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Results of the April 26, 2007 Ammonium Perfluorooctanoate (APFO) Emissions Test on the MA Tower Exhaust

Barr Project No. 29/06-004

Prepared for
Saint Gobain Performance Plastics
Merrimack, New Hampshire

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Results of the April 26, 2007 Ammonium Perfluorooctanoate (APFO) Emissions Tests on the MA Tower Exhaust

Table of Contents

Executive Summary iii

Introduction 1

Results 2

Process Description 3

Review of Quality Assurance Data 4

Stack Test Procedures and Methods 5

List of Tables

Table 1 APFO Test Results – Low APFO Coating – MA Tower

List of Figures

Figure 1 Test Port Locations – MA Tower Exhaust Stack

Figure 2 Traverse Point Locations – MA Tower Exhaust Stack

List of Appendices

Appendix A Report Calculations and Nomenclature

Appendix B Field Data Sheets

Appendix C Laboratory Reports and Sample Chain of Custody

Appendix D Calibration Data

Appendix E Stack Test Plan

Appendix F Project Participants

Executive Summary

Barr Engineering Company performed an emission test on the MA tower exhaust at the Merrimack, New Hampshire facility operated by Saint Gobain Performance Plastics. The test was performed to determine the ammonium perfluorooctanoate (APFO) emission rate. The aqueous fluoropolymer dispersion coating was also sampled and analyzed to determine APFO content.

The tests were performed following US EPA Methods 1-5, modified to collect and analyze for APFO and adapted as necessary to serve this project. Results of the emission test are provided in the Executive Summary Table below:

EXECUTIVE SUMMARY TABLE

MA TOWER APFO TEST

TEST PARAMETER EPA Methods 1-4, Modified Method 5	TEST RESULTS AVERAGE OF 3 TEST RUNS
Gas Temperature, °F	209
Air Flow Rate	
Actual, acfm	3,700
Dry, standard conditions, dscfm	2,900
APFO Concentration	
mg/m3	0.0107
APFO Emissions Rate, g/hr	0.052
Coating APFO Concentration (wet), µg/ml	8.68

Introduction

Barr Engineering Co. performed a modified USEPA Method 5 test to determine ammonium perfluorooctanoate (APFO) emissions at the MA tower exhaust. The coating was also sampled and analyzed to determine APFO concentration. The test was performed on April 26, 2007.

The production coating tower chosen for sampling was Tower MA, an intermediate width tower that has an above average run time percentage, compared to all towers at the Saint Gobain Merrimack facility. The products that are processed on this line are representative of those that are run on all coating towers in Merrimack. This line has been tested on two prior occasions and is most relevant in terms of comparing current, APFO emission rates to the products formerly processed. The same product families were selected for this test as were tested previously.

Tim Russell led the Barr test team. Bruce Briand of Saint Gobain Performance Plastics provided test coordination between process operations and the Barr test team. Jack Glenn of the New Hampshire Department of Environmental Services witnessed the test. A list of project participants is provided in Appendix F.

Results

Results of the ammonium perfluorooctanoate (APFO) test are summarized in the Executive Summary Table. Detailed results are provided in Table 1 and supported by the calculations documented in Appendix A.

Results of the MA Tower test performed on April 26, 2007 are presented in Table 1. The results demonstrate good run-to-run consistency with average measured APFO concentration and emission rate of 0.0107 mg/m ^{3} and 0.052 g/hr, respectively. As stated in the stack test plan, data that was monitored and recorded during the test to characterize manufacturing operations will be provided under separate confidential cover.

Calculations, laboratory, and other supporting data are located in the appendices.

Process Description

The process is a glass fabric coating application involving a preparation of aqueous fluoropolymer dispersion coating, coating application to a glass cloth web in a dip tank, and finally heat treating in three stages—dry zone, bake zone and fuse zone. The product being manufactured requires multiple coating and heat treating applications. The test was conducted during the first coating pass, as the first pass is the heaviest application of coating.

Ammonium perfluorooctanoate (APFO) is contained in the dispersion. Hot gases containing APFO liberated from the three stages of heating are exhausted through the MA tower vent. Operating parameters were recorded for each test run. The parameters included line speed, dispersion specific gravity, scanner product weight, and heating zone temperatures. A summary of the process operating conditions and data will be provided under confidential cover, as the information is proprietary to Saint Gobain.

Review of Quality Assurance Data

Validation of the Exygen Research, A division of MPI Research, Inc. (Exygen) data for the Ammonium Perfluorooctanoate (APFO) analyses of liquid and air samples contained in the Exygen report #L0011423 was completed. The results of this validation are included in the following paragraphs.

All analysis was performed using high pressure liquid chromatography (HPLC) with a tandem mass spectrometric detector (MS/MS). In general, the areas covered by the validation process were, holding times, analytical methodology, calibration procedures, blank analysis, precision and accuracy, analytical quantitation, data package completeness, and an overall data assessment. All raw data were validated to ensure compliance in all areas.

All analytical holding times (analysis within 60 days of collection) were met, all analytical methods followed were appropriate, all calibration procedures were followed and all laboratory and reagent blanks were non-detect for all target parameters. One sample train blank (SG-A-008) reported slight contamination (0.876 ng/ml versus the detection limit of 0.5 ng/ml). Slight contamination of the methanol rinse sample train blank is not unexpected given the sample train blank procedure described in the following section and the effectiveness of methanol as an extraction solvent. All the associated samples had concentrations much greater than five times the blank contamination and no qualification was necessary. The remaining sample train blanks were non-detect for APFO.

APFO detections were identified and quantified correctly. Sample precision and accuracy were evaluated using laboratory and field quality assurance samples including, laboratory control samples, matrix spikes, laboratory duplicates and field spikes and comparing them to set criteria (70 -130% recovery for spikes and <30% relative percent difference for duplicates). All laboratory control samples, matrix spikes and duplicates met all acceptance criteria. All of the field spike values also fell within the set acceptance criteria (70-130%). Therefore none of the data were qualified and should be considered valid as presented.

Overall the laboratory data met the project requirements. The final data met the project data quality objectives and should be considered acceptable.

Stack Test Procedures and Methods

Testing was performed from ports meeting EPA Method 1 (40 CFR Part 60, Appendix A) criteria. Sample port locations and traverse point detail are provided in Figures 1 and 2, respectively. An assessment of cyclonic flow was performed at the measurement location and the source was found to satisfy EPA Method 1 criteria.

Volumetric airflow determinations were performed in accordance with EPA Method 2 (40 CFR Part 60, Appendix A) using an S-type pitot tube in conjunction with the modified EPA Method 5 tests. A 20-point traverse was performed to determine air flow.

Stack oxygen concentrations were monitored with a portable oxygen meter during each run. Stack gas oxygen and carbon dioxide concentrations were measured in accordance with EPA Method 3 (40 CFR Part 60, Appendix A). Stack gas moisture content was determined by the performance of EPA Method 4 (40 CFR Part 60, Appendix A), in conjunction with the modified EPA Method 5 performance test.

Concentrations and emission rates were determined in accordance with EPA Method 5 (40 CFR Part 60, Appendix A) modified to collect and analyze for APFO. APFO was collected in three impingers followed by a glass fiber filter. The three impingers contained 100 ml each of deionized water, 0.1N sodium hydroxide, and 0.01N sodium borate buffer. The filter was recovered and placed in a sample bottle with the methanol rinse of all components of the sample train. The APFO concentrations in air were determined from the sum of the mass of all APFO fractions from the sample collection train. Reagent blanks were taken of all impinger solutions, the methanol used for sample train rinsing and of a glass fiber filter. A sample train blank was taken just prior to sample collection. The sample train was loaded and transported to the test site, a leak check was made of the sample train, and the sample train was then moved to the sample recovery area and recovered in a manner identical that that of subsequent test runs.

Tables

St. Gobain Performance Plastics
Merrimack Plant
Merrimack, NH

Barr Engineering Co.
June 19, 2007

TABLE 1

APFO TEST RESULTS
LOW APFO COATING
Tower MA

Test No. 1

Parameter	Run 1	Run 2	Run 3	Average
Test Date	4/26/2007	4/26/2007	4/26/2007	---
Test Period	945 - 1048	1120 - 1225	1307 - 1410	---
Test Duration, min.	60	60	60	60
Avg. Stack Temperature, deg.F	211	207	209	209
Avg. Moisture Content, %V/V	1.4	2.1	1.9	1.8
Mass of APFO Collected				
All Fractions, ng	10,500	9,640	9,840	9,990
Air Flow Rate				
acfm	3,900	3,500	3,700	3,700
scfm	3,100	2,800	3,000	3,000
dscfm	3,000	2,700	2,900	2,900
Sample Volume				
acf	34.98	32.70	35.05	34.24
dscf	34.28	31.35	33.24	32.96
Isokinetic Variation, %	97.8	100.5	98.8	99.0
APFO Concentration				
mg/m3	0.0108	0.0109	0.0105	0.0107
APFO Emissions Rate				
g/hr	0.056	0.050	0.052	0.052

