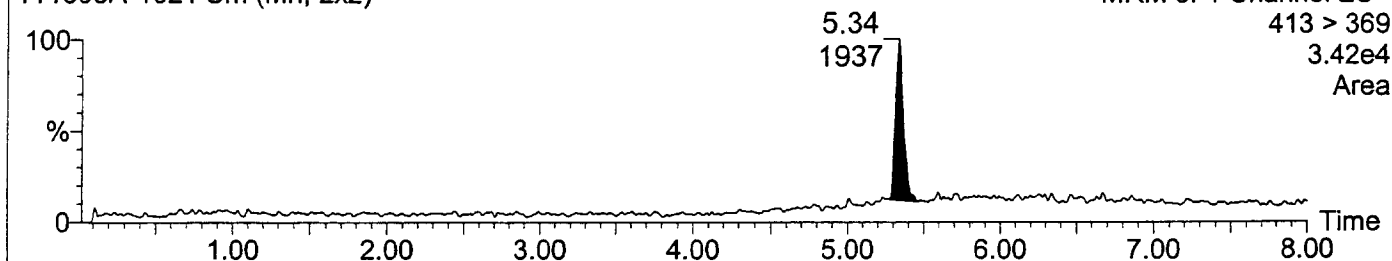
MRM of 1 Channel ES-

413 > 369

3.42e4

Area

111805A-1021 Sm (Mn, 2x2)



Quantify Sample Report

Page 22

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1022

Text:

1: PFOA

XC110705-4, 50 ng/L standard

18-Nov-2005 19:24:47

LC/MS/MS #7

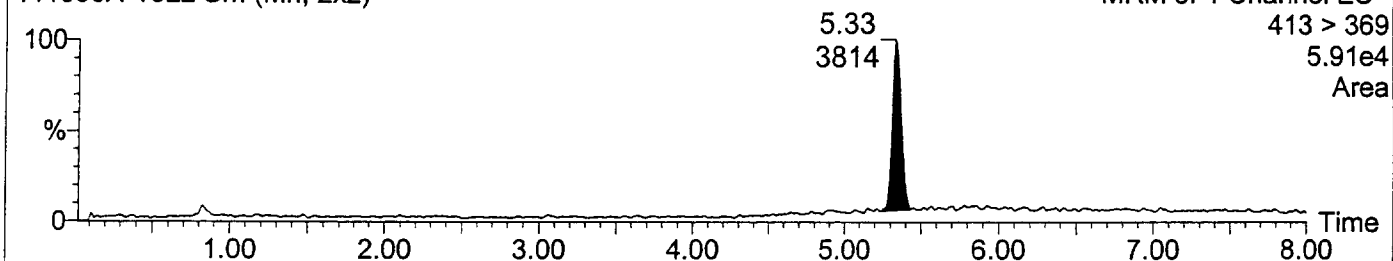
MRM of 1 Channel ES-

413 > 369

5.91e4

Area

111805A-1022 Sm (Mn, 2x2)



Quantify Sample Report

Page 23

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

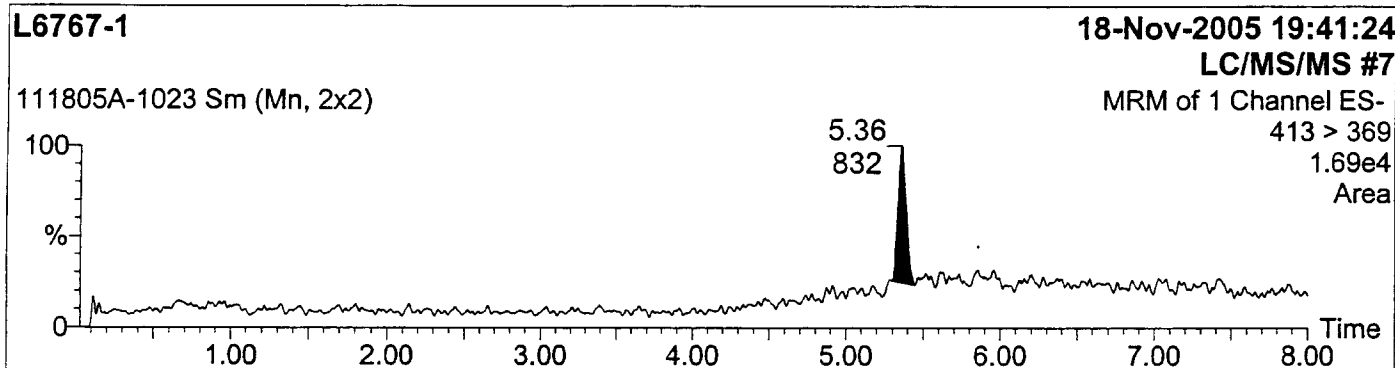
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1023

Text:

1: PFOA



Quantify Sample Report

Page 24

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

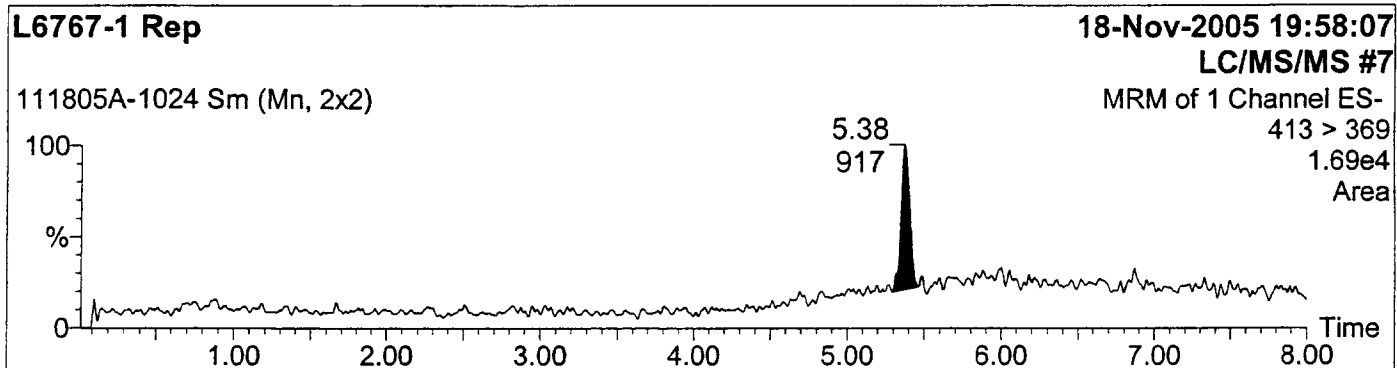
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1024

Text:

1: PFOA



Quantify Sample Report

Page 25

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

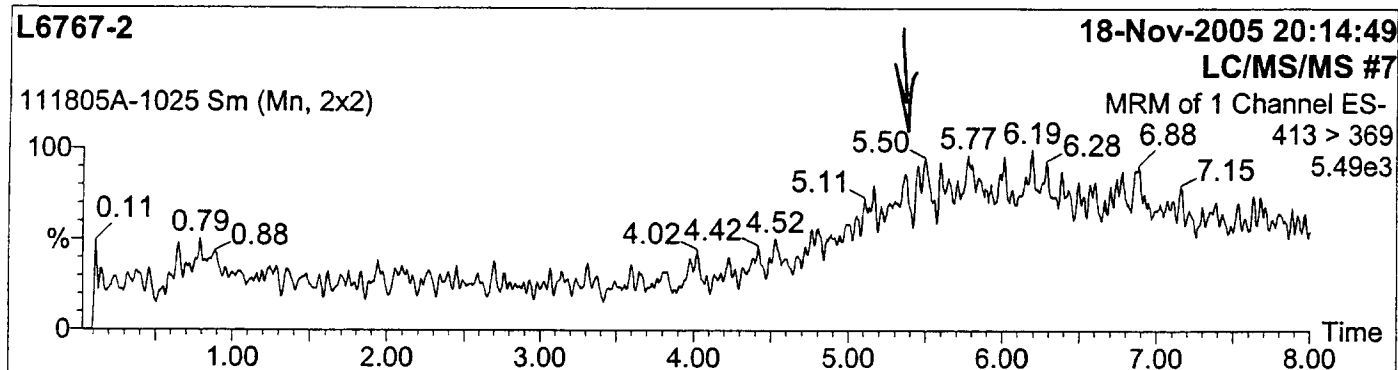
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1025