Figures

SAINT GOBAIN PERFORMANCE PLASTICS
MERRIMACK, NEW HAMPSHIRE

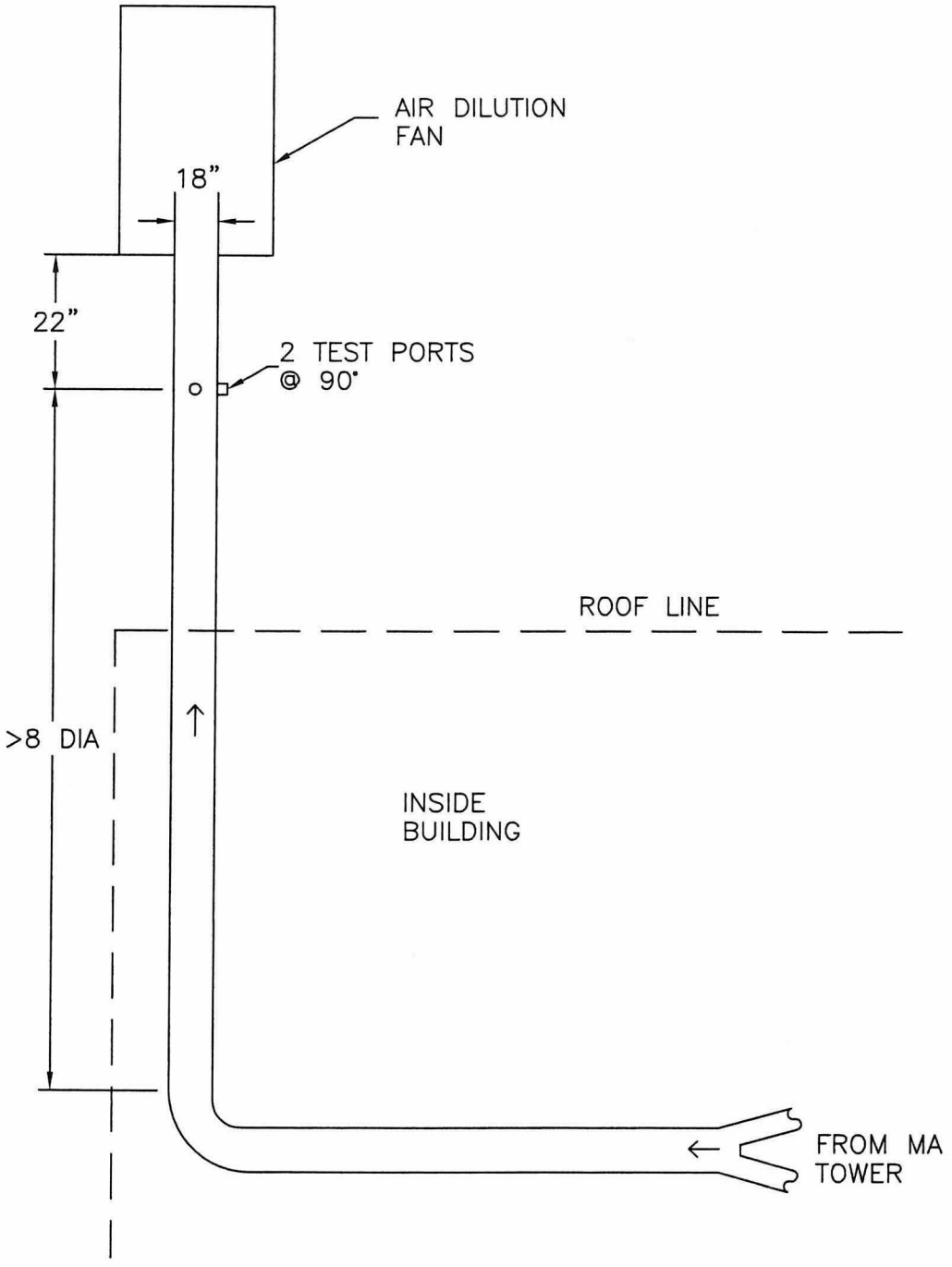
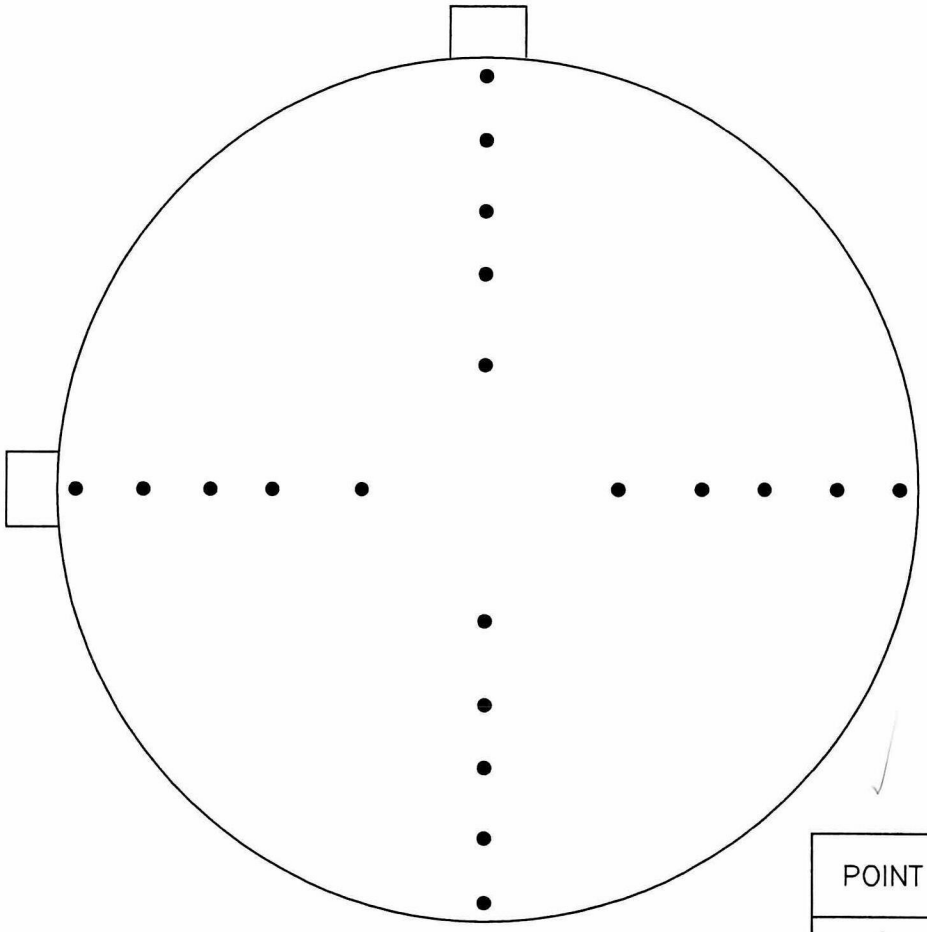


FIGURE 1

TEST PORT LOCATIONS
MA TOWER EXHAUST STACK

NOT TO SCALE

SAINT GOBAIN PERFORMANCE PLASTICS
MERRIMACK, NEW HAMPSHIRE



POINT	INSERTION DEPTH IN "
1	0.50
2	1.48
3	2.63
4	4.07
5	6.16
6	11.84
7	13.93
8	15.37
9	16.52
10	17.50

NO. OF TEST PORTS	2
PORT LENGTH	8.0"
PORT DIAMETER	4"
NO. OF TRAVERSE POINTS	20
DUCT DIAMETER	18.0"

FIGURE 2

TRAVERSE POINT LOCATIONS
MA TOWER EXHAUST STACK

NOT TO SCALE

Appendices

Appendix A

Report Calculations and Nomenclature

St. Gobain Performance Plastics
Merrimack Plant
Merrimack, NH

Barr Engineering Co.
June 19, 2007

Determination of Volumetric Airflow Rate, Gas Composition, Moisture Content, and APFO Emissions
EPA Methods 2, 3, 4, Modified EPA Method 5 for APFO
TEST 1
Tower MA

Input Data	Symbol	Units	Run 1	Run 2	Run 3
Test Date	-	-	4/26/2007	4/26/2007	4/26/2007
Test Period	-	-	945 - 1048	1120 - 1225	1307 - 1410
Number of Sample Ports	-	-	2	2	2
Number of Traverse Points	-	-	20	20	20
Duct Dimensions (diameter or Length x Width)	D, L X W	inches	18.00	18.00	18.00
Barometric Pressure	Pbar	in. Hg	30.05	30.05	30.05
Stack Static Pressure	Pg	in. H2O	-0.44	-0.44	-0.44
Average Stack Temperature, dry bulb	Tsf	degrees F	211	207	209
Actual Dry Gas Meter Volume	Vm	cubic feet	34.98	32.70	35.05
Dry Gas Meter Calibration Factor	Y	-	1.0010	1.0010	1.0010
Average Orifice Meter Pressure Drop	DH	in H2O	1.12	0.94	1.11
Average Meter Temperature	Tmf	degrees F	83	95	101
Pitot Tube Coefficient	Cp	-	0.840	0.840	0.840
Average Square Root of Velocity Head	(DP)^0.5	-	0.580	0.518	0.558
Collected Moisture Volume	V	ml	10	14	14
Orsat Results, Dry Basis					
Carbon Dioxide	%CO2	%v/v	0.50	0.50	0.50
Oxygen	%O2	%v/v	20.5	20.5	20.5
Carbon Monoxide + Nitrogen	-	%v/v	79.0	79.0	79.0
Nozzle Diameter	Dn	in	0.250	0.250	0.250
Run Time	theta	min	60	60	60
Mass of APFO Collected					
All Fractions	APFO	ng	10,508	9,635	9,839
Calculated Data	**Symbol**	**Units**	**Run 1**	**Run 2**	**Run 3**
Average Absolute Stack Temperature (R) Tsr = Tsf + 460	Tsr	degrees R	671	667	669
Stack Pressure Ps = Pbar + Pg / 13.6	Ps	in Hg	30.02	30.02	30.02
Duct Area A = L x W/144	A	Sq. ft	1.767	1.767	1.767
Meter Volume at Standard Conditions Vmstd = 17.64 x Vm x Y x ((Pbar +(DH/13.6))/Tmr)	Vmstd	cubic feet	34.28	31.35	33.24
Average Moisture Content of Stack Gas MC = ((.04707 x V)/((.04707 x V) + Vmstd))) x 100	MC	% Vol	1.35	2.06	1.94
Molecular Weight of Stack Gas, dry Md = (0.44x(%CO2))+(0.32x(%O2))+(0.28x(%N2+%CO))	Md	lb/lbmol	28.90	28.90	28.90
Molecular Weight of Stack Gas, wet Ms = Md x (1-(MC/100))+18 x (MC/100)	Ms	lb/lbmol	28.75	28.68	28.69
Average Stack Gas Velocity Vs = 85.49 x Cp x (dP)^0.5 x ((Tsr/(Ps x Ms))^0.5)	Vs	ft/sec	36.71	32.73	35.35
Actual Volumetric Flowrate Qa = 60 x Vs x A	Qa	acfm	3,892	3,471	3,748
Volumetric Flowrate at Standard Conditions Qs = Qa x (528/(Ts+460)) x (Ps/29.92)	Qs	scfm	3,073	2,755	2,966
Dry Volumetric Flowrate at Standard Conditions Qd = Qa x (1-(MC/100)) x (528/Tsr) x (Ps/29.92)	Qd	dscfm	3,031	2,699	2,909
Nozzle Cross-Sectional Area An =(3.14 x Dn^2) /(4 x 144)	An	sq. ft	0.000341	0.000341	0.000341
Isokinetic Variation I = (0.0945xTsrxVmstd)/(PsxVsxAn x theta x(1-(MC/100)))	I	%	97.8	100.5	98.8
APFO Concentration					
Cs = M / Vmstd	Cs	ng/dscf	307	307	296
Cs = M / 10^6 / Vmstd x 35.31467	Cs	mg/m3	0.0108	0.0109	0.0105
APFO Emission Rate					
E(g/hr) = Cs /10^9 x Qd x 60	E	g/hr	0.0557	0.0498	0.0517

Appendix B

Field Data Sheets

EPA METHOD 1
FIELD DATA SHEET

ST. GOBAIN PERFORMANCE PLASTICS

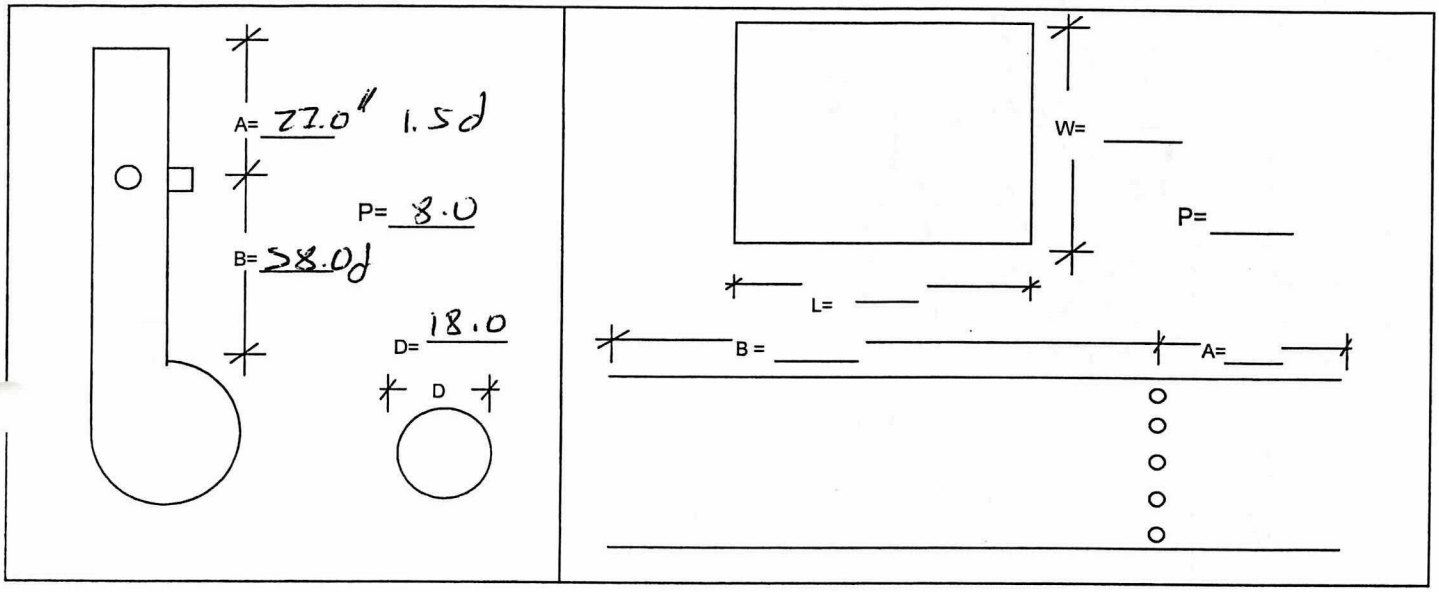
Project APFO Performance Test

Sample Location Tower MA Stack

Sample Date 4-26-07

Test Number 1

Port Locations



Flow Geometry

BARR

EPA METHOD 2
FIELD DATA SHEET

ST. GOBAIN PERFORMANCE PLASTICS

Project	St. Gobain Performance Plastics
Sample Location	Tower MA Stack
Date	7-25-07
Operators	TMR / CAL
Duct Dimensions	18.0 inches
Port Length	8.0 inches
Pitot Tube No.	12 Cp 0.84
Manometer ID	M-8 Bar. ID BA-03
Digital Therm ID	D-13 T.C. ID T-31,33

	Run 1	Run 2	Run 3	Run 4
Bar Press (In Hg)	29.95			
Stat. Press (In H_2O)	-0.44			
Temp - Dry Bulb °F	213			
Temp - Wet Bulb °F	107			
Moist Content - %				
O_2 %	20.0			
Time of Meas.	1530			

Pitot Leak Check Positive: OK Negative: OK

Traverse Point Information			Cyclonic Flow	Velocity Head - Inches H_2O				Stack Temperature - °F			
Point Number	Inches From: Wall	Port	∠°	Run 1 ΔP	Run 2 ΔP	Run 3 ΔP	Run 4 ΔP	Run 1 Temp.	Run 2 Temp.	Run 3 Temp.	Run 4 Temp.
A-1	0.50	8.5	5	0.18							
-2	1.48	9.48	6	0.29							
-3	2.63	10.63	9	0.35							
-4	4.07	12.07	11	0.40							
-5	6.16	14.16	12	0.44							
-6	11.84	19.84	14	0.42							
-7	15.93	21.93	16	0.41							
-8	18.37 ~~24.52~~	23.37	17	0.36							
-9	16.52	24.32	17	0.32							
-10	17.50	25.52	17	0.25							
B-1			6	0.21							
-2			3	0.29							
-3			7	0.33							
-4			8	0.35							
-5			11	0.36							
-6			12	0.40							
-7			12	0.41							
-8			12	0.40							
-9			12	0.31							
-10			12	0.27							

Schematic of Duct Cross-Section

	Run 1	Run 2	Run 3	Run 4
Stack Pres. - In Hg				
Duct Area - Sq Ft.				
Mole Weight - Md				
Mole Weight - Ms				
Avg. Temp. - °F				
Average $\sqrt{\Delta P}$				
Gas Vel - Ft/Sec				
ACFM				
SCFM				
DSCFM				

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EPA METHOD 3
FIELD DATA SHEET

ST. GOBAIN PERFORMANCE PLASTICS

Project: St. Gobain Performance Plastics
Sample Location: Tower MA Stack
Sample Date: 4-26-07

Analyst: T. Russell
Analysis Date: 4-26-07
ORSAT ID: Fyrite - BACHARACH 10-5020
ORSAT Leak Check:
Positive OK Negative OK

Sample Time: 2230
Analysis Time: 2230

Ambient Blank

		CO2		O2	
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	0	Final Point:	0.0	Final Point:	20.9
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.0	Vol. O2 (mL):	20.9
		% CO2:	0.0	**% O2:**	20.9
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	0	Final Point:	0.0	Final Point:	20.9
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.0	Vol. O2 (mL):	20.9
		% CO2:	0.0	**% O2:**	20.9
Test Number:		Initial Point:		Initial Point:	
Run Number:		Final Point:		Final Point:	
Sample Vol. (mL)		Vol. CO2 (mL):		Vol. O2 (mL):	
		% CO2:		**% O2:**	
		Average:	0.0		20.9

Sample Time: 0945-1048
Analysis Time: 2235

		CO2		O2	
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	1	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	1	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	1	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
		Average:	0.5		20.5

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EPA METHOD 3
FIELD DATA SHEET

ST. GOBAIN PERFORMANCE PLASTICS

Project: St. Gobain Performance Plastics
Sample Location: Tower MA Stack
Sample Date: 4-26-07

Analyst: T. Russell
Analysis Date: 4-26-07
ORSAT ID: Fyrite- Bacharach 10-5020
ORSAT Leak Check:
Positive: OK Negative: OK

Sample Time: 1120-1225
Analysis Time: 2245

		CO2		O2	
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	2	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	2	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	2	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
		Average:	0.5		20.5

Sample Time: 1307-1410
Analysis Time: 2255

		CO2		O2	
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	3	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	3	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
Test Number:	1	Initial Point:	0.0	Initial Point:	0.0
Run Number:	3	Final Point:	0.5	Final Point:	20.5
Sample Vol. (mL)	—	Vol. CO2 (mL):	0.5	Vol. O2 (mL):	20.5
		% CO2:	0.5	**% O2:**	20.5
		Average:	0.5		20.5

MODIFIED
EPA METHOD 5 FOR APFO
FIELD DATA SHEET

ST. GOBAIN PERFORMANCE PLASTICS

APFO Performance Test
oc Tower MA Stack Test No. 1 Runs 1 - 2
4-26-07 Pitot No. 7-4
or TMR/CCL CM 5 SM —

Meter γ 1.0010 ΔH @ 1.8331 Cp 0.84 P —
Bar. Pres 30.05 in Hg Stat. Pres -0.44 in H_2O Probe Lngth 7 ft
Liner Type: ☒Glass ☐S.S. ☐Other

Sample Train Leak Rate (cfm)			
Run 3		Run 4	
Pretest 0 at 15 in Hg		Pretest 0 at 15 in Hg	
Posttest 0 at 5 in Hg		Posttest 0 at 5 in Hg	
Pitot (3 in.) Pos.☒ Neg.☒		Pitot (3 in.) Pos.☒ Neg.☒	

TEST RUN 1

Sample Time ΔT	Meter Volume Vm, ft³	Velocity ΔP, in H_2O	Orifice ΔH, in H_2O	Train Vacuum in. Hg	Stack Temp. Ts, °F	Sample Train Temp, °F: Probe	Bypass Filter	Impngr Outlet	Meter Inlet Temp, °F	Meter Outlet Temp, °F
	85.70									
3	92.43	0.34	1.11	0	214	252	253	52	77	76
6	89.17	0.36	1.18	0	214	249	250	49	78	76
9	91.03	0.39	1.27	1.0	215	250	250	48	80	76
12	97.92	0.40	1.30	1.0	217	249	250	48	81	76
15	94.84	0.41	1.34	1.0	216	250	250	48	82	77
18	96.70	0.39	1.28	1.0	214	250	248	50	83	77
21	98.50	0.36	1.19	0.5	212	250	250	51	83	78
24	100.14	0.29	0.96	0	209	250	250	52	84	78
27	101.63	0.25	0.83	0	205	251	251	53	85	79
30	103.11	0.23	0.77	0	206	250	251	54	85	79
33	104.83	0.34	1.12	0.5	212	249	251	56	86	81
36	106.70	0.37	1.23	1.0	210	250	248	54	87	81
39	108.60	0.38	1.26	1.0	211	250	249	54	88	82
42	110.35	0.40	1.33	1.0	211	250	250	55	89	82
45	112.21	0.42	1.40	2.0	211	249	250	55	89	83
48	114.10	0.34	1.14	0.5	210	250	251	56	90	83
51	115.84	0.32	1.07	0	210	251	249	57	91	84
54	117.58	0.30	1.01	0	209	250	250	57	91	85
57	119.27	0.28	0.94	0	208	249	251	57	91	85
60	120.63	0.21	0.71	0	207	251	250	58	91	86
θ= 60	Vm= 34.93		ΔH= 1.12		Ts= 211.1					Tm= 82.83

TEST RUN 2

Meter Volume Vm, ft³	Velocity ΔP, in H_2O	Orifice ΔH, in H_2O	Train Vacuum in. Hg	Stack Temp. Ts, °F	Sample Train Temp, °F: Probe	Bypass Filter	Impngr Outlet	Meter Inlet Temp, °F	Meter Outlet Temp, °F
121.00									
122.67	0.30	1.02	1.0	208	249	251	66	89	88
124.36	0.31	1.05	1.0	209	249	249	65	91	88
126.09	0.34	1.15	1.0	209	248	250	65	92	88
127.83	0.35	1.19	1.0	209	250	249	64	93	88
129.67	0.36	1.22	1.0	209	251	251	63	94	89
131.40	0.36	1.23	1.0	209	249	249	62	95	89
133.18	0.32	1.09	1.0	208	250	250	62	96	90
134.82	0.27	0.93	1.0	207	250	251	62	97	90
136.30	0.22	0.76	0.0	206	251	251	62	97	91
137.75	0.18	0.62	0.0	205	251	250	62	98	91
139.45	0.30	1.03	1.0	208	248	247	62	98	93
141.14	0.32	1.10	1.0	209	248	247	61	99	94
142.77	0.34	1.17	1.0	210	251	250	60	100	94
144.70	0.36	1.24	1.0	210	250	252	59	100	95
146.17	0.27	0.93	0.5	210	249	247	58	101	95
147.70	0.22	0.76	0.0	207	250	250	58	101	95
149.10	0.18	0.63	0.0	203	251	251	59	101	95
150.54	0.17	0.59	0.0	202	252	250	59	101	96
151.80	0.15	0.52	0.0	204	248	250	59	101	96
153.70	0.15	0.52	0.0	203	250	251	60	101	96
Vm= 31.70		ΔH= 0.94		Ts= 207.1	3				Tm= 94.65

O2 - 20.2, 20.2, 20.3, 20.3, 20.4, 20.3, 20.3, 20.3 11 O2 = 20.4

	Initialization Values: Meter Temp	Oxygen Content	Moisture Content	Test Run Times: Start Time	End Time	ORSAT System: Box No.	Bag No.	Bag Vol	cc/min * at 15 in Hg	O2 Reading	Sample Train Components: Filter No.	Nozzle No.	Nozzle Dn	Air Flows: ACFM	DSCFM
Run 1	75	20	2.5	0945	1048	1	1	1.5L	0	—	Glass	2-8	0.250	3900	3000
Run 2	87	20	2.0	1120	1225	1	1	1.5L	0	—	Glass	2-8	0.250	3500	2700