Text:

1: PFOA



Quantify Sample Report

Page 26

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

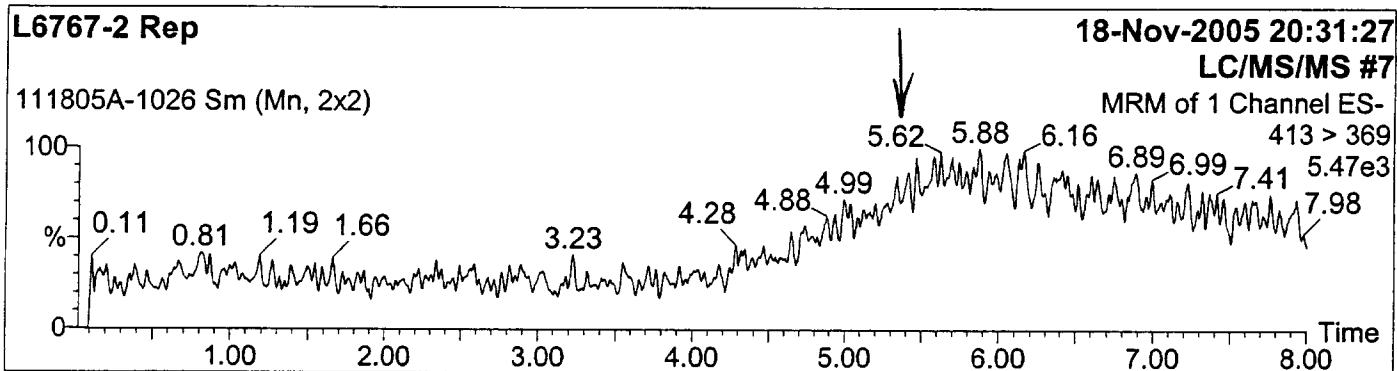
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1026

Text:

1: PFOA



Quantify Sample Report

Page 27

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A

Last modified: Mon Nov 21 06:36:20 2005

Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA

Last modified: Wed Dec 01 06:25:13 2004

Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1027

Text:

1: PFOA

XC110705-5, 100 ng/L standard

18-Nov-2005 20:48:15

LC/MS/MS #7

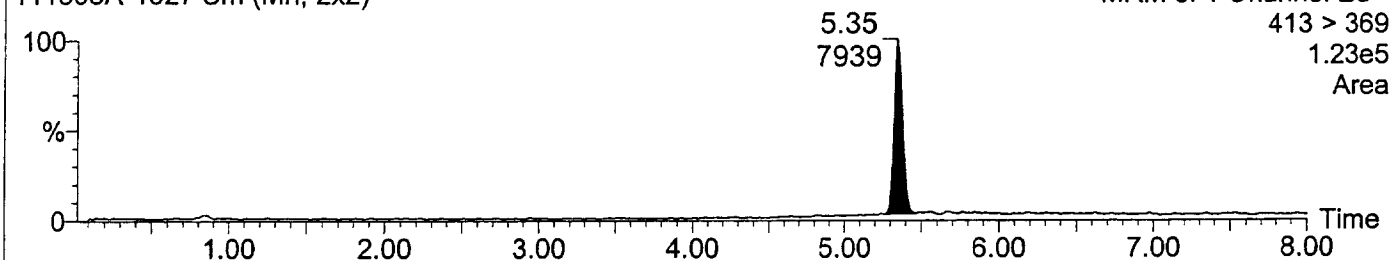
MRM of 1 Channel ES-

413 > 369

1.23e5

Area

111805A-1027 Sm (Mn, 2x2)



Quantify Sample Report

Page 28

Study No.: L6767, Set No.: 111805A, Ext. Date: 11/18/05, Analyst: K. Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1028

Text:

1: PFOA

XC110705-6, 250 ng/L standard

18-Nov-2005 21:04:52

LC/MS/MS #7

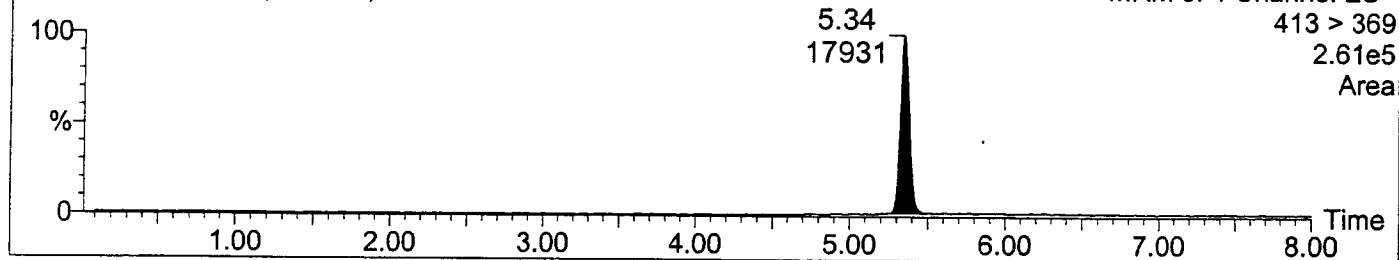
111805A-1028 Sm (Mn, 2x2)

MRM of 1 Channel ES-

413 > 369

2.61e5

Area



Quantify Sample Report

Page 29

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1029

Text:

1: PFOA

XC110705-7, 500 ng/L standard

18-Nov-2005 21:21:39

LC/MS/MS #7

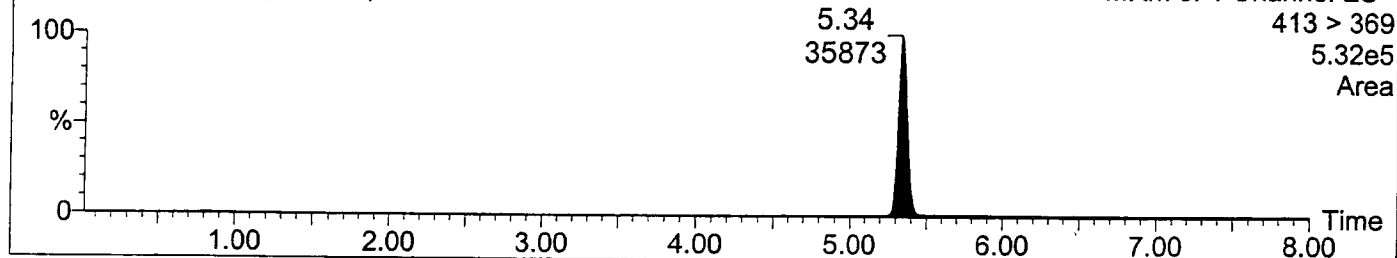
MRM of 1 Channel ES-

413 > 369

5.32e5

Area

111805A-1029 Sm (Mn, 2x2)



Quantify Sample Report

Page 30

Study No.:L6767, Set No.:111805A, Ext.Date:11/18/05, Analyst:K.Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\SampleDB\111805A
Last modified: Mon Nov 21 06:36:20 2005
Method: P:\Data\LCMSMS7\Masslynx\008 APFO.PRO\MethDB\112204 PFOA
Last modified: Wed Dec 01 06:25:13 2004
Job Code:

Printed: Mon Nov 21 06:44:55 2005

Name: 111805A-1030

Text:

1: PFOA

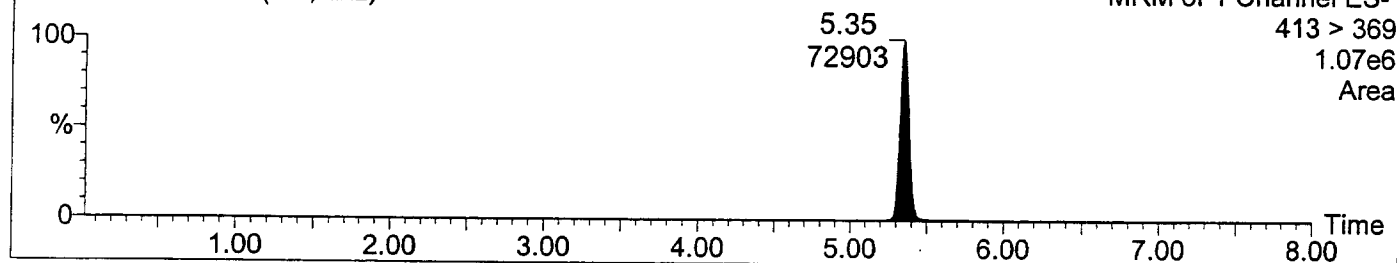
XC110705-8, 1000 ng/L standard

18-Nov-2005 21:38:22

LC/MS/MS #7

111805A-1030 Sm (Mn, 2x2)