Nozzle Calibration	
Tech. TMR	Date 4-25-07
Nozzle No.	
1	0.248
2	0.250
3	0.251
Avg. in.	0.250

MOISTURE RECOVERY: D.I. NaOH Buff RUN 1	1	2	3	4	Desiccant	Total
Final wt., g	100	102	100	X	1008	
Initial wt., g	100	100	100		1000	
Difference	0	2	0		8	10

Field Train 210mK leak check 15 in Hg 0.0 f.63

D.I. NaOH Buff RUN 2	1	2	3	4	Desiccant	Total
Final wt., g	100	104	101	X	1095	
Initial wt., g	100	100	100		1086	
Difference	0	4	1		9	14

BARR

MODIFIED EPA METHOD 5 FOR APFO FIELD DATA SHEET

ST. GOBAIN PERFORMANCE PLASTICS

Project: APFO Performance Test
Smpl Loc: Tower MA Stack
Test No.: 1
Runs: 3 - 4
Date: 4-26-07
Pitot No.: 7-4
Operator: TMR/ICL
CM: 5
SM: —
Meter γ: 1.0010
ΔH @: 1.8381
Cp: 0.84
P: —
Bar. Pres: 30.05 in Hg
Stat. Pres: -0.44 in H_2O
Probe Lngth: 7 ft
Liner Type: ☒ Glass ☐ S.S. ☐ Other

Sample Train Leak Rate (cfm)			
Run 3		Run 4	
Pretest	0 at 15 in Hg	Pretest	at 15 in Hg
Posttest	0 at 5 in Hg	Posttest	at in Hg
Pitot (3 in.)	Pos. ☒ Neg. ☒	Pitot (3 in.)	Pos. ☐ Neg. ☐

		TEST RUN 3										TEST RUN 4									
Sample Point	Sample Time ΔT	Meter Volume Vm, ft³	Velocity ΔP, in H_2O	Orifice ΔH, in H_2O	Train Vacuum in. Hg	Stack Temp. Ts, °F	Sample Train Temp, °F: Probe	Bypass Filter	Impngr Outlet	Meter Inlet Temp, °F	Meter Outlet Temp, °F	Meter Volume Vm, ft³	Velocity ΔP, in H_2O	Orifice ΔH, in H_2O	Train Vacuum in. Hg	Stack Temp. Ts, °F	Sample Train Temp, °F: Probe	Filter	Impngr Outlet	Meter Inlet Temp, °F	Meter Outlet Temp, °F
		153.40																			
A-10	3	155.15	0.35	1.20	1.5	208	249	250	60	98	97										
-9	6	157.07	0.33	1.31	2.0	210	249	250	56	99	97										
-8	9	158.88	0.40	1.38	2.0	211	250	251	55	100	97										
-7	12	160.90	0.41	1.38	2.0	211	250	251	55	101	97										
-6	15	162.72	0.41	1.41	2.0	211	250	250	56	102	97										
-5	18	164.67	0.40	1.38	2.0	211	251	251	57	103	97										
-4	21	166.60	0.37	1.28	2.0	211	250	251	57	103	98										
-3	24	168.50	0.36	1.25	2.0	210	249	249	58	104	98										
-2	27	170.35	0.32	1.11	2.0	209	250	249	58	104	98										
-1	30	172.20	0.30	1.04	2.0	209	251	251	59	104	98										
0-10	33	174.04	0.33	1.15	2.0	208	249	252	57	104	99										
-9	36	175.84	0.34	1.18	2.0	210	249	249	57	104	99										
-8	39	177.68	0.38	1.31	2.0	211	250	251	58	104	99										
-7	42	179.54	0.42	1.45	2.0	211	249	250	59	105	100										
-6	45	181.50	0.36	1.25	2.0	211	250	249	60	105	100										
-5	48	183.00	0.24	0.84	1.0	208	250	250	61	105	100										
-4	51	184.44	0.15	0.52	1.0	207	250	250	62	105	100										
-3	54	185.75	0.15	0.52	1.0	207	251	250	62	105	101										
-2	57	187.05	0.17	0.59	1.0	206	251	251	63	105	101										
-1	60	188.45	0.16	0.56	1.0	205	250	250	64	105	101										
	θ= 60	Vm= 35.05		ΔH= 1.11		Ts= 209.3					Tm= 101.0	Vm=		ΔH=		Ts=					Tm=

O2 = 20.4, 20.4, 20.4

	Initialization Values			Test Run Times		ORSAT System					Sample Train Components			Air Flows	
	Meter Temp	Oxygen Content	Moisture Content	Start Time	End Time	Box No.	Bag No.	Bag Vol	cc/min * at 15 in Hg	O2 Reading	Filter No.	Nozzle No.	Nozzle Dn	ACFM	DSCFM
Run 3	95	20	2.0	1307	1410	B1	3	15L	0	—	61935	2-8	0.250	2700	2900
Run 4															

Nozzle Calibration	
Tech.	Date
Nozzle No.	
1	see
2	Runs 1-2
3	
Avg. in.	

MOISTURE RECOVERY: 0.1 NaOH RUN 3						
Impinger	1	2	3	4	Desiccant	Total
Final wt., g	100	104	102	X	1016	
Initial wt., g	100	100	100	X	1008	
Diff	0	4	2	X	8	14

RUN 4					
1	2	3	4	Desiccant	Total

Barr
Engineering Company

MODIFIED EPA METHOD 5 FOR APFO -- FIELD DATA SHEET-RUN 1

Project	St. Gobain Performance Plastic		Train C-5, ---, ---		Pitot Tube No.	7-4		Cp 0.840			Sample Train Leak Rate, cfm:				
Sample Location	Tower MA		Meter Y	1.0010			Bar.Press.	30.05	in. Hg		Pretest	0.000	at 15	in. Hg	
Date	04/26/07	Test 1	Run # 1		Orifice H@	1.8381	Stat Press.	-0.44	in. H2O		Posttest	0.000	at 5	in. Hg	
Operators	TMR/CCL	Probe Length	7 feet				Probe Liner Type:	Glass			Other				

Sample Point	Sample Time DT	Actual Meter Vol Vm, ft3	Velocity Head DP, in. H_2O	Orifice DH in. H_2O	Ideal Point Volume Vm, ft^3	Ideal Meter Vol Vm, ft3	Sample Train Vacuum in. Hg	Stack Temp Ts,°F	Sample Train Temperatures, °°F					Oxygen Content, %
									Filter	Probe	Impinger Outlet	Meter Inlet	Meter Outlet	
Start Time	0945	85.70												
1	3.0	87.43	0.340	1.11	1.76	87.46	*	214	*	*	*	77	76	20
2	6.0	89.17	0.360	1.18	1.82	89.28	*	214	*	*	*	78	76	
3	9.0	91.03	0.390	1.27	1.89	91.17	*	215	*	*	*	80	76	
4	12.0	92.92	0.400	1.30	1.92	93.09	*	217	*	*	*	81	76	
5	15.0	94.84	0.410	1.34	1.95	95.04	*	216	*	*	*	82	77	
6	18.0	96.70	0.390	1.28	1.90	96.94	*	214	*	*	*	83	77	
7	21.0	98.50	0.360	1.19	1.83	98.78	*	212	*	*	*	83	78	
8	24.0	100.14	0.290	0.96	1.65	100.43	*	209	*	*	*	84	78	
9	27.0	101.63	0.250	0.83	1.54	101.97	*	205	*	*	*	85	79	
10	30.0	103.11	0.230	0.77	1.48	103.44	*	206	*	*	*	85	79	
11	33.0	104.83	0.340	1.12	1.79	105.23	*	212	*	*	*	86	81	
12	36.0	106.70	0.370	1.23	1.87	107.10	*	210	*	*	*	87	81	
13	39.0	108.60	0.380	1.26	1.90	109.00	*	211	*	*	*	88	82	
14	42.0	110.35	0.400	1.33	1.95	110.96	*	211	*	*	*	89	82	
15	45.0	112.21	0.420	1.40	2.00	112.96	*	211	*	*	*	89	83	
16	48.0	114.10	0.340	1.14	1.80	114.76	*	210	*	*	*	90	83	
17	51.0	115.84	0.320	1.07	1.75	116.51	*	210	*	*	*	91	84	
18	54.0	117.58	0.300	1.01	1.70	118.21	*	209	*	*	*	91	85	
19	57.0	119.27	0.280	0.94	1.65	119.86	*	208	*	*	*	91	85	
20	60.0	120.68	0.210	0.71	1.43	121.29	*	207	*	*	*	91	86	
End Time	1048													
Run Time	60		Avg DH=	1.12			Avg Ts=	211.05				Avg Tm=	82.88	

Integrated Gas Sampling Data :

Box No.	NA	Leak Rate	0	cc/min	Filter No.	Glass	
Bag No.	1	O_2 Reading	NA	%	Nozzle No.	2-8	
Bag Vol.	15L	O_2 Analyzer No.	NA		Nozzle Dn.	0.250	

MOISTURE RECOVERY DATA :

Impinger	1	2	3	4	Desiccant	Total
Final wt., g	100	102	100	0	1008	
Initial wt., g	100	100	100	0	1000	
Difference	0	2	0	0	8	10

Barr
Engineering Company

MODIFIED EPA METHOD 5 FOR APFO -- FIELD DATA SHEET-RUN 2

Project	St. Gobain Performance Plastic	Train C-5, ---, ---	Pitot Tube No.	7-4		Cp 0.840		Sample Train Leak Rate, cfm:				
Sample Location	Tower MA	Meter Y	1.0010		Bar.Press.	30.05	in. Hg	Pretest	0.000	at 15	in. Hg	
Date	04/26/07	Test 1	Run # 2	Orifice H@ 1.8381	Stat Press.	-0.44	in. H2O	Posttest	0.000	at 5	in. Hg	
Operators	TMR/CCL	Probe Length	7 feet		Probe Liner Type:	Glass		Other				

Sample Point	Sample Time DT	Actual Meter Vol Vm, ft3	Velocity Head DP, in. H_2O	Orifice DH in. H_2O	Ideal Point Volume Vm, ft^3	Ideal Meter Vol Vm, ft3	Sample Train Vacuum in. Hg	Stack Temp Ts,°F	Sample Train Temperatures, °F					Oxygen Content, %
									Filter	Probe	Impinger Outlet	Meter Inlet	Meter Outlet	
Start Time	1120	121.00												
1	3.0	122.67	0.300	1.02	1.71	122.71	*	208	*	*	*	89	88	20
2	6.0	124.36	0.310	1.05	1.74	124.45	*	209	*	*	*	91	88	
3	9.0	126.09	0.340	1.15	1.82	126.27	*	209	*	*	*	92	88	
4	12.0	127.83	0.350	1.19	1.85	128.12	*	209	*	*	*	93	88	
5	15.0	129.67	0.360	1.22	1.88	130.01	*	209	*	*	*	94	89	
6	18.0	131.40	0.360	1.23	1.88	131.89	*	209	*	*	*	95	89	
7	21.0	133.18	0.320	1.09	1.78	133.67	*	208	*	*	*	96	90	
8	24.0	134.82	0.270	0.93	1.64	135.31	*	207	*	*	*	97	90	
9	27.0	136.30	0.220	0.76	1.48	136.79	*	206	*	*	*	97	91	
10	30.0	137.75	0.180	0.62	1.34	138.13	*	205	*	*	*	98	91	
11	33.0	139.45	0.300	1.03	1.73	139.86	*	208	*	*	*	98	93	
12	36.0	141.14	0.320	1.10	1.79	141.65	*	209	*	*	*	99	94	
13	39.0	142.77	0.340	1.17	1.85	143.50	*	210	*	*	*	100	94	
14	42.0	144.70	0.360	1.24	1.90	145.40	*	210	*	*	*	100	95	
15	45.0	146.17	0.270	0.93	1.65	147.05	*	210	*	*	*	101	95	
16	48.0	147.70	0.220	0.76	1.49	148.54	*	207	*	*	*	101	95	
17	51.0	149.10	0.180	0.63	1.35	149.90	*	203	*	*	*	101	95	
18	54.0	150.54	0.170	0.59	1.32	151.21	*	202	*	*	*	101	96	
19	57.0	151.80	0.150	0.52	1.24	152.45	*	204	*	*	*	101	96	
20	60.0	153.70	0.150	0.52	1.24	153.69	*	203	*	*	*	101	96	
End Time	1225													
Run Time	60		Avg DH=	0.94			Avg Ts=	207.25				Avg Tm=	94.65	

Integrated Gas Sampling Data :

Box No.	NA	Leak Rate	0	cc/min	Filter No.	Glass	
Bag No.	2	O_2 Reading	NA	%	Nozzle No.	2-8	
Bag Vol.	15L	O_2 Analyzer No.	NA		Nozzle Dn.	0.250	

MOISTURE RECOVERY DATA :

Impinger	1	2	3	4	Desiccant	Total
Final wt., g	100	104	101	0	1095	
Initial wt., g	100	100	100	0	1086	
Difference	0	4	1	0	9	14

Barr
Engineering Company

MODIFIED EPA METHOD 5 FOR APFO -- FIELD DATA SHEET-RUN 3

Project	St. Gobain Performance Plastic	Train C-5, ---, ---	Pitot Tube No.	7-4		Cp 0.840		Sample Train Leak Rate, cfm:				
Sample Location	Tower MA	Meter Y	1.0010		Bar.Press.	30.05	in. Hg	Pretest	0.000	at 15	in. Hg	
Date	04/26/07	Test 1	Run # 3	Orifice H@	1.8381	Stat Press.	-0.44	in. H2O	Posttest	0.000	at 5	in. Hg
Operators	TMR/CCL	Probe Length	7 feet			Probe Liner Type:	Glass		Other			

Sample Point	Sample Time DT	Actual Meter Vol Vm, ft3	Velocity Head DP, in. H_2O	Orifice DH in. H_2O	Ideal Point Volume Vm, ft^3	Ideal Meter Vol Vm, ft3	Sample Train Vacuum in. Hg	Stack Temp Ts,°F	Sample Train Temperatures, °F					Oxygen Content, %
									Filter	Probe	Impinger Outlet	Meter Inlet	Meter Outlet	
Start Time	1307	153.40												
1	3.0	155.15	0.350	1.20	1.87	155.27	*	208	*	*	*	98	97	20
2	6.0	157.07	0.380	1.31	1.96	157.23	*	210	*	*	*	99	97	
3	9.0	158.88	0.400	1.38	2.01	159.23	*	211	*	*	*	100	97	
4	12.0	160.90	0.400	1.38	2.01	161.24	*	211	*	*	*	101	97	
5	15.0	162.72	0.410	1.41	2.04	163.28	*	211	*	*	*	102	97	
6	18.0	164.67	0.400	1.38	2.01	165.29	*	211	*	*	*	103	97	
7	21.0	166.60	0.370	1.28	1.94	167.23	*	211	*	*	*	103	98	
8	24.0	168.50	0.360	1.25	1.91	169.14	*	210	*	*	*	104	98	
9	27.0	170.35	0.320	1.11	1.81	170.95	*	209	*	*	*	104	98	
10	30.0	172.20	0.300	1.04	1.75	172.70	*	209	*	*	*	104	98	
11	33.0	174.04	0.330	1.15	1.84	174.53	*	208	*	*	*	104	99	
12	36.0	175.84	0.340	1.18	1.86	176.40	*	210	*	*	*	104	99	
13	39.0	177.68	0.380	1.31	1.97	178.37	*	211	*	*	*	104	99	
14	42.0	179.54	0.420	1.45	2.07	180.44	*	211	*	*	*	105	100	
15	45.0	181.50	0.360	1.25	1.92	182.36	*	211	*	*	*	105	100	
16	48.0	183.00	0.240	0.84	1.57	183.93	*	208	*	*	*	105	100	
17	51.0	184.44	0.150	0.52	1.24	185.17	*	207	*	*	*	105	100	
18	54.0	185.75	0.150	0.52	1.24	186.41	*	207	*	*	*	105	101	
19	57.0	187.05	0.170	0.59	1.32	187.74	*	206	*	*	*	105	101	
20	60.0	188.45	0.160	0.56	1.29	189.02	*	205	*	*	*	105	101	
End Time	1410													
Run Time	60		Avg DH=	1.11			Avg Ts=	209.25				Avg Tm=	100.98	

Integrated Gas Sampling Data :

Box No.	NA	Leak Rate	0	cc/min	Filter No.	Glass
Bag No.	3	O_2 Reading	NA	%	Nozzle No.	2-8
Bag Vol.	15L	O_2 Analyzer No.	NA		Nozzle Dn.	0.250

MOISTURE RECOVERY DATA :

Impinger	1	2	3	4	Desiccant	Total
Final wt., g	100	104	102	0	1016	
Initial wt., g	100	100	100	0	1008	
Difference	0	4	2	0	8	14

Appendix C

Laboratory Reports and Sample Chain of Custody

SAINT-GOBAIN
PERFORMANCE PLASTICS

Health, Safety and Environment

July 30, 2007

Ms. Pamela Monroe
Compliance Bureau Administrator
N.H. Department of
Environmental Services
Air Resources Division
29 Hazen Drive
Concord, NH 03302-0095

RECEIVED
NEW HAMPSHIRE
JUL 30 2007
AIR RESOURCES DIVISION

Re: Saint-Gobain Performance Plastics Site in Merrimack, NH
Outcome of APFO Emissions Test Conducted in April '07
Administrative Order by Consent: ARD 06-006

Dear Pamela:

Under the terms of the above referenced AOC the Saint-Gobain Performance Plastics site in Merrimack, NH was required to demonstrate that the conversion to "APFO" lite fluoropolymer dispersions during the course of 2006 (100% converted effective January 1, 2007) has allowed the site to meet the NHDES RTAP standards for ammonium perfluorooctanoate (APFO).

A NHDES approved emissions test was conducted on April 26, 2007. The test report was provided to Saint-Gobain several weeks ago and we have been busy analyzing and interpreting the results. Attachment "A" shows the progression of key dates and accomplishments relative to the conversion effort.

I am very pleased to report that the recent testing shows that the SGPPL Merrimack site has met the 24 hour and annual RTAP criteria since January 1, 2007. A full report of the Barr Engineering emissions test is included for your review. In the course of developing the AOC that was signed by relevant parties in August '06 and prior to the emissions test in April '07 there was much interaction between technical resources within SGPPL and NHDES (and Barr Engineering). Key to those discussions were exchanges concerning the basis of emission rate estimates for non-tested sources, input parameters for ensuing air dispersion modeling and sampling and analytical methods used during the tests.

The table shown below highlights the average daily APFO emission rate for 2007 all sources combined. This rate was derived using assumptions and methods previously discussed and approved by NHDES personnel (and reflects the new test data). Attachment "B" shows a summary of the test results. Attachment "C" is intended to demonstrate how the test results were used to determine estimated facility emissions.

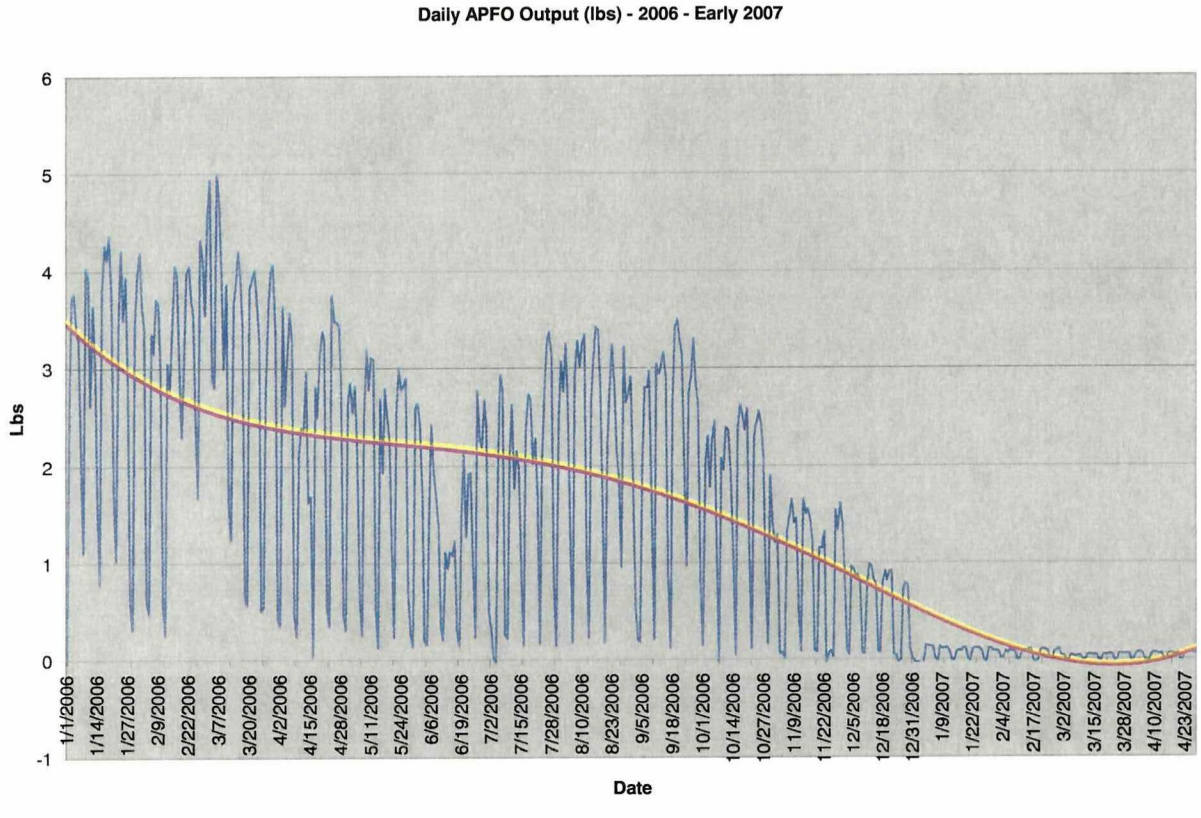
Calculated APFO Emissions

	RTAP Standard	SGPPL Merrimack 2007 Daily Average
24 hour basis	0.05 $\mu g/m^3$	0.021 $\mu g/m^3$
Annual basis	0.024 $\mu g/m^3$	0.0052 $\mu g/m^3$

The table below shows the incoming APFO concentration present in the fluoropolymer dispersions as received from the suppliers. Note the average reduction is 99.5 percent versus pre-APFO "lite" dispersions.