MRM of 1 Channel ES-
413 > 369
1.07e6
Area



1

1

The following pages are exact copies of information for the standards used in this study.

Pentadecafluorooctanoic acid (Inventory #: SP3800), obtained from Aldrich (lot # 23116HB) was used to make a stock solution (ID # S061605-1), which in turn, was used to make intermediate solutions (ID#'s F091305-1, F091305-2, F091305-3, F091305-4 and F091305-5). These intermediate solutions are used to prepare extracted calibration standards, laboratory control samples, and matrix spike samples.

Pentadecafluorooctanoic acid (Inventory #: SP5444), obtained from Oakwood Products Inc., Fluorochem USA (lot # 203) was used to make a stock solution (ID # S040505-1), which in turn, was used to make intermediate solutions (ID#'s F091305-6 and F091305-7). These intermediate solutions are used to prepare the check standard, which is designed as an independent source verification of the calibration standards.

The information on the following pages provides the exact sources, lot numbers, shipping information, expiration dates and storage conditions of the neat standards. Also included are the preparation of stock standard and fortification solution forms. These pages show the exact details of the preparation of each of the stock and fortification solutions, as well as their expiration dates and storage conditions.

2
12/12/05



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

Standards - Primary

SP0003800 > 3845

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

Company Name:
Contact:
Phone #:
Notes:

Date Received: 12/08/2003
Expires: 12/8/2005 12:00:00AM
Special: No significant hazard
Flammable: Non-combustible (will not burn)
Health: Slightly toxic
Reactive: No significant hazard
Gross: 43.03219
Gross Units: g
Net Balance: 4.24186
Net Units: g

Aliquot - SP0003800

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

THIS IS AN EXACT COPY OF
THE ORIGINAL DOCUMENT

0061



00000000 > 0045

Description: Pentadecafluorooctanoic acid

Type: Routine

Sponsor Contact:

Matrix: Solids (Gravimetric)

Catalog #: 171468

Lot #: 23116HB

CAS #: 335-67-1

Solvent: NONE

Removed: False

Condition Rec'd: Room Temperature

Condition Stored: Room Temperature

Current Location: B1-105

Vendor: SIGMA-ALDRICH, INC.

Company Name:

Contact:

Phone #:

Notes:

Date Received: 12/08/2003

Expires: 12/8/2005 12:00:00AM

Special: No significant hazard

Flammable: ; Non-combustible (will not burn)

Health: Slightly toxic

Reactive: No significant hazard

Gross: 43.03219

Gross Units: g

Net Balance: 4.24186

Net Units: g

Aliquot - SP0003800, cont'd

Addition	Component	Withdrawal	Units	Component	Purpose	Computer	Created On	Created By
00062		0.01024	g	Balance 14	PREPARE		2/22/2005 9:06:53AM	Connolly, Paul
		0.10242	g	Balance 17	OTHER	BALANCE8	5/17/2005 10:55:17AM	Edwards, Chrissy
		0.10242	g	Balance 17	ANALYSIS	BALANCE8	6/16/2005 8:18:43AM	Sheehan, Amy
		0.10242	g	Balance 17	ANALYSIS	BALANCE8	6/20/2005 3:09:46PM	Kravets, Brittany
			Notes:	Notes: Prepare stock for P760				
			Notes:	Notes: Fluorochemicals				
			Notes:	Notes: Stocks for fluorochemicals.				