Supplier	Prior APFO concentration, ppm wet weight basis	Current 2007 APFO concentration, ppm actual 2007 results	% Reduction
"A"	2000	20	99
"B"	600	1.5	99.75
"C"	2000	5.9	99.7

Average reduction is 99.5%.



As the table at the top of the preceding page shows, 0.021 ug/m^3 is less than half the NHDES RTAP 24 hour limit. Modeled conditions represent an average 2007 processing day. Heavier processing days were also evaluated and the results were still below the NHDES RTAP 24 hour and annual limits.

We hope this information satisfactorily demonstrates that SGPPL Merrimack does meet the requisite NHDES standards established for APFO as a result of the conversion to the new generation of fluoropolymer dispersions that were phased in during 2006.

Please direct any questions that DES technical personnel might have to my attention and I will ensure that they are answered in a timely manner.

I want to thank everyone who has been involved in this effort at DES for the effort expended and for fostering the extraordinarily cooperative environment that helped us navigate through this complex project.

As always, please let me know if you have additional questions.

Sincerely,

Edward J. Canning
Manager Health, Safety & Environment
Saint-Gobain Performance Plastics
One Sealants Park
Granville, NY 12832
(518) 642-2200 x231
Mobile: (518) 796-3858

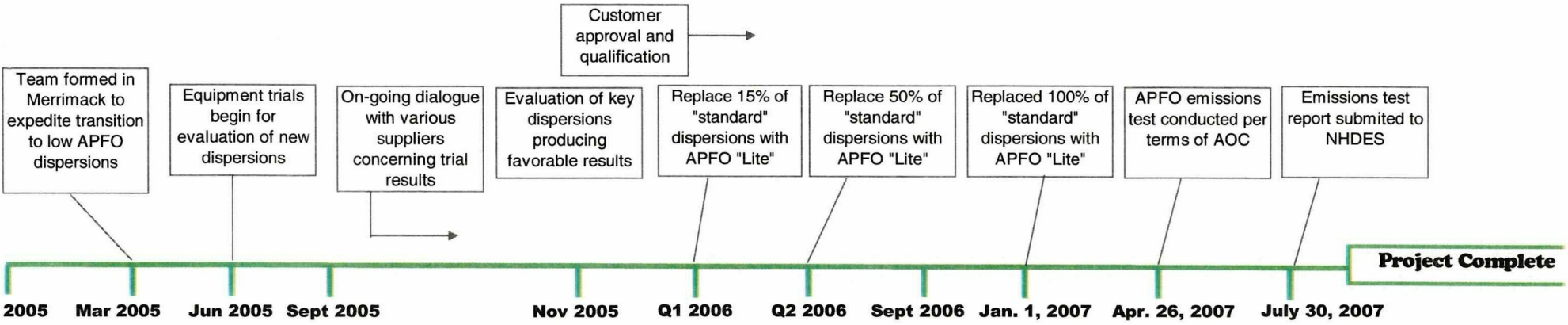
cc: Barbara Hoffman
Enforcement Section Supervisor
DES - Air Resources Division
Concord, NH 03302-0095
w/o report

Bruce Briand – SG Merrimack

Attachment "A"

Merrimack, New Hampshire

Transition to APFO "Lite" Dispersions



Transition Results

Attachment "B"

St. Gobain Performance Plastics
Merrimack Plant
Merrimack, NH

Barr Engineering Co.
June 7, 2007

TABLE 1

APFO TEST RESULTS
LOW APFO COATING
Tower MA

Test No. 1

Parameter	Run 1	Run 2	Run 3	Average
Test Date	4/26/2007	4/26/2007	4/26/2007	---
Test Period	945 - 1048	1120 - 1225	1307 - 1410	---
Test Duration, min.	60	60	60	60
Avg. Stack Temperature, deg.F	211	207	209	209
Avg. Moisture Content, %V/V	1.4	2.1	1.9	1.8
Mass of APFO Collected				
All Fractions, ng	10,500	9,640	9,840	9,990
Air Flow Rate				
acfm	3,900	3,500	3,700	3,700
scfm	3,100	2,900	3,000	3,000
dscfm	3,000	2,700	2,900	2,900
Sample Volume				
acf	34.98	32.70	35.05	34.24
dscf	34.25	31.95	33.24	32.96
Isokinetic Variation, %	97.9	100.5	99.6	99.3
APFO Concentration				
mg/m3	0.0106	0.0109	0.0105	0.0107
APFO Emissions Rate				
g/hr	0.054	0.050	0.052	0.052

Attachment "C"

The average measured output from the Barr Engineering emissions test of tower MA on April 26 was **0.052 grams per hour**. This emission rate was extrapolated to all other sources (e.g., coating lines) using the same assumptions that were previously explained to NHDES (see below).

Emissions from three of the 13 coating towers in Merrimack have been tested. The tests conducted previously have allowed us to extrapolate to the remaining 10 towers taking the following information into account:

- Destruction efficiency
- Tower width (three times the width = 3X the emissions);
- Number of stages (i.e., if we measured a five stage casting line—a single stage line would have 1/5 the emissions and vice versa) ;
- Speed – a coating speed twice as high as that on a measured tower would result in twice the input of APFO and twice the output of APFO;
- Weight pick up per pass (OSY = ounces per square yard);
- Hours that the tower is run (i.e. whether the tower is run 24 hours a day or 8 hours a day);

An air dispersion model (AERMOD version #07026) was rerun using the newly adjusted process rates.

The process rates were calculated as follows:

Equation :

$$\text{Twr MA emissions} \times \frac{\text{1- dest Eff}}{\text{1-MA Des Eff}} \times \frac{\text{Tower width}}{\text{MA width}} \times \frac{\text{Stages}}{\text{MA stages}} \times \frac{\text{Tower speed}}{\text{MA Speed}} \times \frac{\text{input APFO conc}}{\text{conc of MA ppm}} \times \frac{\text{pickup in osy}}{\text{pick up at MA}} \times \frac{\text{hours run}}{24} = \text{Emission Rate}$$

Example for Tower MB:

$$\text{0.052g/hr} \times \frac{1-.863}{1-.887} \times \frac{172}{60} \times \frac{1}{1} \times \frac{5}{18} \times \frac{2.7}{0.8} \times \frac{20.5}{24} \times \frac{26.3}{5.99} = .635\text{g/hr or } 635\text{ mg/hr}$$

The same methodology has been applied to all other towers (sources)

above information is provided and we've had a chance to evaluate.

TEST PLAN FOR POLYMER CLOTH COATING FACILITY

SAINT GOBAIN PERFORMANCE PLASTICS CORPORATION
MERRIMACK, NEW HAMPSHIRE

Date test plan created/revised: March 23, 2007

Scheduled test date(s): April 25, 2007

PART I. GENERAL INFORMATION

Name and address of emission facility: Saint Gobain Performance Plastics Corporation
701 Daniel Webster Highway
Merrimack, NH 03054

Facility contact: Bruce Briand (603) 424-9000, x2341
Health, Safety & Environmental Manager
Fax (603) 424-9012

Air Emission Facility ID Number: 3301100165

Reason for the test: Determine ammonium perfluorooctanoate (APFO) stack emission rate for air dispersion modeling input.

Testing Company and Contact: Barr Engineering Company (952) 832-2630
Tim Russell Fax (952) 832-2601
Vice President, Chemical Engineer trussell@barr.com
4700 W. 77th Street
Minneapolis, MN 55435

PART II. TESTING REQUIREMENTS

The table below provides a summary of the pollutants to be tested and test methods.

Source Description	Limitation Basis of Pollutant Tested	Pollutant Tested and Applicable Emission Limit	Specific Methods/Procedures Citation
MA Coating Tower Exhaust	none	Ammonium Perfluorooctanoate (APFO) 3825-26-1	EPA Methods 1-4, Modified EPA Method 5 40 CFR Part 60, Appendix A

PART III. OPERATING CONDITIONS (CB)

The table and paragraph below identifies the process equipment to be tested, the operating parameters to be monitored and reported, and the rationale for testing.

Process Equipment Description for Units to be Tested	MA Coating Tower
Process Equipment Parameter Monitoring During Performance Test	Dispersion identification, dispersion application rate, web width, line speed, coating pass number. Information will be recorded during the test and provided separate from the test report under confidential cover.
Control Equipment Description	The source is uncontrolled
Control Equipment Operating Parameter During Performance Test	Not applicable

The production coating tower chosen for sampling is Tower MA, an intermediate width tower that has an above average run time percentage, compared to all towers in Merrimack. The products that are processed on this line are representative of those that are run on all coating towers in Merrimack. This line has been tested on two prior occasions and we feel would be the most relevant in terms of comparing current, APFO light emission rates to the products formerly processed. The same product families would be selected for the upcoming test as were tested previously.

PART IV. TEST AND ANALYTICAL METHODS

The following is a description of the test methods, number of test runs, length of test runs, and sampling rate for each pollutant

A. EPA Method 1 for the location of sampling ports and points. One determination per test location.

B. EPA Method 2 for stack gas velocity and volumetric flow rate. One determination per EPA Method 5 test run.

C. EPA Method 3 to determine stack gas molecular weight. One determination per EPA Method 5 test run.

D. EPA Method 4 for determination of moisture content in stack gas. One determination per EPA Method 5 test run.

E. EPA Method 5 Modified to collect APFO. The sample train will consist of three impingers followed by an unheated glass fiber filter. The three impingers will be charged in order with 100 ml each of de-ionized water, 0.1*N* sodium hydroxide, and 0.01*N* sodium borate buffer. The filter will be collected and placed in a sample bottle with a methanol rinse of all components of the

sample train. Each of the four sample fractions will be recovered and analyzed separately for the determination of total APFO mass. The modification of Method 5 follows test protocols previously accepted by the USEPA.

F. Sampling times will be three runs of 60 minute duration.

G. Exygen Research of State College, PA will perform the laboratory analysis of the air impinger and dispersion coating samples using high pressure liquid chromatography and tandem mass spectrometry (LC/MS/MS) by analytical methods they have developed for the measurement of APFO.

PART V. CALIBRATION METHODS AND PRETEST PREPARATIONS

A. The EPA Method 5 Equipment will be calibrated prior to the test in accordance with the requirements in 40 CFR Part 60, Appendix A, EPA Quality Assurance Handbook for Air Pollutant Measurement Systems: Volume III Stationary Sources Specific Methods and Barr Standard Operating Procedures. Calibration data will be available onsite for review. Samples of calibration and field sample data sheets are provided as an attachment to this document.

B. Pretest preparations include cleaning and methanol rinse of all sample train glassware components and the preparation of sampling reagents in addition to that standard to the mobilization of particulate measurement and support equipment. Special care is made to eliminate all components of the sample train that may contain fluoropolymer constituents in order to avoid trace contamination of the samples from those components. Examples include avoiding the use of Teflon™ compression fittings or ferrules, sealing tape or sample jar liners, and fluoropolymer encapsulated o-ring seals.

PART VI. QUALITY ASSURANCE AND LABORATORY STANDARD OPERATING PROCEDURES

A. MPI Research, State College Facility, (previously Exygen Research) has developed standard operating procedures for the laboratory analysis of APFO in air impinger and liquid samples using analytical method number ExM-079-007C entitled "Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study." Dispersion samples will be analyzed using analytical method number ExM-079-007A entitled "Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Water by

LC/MS/MS for SPI Processors Mass Balance Study." The SOPs are considered to be proprietary business information by Exygen and can be shared upon execution of a confidentiality agreement.

B. A complete review of the analytical laboratory recoveries, duplicates, and raw data by a member of Barr's Data Quality Assurance staff will be performed and presented in the test report.

PART VII. SAFETY

Emergency procedures are outlined in Saint Gobain's "Call to Action Safety Awareness Program" which can be reviewed upon arrival at the facility. Facility emergencies notifications are made to the facility main front desk at 603-424-900 in the event that medical, fire or other emergency response is needed.

PART VIII. TEST SCHEDULE

Week of April 23-27, 2007

April 23 Monday	April 24 Tuesday	April 25 Wednesday	April 26 Thursday	April 27 Friday
		Safety Meeting Pretest Coordination Meeting Equipment Mobilization	Perform Test (0700-1400) Recover Samples (1400-1500) Demobilize (1500-1700)	Contingency Test Day

PART VII. TEST REPORT

One complete test report (bound copy) will be submitted within 60 days of the test performance date.

Submittal Address: Mr. Jim Black
New Hampshire Department of Environmental Services
29 Hazen Drive
P.O. Box 95
Concord, NH 03302-0095

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1: J Occup Environ Hyg. 2005 Jun;2(6):307-13.

MetaPress Links

Method for the determination of perfluorooctanoic acid in air samples using liquid chromatography with mass spectrometry.

Kaiser MA, Larsen BS, Dawson BJ, Kurtz K, Lieckfield R Jr, Miller JR, Flaherty J.

E.I. DuPont de Nemours, Inc., Wilmington, Delaware, USA. mary.A.Kaiser@usa.dupont.com

Perfluorooctanoic acid is a completely fluorinated carboxylic acid that is usually used in the ammonium salt form as a processing aid in the production of many fluoropolymers and fluoroelastomers. Ammonium perfluorooctanoate readily dissociates in water to give the ammonium and perfluorooctanoate ions. Perfluorooctanoate has been reported to be present in low levels in human serum in the United States and Europe. This study reports on the development and validation of a method for the determination of perfluorooctanoic acid in air samples. This method uses the Occupational Safety and Health Administration (OSHA) Versatile Sampler (OVS) with a nominal 0.3 micro m filter and polystyrene resin sorbent (XAD-2 or XAD-4) followed by determination of the perfluorooctanoate anion by liquid chromatography mass spectrometry. The method was validated in the range of 0.474 to 47.4 microg/m3 for a 480-L sample. Breakthrough studies showed samples could be collected at 1 L/min for 24 hours or at 15 L/min up to 8 hours without breakthrough. Extract storage stability tests showed that sample extracts in methanol remain stable in glass autosampler vials for up to 13 days following initial injection. Perfluorooctanoic acid stability on OVS tubes was unaffected at both refrigerated and ambient temperatures. The overall average retention efficiency was 92.1% with a pooled RSD95 of 5.8% at five concentration levels (0.474 microg/m3 to 47.4 microg/m3).

Related Links

Analysis of perfluorocarboxylic acids in air. [J Occup Environ Hyg. 2007]

Analytical techniques and method validation for the measurement of selected semivolatile and nonvolatile organofluorochemicals in air. [J Occup Environ Hyg. 2004]

Characterizing perfluorooctanoate in ambient air near the fence line of a manufacturing facility: comparing modeled and monitored values. [J Air Waste Manag Assoc. 2006]

Quantitative determination of perfluorooctanoic acid in serum and plasma by liquid chromatography tandem mass spectrometry. [J Chromatogr Sci. 2005]

Sampling and analysis of butyltin compounds in air using gas chromatography and flame photometric detection. [J Occup Environ Hyg. 2004]

See all Related Articles...

4.6.4 Analysis Methods

Method 8330 provides high performance liquid chromatographic conditions for detecting explosives-related organic components. It recommends a C-18 (or equivalent) reversed-phase column with water/methanol as the mobile phase and UV detection. Minor modifications, including column and mobile phase choices, will be applied to Method 8330 for the analysis of picric acid.

4.6.5 Modification to Regulatory DQO Recommended Method

Minor modifications will be made to SW-846 preparative methods (e.g. sample size, pH for extraction) used for the liquid and solid samples. Minor modifications, column (e.g. Synergi 4 μm C12, Phenomenex) and mobile phase composition (e.g. 10 mM ammonium acetate in water and 10 mM ammonium acetate in methanol), will be made to SW-846 Method 8330 for the analysis of picric acid.

4.6.6 Experience with Analysis of Tank Waste Matrices

Picric acid is expected to exist in its anionic form, picrate, in the alkaline tank waste. Method 8330 or a modified version has not yet been used for the analysis of picric acid in tank waste.

4.6.7 Limitations

Due to the lack of experience with analyzing picric acid in tank waste matrices, limitations of analysis methods cannot be described at this time.

4.6.8 Summary

There is no experience with the analysis of picric acid in a tank waste matrix. Minor modifications to Method 8330 that include changes to the column and mobile phase composition will be used for the analysis of picric acid in tank wastes. The MDLs for picric acid will be determined on a best-effort basis. Success will be based on currently undefined criteria. Depending on the chosen method for analyses, the QC criteria listed for mass spectrometry or a chromatography technique in Tables 3-3 and 3-4 should be met.

DOE-ORP and Ecology agreed on 10 Jan 01 that analysis of picric acid would be deferred. The need for characterization work on picric acid will be reevaluated by ORP and Ecology in light of questionable stability in the tank waste matrix, the anticipated cost for analyses and that a unique method will need to be developed. No analysis of picric acid will be initiated by the contractor without further direction from ORP.

4.7 Ammonium Perfluorooctanoate and Methyl Isocyanate

The Regulatory DQO suggests Method 8330, which is a high-performance liquid chromatographic (HPLC) technique, for ammonium perfluorooctanoate and methyl isocyanate analysis. Method 8330 was developed for analyzing part per billion concentrations of explosive residues is water, soil, and sediment. The method includes a low-level salting out extraction process for preparation of the sample for injection into the chromatograph. Ammonium perfluorooctanoate and methyl isocyanate are not listed in the method's compound list. Analytes included in that method are the nitroamines and other explosives-related organic compounds. Method 8330 is discussed in more detail in Section 4.6.1.

Ammonium perfluorooctanoate does not contain a chromophore like the nitroaromatics listed in Method 8330 that makes it amenable to UV analysis at 254 nm; therefore, it can not be analyzed by Method 8330 without major modifications. It is recommended that a method that has been developed for the analysis of highly fluorinated compounds in environmental samples[a] be used for ammonium perfluorooctanoate analysis. The method uses of solid phase extraction, a sample preparation technique, with subsequent analysis using electrospray mass spectrometry in the negative ion mode with selected ion monitoring. Electrospray ionization is a liquid chromatographic interface to a mass spectrometer in which the sample solution is sprayed across a high potential difference from a needle into an orifice in the interface. Heat and gas flows are used to desolvate the ions existing in the sample solution. A mass spectrometer is used as the detector. The highly fluorinated species respond very well under negative ion conditions. These preparation and analysis methods have not been extended to the analysis of tank wastes. In order for this method to be applied to tank waste, the radioactivity levels must be reduced prior to analysis to prevent severe instrumental contamination. As a result, a cleanup method involving cation exchange may have to be performed to reduce the radioactivity levels. Because there are no equivalent SW-846 methods involving electrospray mass spectrometry, modifications to Method 8330 for the analysis of ammonium perfluorooctanoate are designated as major. Therefore, analysis of ammonia perfluorooctanoate will proceed under a future test plan that addresses method development and major modifications to SW-846 methods.

It is not recommended to use Method 8330 without major modifications for methyl isocyanate analysis. Methyl isocyanate is a volatile compound but it does not have a chromaphore, which is required for HPCL analysis. Therefore, methyl isocyanate is not amenable to analysis by Method 8330 without major modifications. An alternative extraction method that can be used with HPLC has been identified from the Occupational Safety and Health Act (OSHA) document *OSHA Index of Sampling and Analytical Methods* (OSHA 2000). Samples are collected by drawing a known volume of air through XAD-7 tubes coated with 1-(2-pyridyl) piperazine. Samples are desorbed with acetonitrile and analyzed by HPLC using fluorescence or ultraviolet detectors. Possible interferences include anhydrides, amines, alcohols, and carboxylic acids. This method of analysis is considered a major modification to Method 8330 because it deviates from the extraction method delineated by Method 8330. Method 8330 also specifies HPCL with a UV detector. Use of a fluorescence detector would be a minor modification to the method. Methyl isocyanate analysis will proceed under a future test plan that addresses method development and major modifications to SW-846 methods.

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[a] Campbell, J.A. and Y. Shi. 1999. *Analysis of Highly Perfluorinated Organics Using Negative Ion Electrospray Mass Spectrometry*. Unpublished data.

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Final Enforceable Consent Agreement and Testing Consent Order for Four Formulated Composites of Fluoropolymer Chemicals; Export Notification

[Federal Register: July 8, 2005 (Volume 70, Number 130)]
[Rules and Regulations]
[Page 39630-39637]
From the Federal Register Online via GPO Access [wais.access.gpo.gov]
[DOCID:fr08jy05-8]

ENVIRONMENTAL PROTECTION AGENCY
40 CFR Part 799
[OPPT-2003-0071; FRL-7710-5]

Final Enforceable Consent Agreement and Testing Consent Order for Four Formulated Composites of Fluoropolymer Chemicals; Export Notification

AGENCY: Environmental Protection Agency (EPA).
ACTION: Notice of final consent agreement and order.

SUMMARY: Under section 4 of the Toxic Substances Control Act (TSCA), EPA has issued a testing consent order (Order) that incorporates an enforceable consent agreement (ECA) with AGC Chemicals Americas, Inc.; Daikin America, Inc.; Dyneon, LLC; and E.I. du Pont de Nemours and Company (the Companies). The Companies have agreed to perform incineration testing of four formulated composites of fluoropolymer (FP) chemicals representative of products currently available in the marketplace. This document announces the ECA and the Order that incorporates the ECA for this testing, and summarizes the terms of the ECA. As a result of the ECA and Order that incorporates the ECA,

exporters of any of the formulated composites containing FP chemicals, including persons who do not sign the ECA, are subject to export notification requirements under section 12(b) of TSCA. This document adds the four formulated composites of FP chemicals to the table of testing consent orders for substances and mixtures without Chemical Abstract Service (CAS) Registry Numbers. Data developed from the ECA testing will contribute to the Agency's efforts to determine whether municipal and/or medical waste incineration of FPs is a potential source and/or pathway of environmental and human exposure to perfluorooctanoic acid (PFOA). The data will also contribute to the Agency's continuing efforts to achieve healthy communities and ecosystems.

DATES: The effective date of the ECA, the Order that incorporates the ECA, and this action is July 8, 2005.