Characterization - SP0003800

Purity
97.64 %

Date Characterized
12/08/2003

Date Expires
12/08/2005

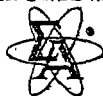
Characterized By
Sigma-Aldrich

Created By
Risha, Karen

12/10/2005 10:28 AM

11/21/05





SIGMA-ALDRICH

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

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

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

PAGE 1 of 1 DELIVERY 514710282

DATE	SOLD TO ACCT	SOLD TO NAME	PURCHASE ORDER NUMBER	REFERENCE #
12/05/2003	49435591	EXYGEN RESEARCH	4031KR10	9270247
ROUTE		PERSON TO CONTACT	PHONE NUMBER	
AIRBORNE GROUND FCA SHIPPING POINT		KIM DOOLITTLE	5142318032	
STOCK NO	LOT NO	ORDERED	SHIPPED	BACKORD
171468-5G	23116HB	1	1	0
				PENTADECYLFLUOROOCTANOIC ACID, 96%
				CNTRY OF OR: US
Reserve your FREE copy of the Best Sellers in Science. The 2004-2005 Sigma General Catalog will be available soon, featuring the world's largest selection of research products. Get a FREE Metabolic Pathways Chart when you reserve your copy now. www.sigma-aldrich.com/genca1				

The
SIGMA-ALDRICH
Family

SIGMA
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Reagents for Life
Science Research

Fluka
Specialty Chemicals
and Analytical
Reagents for Research

ALDRICH
Organics and
Inorganics for
Chemical Synthesis

Reagent
Laboratory Chemicals
and Reagents for
Research and Analysis

SUPELCO
Chromatography
Products for Analysis
and Purification

ALL SALES ARE EXPRESSLY LIMITED TO AND CONDITIONED UPON THE TERMS AND CONDITIONS APPEARING ON THE FRONT AND BACK OF THIS FORM.
SIGMA-ALDRICH BRAND PRODUCTS ARE SOLD THROUGH SIGMA-ALDRICH, INC.

0063

THIS IS AN INVENTORY OF
THE SIGMA-ALDRICH
FAMILY
11/21/05

1 OF 1
TR v3.5.00 CL488 04/82

0064

SP3800



SIGMA-ALDRICH

Certificate of Analysis

Product Name	Pentadecafluorooctanoic acid
Product Number	17,146-8
CAS Number	335-67-1
Molecular Formula	$C_8H_{15}O_2$
Molecular Weight	414.1

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

Ronnie J Martin, Supervisor
Quality Control

11/21/05

0065



3058 Research Drive
State College, PA 16801

SP 3800

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

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

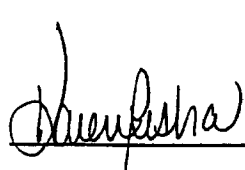
STUDY TELEPHONE/COMMUNICATIONS LOG

Person: Ken Originator: Exygen x
Company: Aldrich Sponsor _____

Date: 12/08/03
Time: 1022

Notes:

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

Signature: Date: 12/08/03

0066

August 7, 2001/1



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State College, PA 16801

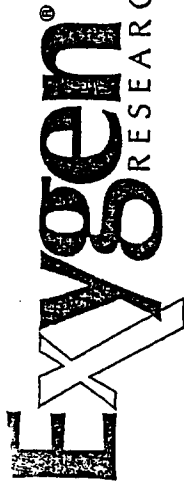
Phone: 814-272-1039
Fax: 814-231-1580

PREPARATION OF STOCK STANDARD SOLUTION

Project		
Oxygen Study / Project Number: <u>P418</u>		Customer Project Number: <u>NA</u>
Primary Standard		
Compound Name: <u>Pentadecafluorooctanoic Acid (PFOA)</u>		Oxygen Inventory ID: <u>SP0003800</u>
Purity: <u>97.64 %</u>		Primary Standard Expiration Date: <u>12/08/05</u>
Stock Solution Preparation		
Net Wt. of Compound: <u>0.10242 g</u>		Net Wt. Adjusted for Purity: <u>0.10000 g</u>
Final Volume: <u>100 mL</u>		Stock Solution Concentration: <u>1000 µg/mL</u>
Solvent Oxygen Inventory ID: <u>REDD19713</u>		Solvent Name: <u>Methanol</u>
Stock Solution ID: <u>(SMMDDYY-1,2,3, etc.) S061605-1</u>		Stock Solution Expiration Date: <u>12/08/05</u>
Storage Condition and Location		
Freezer ID: <u>-</u>	Refrigerator ID: <u>33</u>	Room Temperature: <u>-</u>
Walkin Freezer ID: <u>-</u>	Walkin Cooler ID: <u>-</u>	Storage Cabinet: <u>-</u>
<i>(Enter the Instrument or Room ID under the appropriate condition)</i>		
Remarks		
 11/21/05		

Signature: Amy Shukan

Date: 06/16/05



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Standards - Primary

SP0005444 > 5480

Description: Pentadecafluorooctanoic acid

Type: Routine

Sponsor Contact:

Matrix: Solids (Gravimetric)

Catalog #: 001319

Lot #: 203

CAS #: 335-67-1

Solvent: NONE

Removed: False

Condition Rec'd: Room Temperature

Condition Stored: Refrigerated

Current Location: B1-105 / IN0000678 - Walkin Cooler 9 / D0002222 - Walkin Cooler 9

Vendor: OAKWOOD PRODUCTS, INC.

Company Name:

Contact:

Phone #:

Notes:

Date Received:

Expires: 03/17/2004

Special:

Flammable: Unknown

Health: Unknown

Reactive: Unknown

Gross: 66.10590

Gross Units: g

Net Balance: 4.98989

Net Units: g

The standard was stored in Refrigerator 33 until the Certificate of Analysis was received.

Aliquot - SP0005444

Addition	Component	Withdrawal	Units	Component	Purpose	Computer	Created On	Created By
5	Balance 14		g		INITIAL		3/18/2005 7:06:35AM	Sheehan, Amy
		0.01011	g	Balance 14	PREPARE	BALANCE5	4/5/2005 1:50:15PM	Edwards, Chrissy
			Notes:					
			Notes:					

Characterization - SP0005444

Purity
99 %

Date Characterized
03/31/2005

Characterized By
NA

Created By
Sheehan, Amy

0068

11/21/05



SP5444

Oakwood Products, Inc
741 Old Dunbar Road
West Columbia, SC 29172
Phone(803)739-8800
Fax(803)739-6957

CERTIFICATE OF ANALYSIS

Date: 3-31-05
Material: Pentadecafluorooctanoic acid
Cat. No.: 1319
Cas No.: [335-67-1]
Lot No.: 203
Assay: 99+% by NaOH Titration

Appearance: Colorless, low-melting solid

We have not performed a standardized stability test on this compound. We have stored this compound at room temperature in a tightly sealed container for many years and not noticed any ill effects.

Y.Y.

Yumei Yang
QC Manager

kg

11/21/05

Mar 31 2005 12:11 P.M.

0069

SP5444

43158

BILL
TO: Oxygen Research
3058 Research Drive
State College, PA 16801
(814) 231-8032

SHIP TO: Oxygen Research
ATTN: AMY
3117 Research Drive
State College, PA 16801
(814) 231-8032

Page 1

CUST NO.		P. O. NUMBER		SHIPPING INSTRUCTIONS		COL	PPD	SHIP DATE	TERMS	INVOICE DATE
10926		8957AS10		DHL ground			X	03/15/05	net 30	03/16/05
QUANTITY ORDERED	QUANTITY SHIPPED	QUANTITY NO		ITEM NUMBER		DESCRIPTION				
5G	5G					CC1319-5G Pentadecafluorooctanoic acid, 97% <i>✓ Received</i> <i>03/17/05 AMB</i>				
PACKING LIST										

0070

SP5444

GND

Parcels:

1/1

From: OAKWOOD PRODS
J. Sanide
1741 OLD DUNBAR RD
WEST COLUMBIA, SC 29172
UNITED STATES

ORIGIN:
CAE

Sender's ref:
#957A510

To: Exyger Research
AMY
3117 Research Drive
State College, PA 16801
UNITED STATES

POSTCODE
16801

Tel. 314-331-9032

Day
17TH

Description: DO IN EXCEPTED QUANTITIES

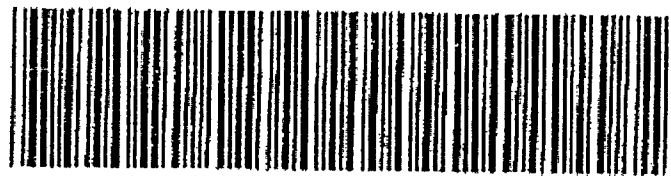
Weight: 1 lbs for 1 pcs
Date: 2005-03-15

DHL standard terms and conditions apply.



**WCEA 3Q
ABH**

(21)JS16301



WAYBILL: 25412924655

(Non-Negotiable)

Bq

11/21/05



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Fax: 814-231-1580

PREPARATION OF STOCK STANDARD SOLUTION

Project	
Exygen Study / Project Number: <u>NA</u>	Customer Project Number: <u>NA</u>
Primary Standard	
Compound Name: <u>Pentadecafluorooctanoic Acid (PFOA)</u>	Exygen Inventory ID: <u>SP0005444</u>
Purity: <u>99 %</u>	Primary Standard Expiration Date: <u>03/31/07</u>
Stock Solution Preparation	
Net Wt. of Compound: <u>0.01011</u> g	Net Wt. Adjusted for Purity: <u>0.01001</u> g
Final Volume: <u>100</u> mL	Stock Solution Concentration: <u>100</u> µg/mL
Solvent Exygen Inventory ID: <u>RE001B205</u>	Solvent Name: <u>Methanol</u>
Stock Solution ID: (SMMDDYY -1,2,3, etc.) <u>S040505-1</u>	Stock Solution Expiration Date: <u>04/05/06</u>
Storage Condition and Location	
Freezer ID: <u>-</u>	Refrigerator ID: <u>33</u>
Walkin Freezer ID: <u>-</u>	Storage Cabinet: <u>-</u>
(Enter the Instrument or Room ID under the appropriate condition)	
Remarks	
Source of PFOA is Oakwood Products. CEE 04/05/05 <i>CE</i> 11/21/05	

Signature: Christine E Edwards

Date: 04/05/05

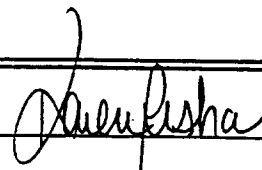


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PREPARATION OF FORTIFICATION STANDARD SOLUTIONS

Project						
Exygen Study / Project Number: <u>PA18</u>				Customer Project Number: <u>—</u>		
Fortification Solution Preparation						
(If 2 or more initial solutions are combined, indicate the combined solutions with a bracket)						
Date Prepared: <u>09/13/05</u>				Fortification Solutions Expiration Date: <u>12/08/05</u>		
Solvent Exygen Inventory ID: <u>RE1021416</u>				Solvent Name: <u>METHANOL</u>		
Initial Soln. ID	Compound Description	Initial Soln. Conc. (µg/mL)	Initial Soln. Aliquoted Volume (mL)	Final Soln. Volume (mL)	Final Soln. Conc. (µg/mL)	Final Soln. ID (FMMDDYY-1,2,3, etc.)
S061605-1	PFOA	1000	10	100	100	F 091305-1
F091305-1	PFOA	100	10	100	10	F 091305-2
F091305-2	PFOA	10	10	100	1.0	F 091305-3
F091305-3	PFOA	1.0	10	100	0.1	F 091305-4
F091305-4	PFOA	0.1	10	100	0.01	F 091305-5
S040505-1	PFOA ^①	100	1.0	100	1.0	F 091305-6
F091305-6	PFOA ^①	1.0	10	100	0.1	F 091305-7
—	—	—	—	—	—	F —
—	—	—	—	—	—	F —
—	—	—	—	—	—	F —
Storage Condition and Location						
Freezer ID: <u>—</u>		Refrigerator ID: <u>33</u>		Room Temperature: <u>—</u>		
Walkin Freezer ID: <u>—</u>		Walkin Cooler ID: <u>—</u>		Storage Cabinet: <u>—</u>		
(Enter the Instrument or Room ID under the appropriate condition)						
Remarks						
① SOURCE OF PFOA IS OAKWOOD PRODUCTS. <u>09/13/05</u> <u>11/21/05</u>						

Signature: 

Date: 09/13/05