ADDRESSES: EPA has established a docket for this action under docket identification (ID) number OPPT-2003-0071. All documents in the docket are listed in the EDOCKET index at http://www.epa.gov/edocket/. Although listed in the index, some information is not publicly available, i.e., Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, will not be placed on the Internet and will be publicly available only in hard copy form. Publicly available docket materials are available either electronically in EDOCKET or in hard copy at the OPPT Docket, EPA Docket Center, EPA West, Room B102, 1301 Constitution Ave., NW., Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The EPA Docket Center Reading Room telephone number is (202) 566-1744 and the telephone number for the OPPT Docket, which is located in EPA Docket Center, is (202) 566-0280.

FOR FURTHER INFORMATION CONTACT: For general information contact: Colby Lintner, Regulatory Coordinator, Environmental Assistance Division (7408M); telephone number: (202) 554-1404; e-mail address: TSCA-Hotline@epa.gov.

For information on the ECA, contact: Richard W. Leukroth, Jr., Chemical Control Division (7405M); telephone number: (202) 564-8167; fax number: (202) 564-4765; e-mail address: leukroth.rich@epa.gov.

For technical information on testing and availability of ECA test data, contact: John Blouin, Economics, Exposure and Technology Division (7406M); telephone number: (202) 564-8519; fax number: (202) 564-8528; e-mail address: blouin.john@epa.gov.

For technical information on export notification, contact: Richard W. Leukroth, Jr., Chemical Control Division (7405M); telephone number:

(202) 564-8167; fax number: (202) 564-4765; e-mail address: leukroth.rich@epa.gov or
Laura L. Bunte, Chemical Control Division (7405M); telephone number: (202) 564-8087; fax number: (202) 564-4765;
e-mail address: bunte.laura@epa.gov.

To contact any of these individuals by mail, identify the individual by name and Division indicated for that person, and use this address: Office of Pollution Prevention and Toxics (OPPT), Environmental Protection Agency, 1200

[[Page 39631]]

Pennsylvania Ave., NW., Washington, DC 20460-0001.

SUPPLEMENTARY INFORMATION:

I. General Information

A. Does this Action Apply to Me?

This action is directed to the public in general. The requirements in the ECA and the Order that incorporates the ECA only apply to those companies that are specifically named in the ECA. As of July 8, 2005, any person who exports or intends to export any of the four formulated composites of FP chemicals that are the subject of the ECA and the Order that incorporates the ECA are subject to the export notification requirements of TSCA section 12(b) (see 40 CFR part 707, subpart D, and Unit IV.B.). Although other types of entities could also be affected, most chemical manufacturers are usually identified under North American Industrial Classification System (NAICS) code 325. If you have any questions regarding the applicability of this action to a particular entity, contact persons listed under FOR FURTHER INFORMATION CONTACT.

B. How Can I Access Electronic Copies of this Document and Other Related Information?

In addition to using EDOCKET (http://www.epa.gov/edocket/), you may access this Federal Register document electronically through the EPA Internet under the ``Federal Register'' listings at http://www.epa.gov/fedrgstr/. A frequently updated electronic version of 40 CFR part 799 is available on E-CFR Beta Site Two at http://www.gpoaccess.gov/ecfr/ EXIT Disclaimer. Information on TSCA 12(b) export notification (40 CFR part 707) is available at http://www.epa.gov/oppt/chemtest/sect12b.htm.

II. Background

A. What are FP Chemicals?

FP chemicals are polymers mainly consisting of carbon and fluorine atoms, such as polytetrafluoroethylene (PTFE). Many, but not all, commercial fluoropolymers are chemicals made using ammonium perfluorooctanoate (APFO). The fluoropolymer structure is predominantly -(CF2)x- which is a potential source of PFOA. For all fluoropolymer products used in commerce, the -(CF2)- moiety is common to all polymers. The four formulated FP composites that are subject to testing under the ECA are representative of all known commercial FP chemicals and the basic chemistries are represented by the four composite test substances that are subject to testing under this ECA (i.e., dry melt fluoropolymer resin, dry nonmelt PTFE homopolymer resin/gum, dry non-melt fluoroelastomer resin/gum, aqueous fluoropolymer dispersions).

FPs possess a set of special properties that make them highly useful in the products in which they are applied. They are highly resistant to extreme temperatures, chemicals, and weather. FPs have a low friction coefficient, and the lowest dielectric constant of all plastics. They are also flame retardant, and are highly non-stick. FPs are used in a wide variety of industries, and their applications encompass a wide variety of industrial and consumer products. Among the major industrial sectors that use FPs are the automotive, chemical processing, electronics/ semiconductor, aerospace/military, medical/ pharmaceutical, building/construction, and commercial food preparation sectors. Some of the specific applications of FPs in those sectors include wire and cable insulation, O-rings and shaft seals, hoses and tubing, heat resistant/low friction metal coatings, non-stick cookware, thread sealant tape, breathable membranes for apparel, weather-resistant architectural fabric coatings, and personal care products.

B. Why Does EPA Need Environmental Effects Data on FP Chemicals?

EPA has identified potential human health concerns from exposure to PFOA and its salts. The Agency is concerned that residual APFO used to manufacture FPs is a source and/or pathway to environmental and human exposure to PFOA. In addition, there is insufficient data to determine whether FPs could degrade to PFOA by mechanisms that are not fully understood at this time. The high temperatures and retention times used during incineration processes, while destroying most of the polymer molecule, may not completely degrade these polymers. Since the strong C-F bonds are common to all FPs, EPA believes that the 17 individual FPs (see Unit III.B.) with their associated chemistries are

representative of the manner in which FPs could degrade, potentially forming PFOA when incinerated under the conditions simulating current municipal and medical waste incinerators as specified by this ECA testing program.

In September 2002, EPA's OPPT initiated a priority review of PFOA because developmental toxicity, carcinogenicity, and blood-monitoring data presented in an interim revised hazard assessment raised the possibility that PFOA might present a significant risk to human health (Ref. 1). On January 4, 2005, OPPT's Risk Assessment Division submitted a draft risk assessment of the potential human health effects associated with exposure to PFOA and its salts to EPA's Science Advisory Board's (SAB) Perfluorooctanoic Acid Risk Assessment Review Panel for peer review (Refs. 2 and 3). These assessments revealed uncertainties associated with the sources and pathways of human exposure. EPA believes that the information to be developed under the ECA testing will better inform the Agency regarding the potential source(s) and/or pathway(s) of environmental and human exposure to PFOA.

III. ECA Development and Conclusion

A. How is EPA Going to Obtain Environmental Testing on FP Chemicals?

In the Federal Register of April 16, 2003 (68 FR 18626) (FRL-7303-8), EPA initiated a public process to negotiate ECAs concerning PFOA and fluorpolymers. The two goals of the ECAs resulting from these public discussions are to develop environmental fate and transport data, as well as other data relevant to identifying the pathway(s) that result in human exposure to PFOA by air, water, or soil; and, to characterize how PFOA gets into those pathways, including the products or processes that are responsible for the presence of PFOA in the environment. EPA anticipates that the data to be developed under such ECAs will be supplemental to data being generated by ongoing testing efforts described under industry letters of intent (LOIs) (Refs. 4-7).

In preparation for the initial public meeting on June 6, 2003, EPA developed a preliminary framework document (Ref. 8) outlining Agency data needs that address the outstanding PFOA source and exposure pathway questions identified in the Federal Register notice of April 16, 2003. EPA's preliminary framework document was intended to serve as a discussion guide for the June 6, 2003, public meeting and to aid in distinguishing between outstanding EPA data needs and industry LOI commitments. The preliminary framework document was not a predetermined list of information needs defining the outcome of the ECA process.

The ECA described in this document provides for a laboratory-scale incineration testing program for four formulated composites of FP

chemicals. Incineration testing of FPs is one of the data needs identified in EPA's preliminary framework document for

[[Page 39632]]

PFOA. On June 6, 2003, the PFOA Plenary Group (consisting of EPA and all parties who had identified themselves as being interested in the ECA development proceedings after publication of the April 16, 2003 Federal Register notice) acknowledged that such a testing program was an opportunity for ECA development. The PFOA Plenary Group tasked the Fluoropolymer Technical Workgroup (a subgroup of the PFOA Plenary Group) with working out the details that could be incorporated into an ECA between the Companies and EPA.

On July 8, 2003, the Fluoropolymer Technical Workgroup received proposals from the Companies and EPA (Refs. 9 and 10) for incineration testing of FPs. Details of the testing program were then developed by members of the Fluoropolymer Incineration Subgroup (a subgroup of the Fluoropolymer Technical Workgroup) and the subgroup and workgroup reached consensus on the testing to be required under the ECA. On March 31, 2004, the Fluoropolymer Technical Workgroup acknowledged that this testing program had sufficient merit for consideration by the PFOA Plenary Group (Ref. 11). On April 1, 2004, the PFOA Plenary Group discussed the merit of this testing program and recommended that EPA consider entering into an ECA with the Companies (Ref. 12). EPA agreed and initiated steps to enter into this ECA with the Companies. On January 25, 2005, EPA received the ECA signed by the Companies, and on June 28, 2005, EPA signed the ECA and the Order that incorporates the ECA. The effective date of the ECA and the Order that incorporates the ECA is July 8, 2005.

EPA uses ECAs to accomplish testing of chemicals for health and environmental effects where a consensus exists concerning the need for and scope of testing (40 CFR 790.1(c)). The procedures for ECA negotiations and the factors for determining whether a consensus exists are described at 40 CFR 790.22 and 790.24, respectively.

B. What is the Subject of the ECA and Order Incorporating the ECA?

As specified under the ECA, four formulated composites of FP chemicals are the subject of and will be tested under the ECA and the Order that incorporates the ECA. Appendix A and Part XXIV. of the ECA (individual company signature pages) of the ECA provide details on: The rationale for formulating four composites that represent FP chemical products currently available in the marketplace, the identity of the FP chemicals used to formulate each composite, the procedures for

formulating each composite, and the procedures by which each company will contribute the FP chemical(s) for which it is obligated under the terms of the ECA. The four formulated composites are identified as: Dry Non-Melt Resin (containing: Ethene, tetrafluoro-, homopolymer, CAS No. 9002-84-0, Polytetrafluoroethylene, Document Control Number (DCN) 63040000018A, and Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5); Dry Melt Fluoropolymer Resin (containing: 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene, CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5; Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; ETFE, DCN 63040000026; and, 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene, CAS No. 35560-16-8); Dry Non-Melt Fluoroelastomer Resin/Gum (containing: 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1- difluoroethene, CAS No. 9011-17-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1- difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; 1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 54675-89-7; 1-Propene, polymer with tetrafluoroethene, CAS No. 27029-05-6; Ethene, tetrafluoro-, polymer with trifluoro(trifluoromethoxy) ethene, CAS No. 26425-79-6; Ethene, chlorotrifluoro-, polymer with 1,1-difluoroethene, CAS No. 9010-75-7; fluoroelastomer, DCN 63040000018C; fluoroelastomer DCN 63040000018D; and a low temperature fluoroelastomer, ACC 137678; and, Aqueous Fluoropolymer Dispersions (containing: Ethene, tetrafluoro-, homopolymer, CAS No. 9002-84-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3- [(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; and polytetrafluoroethylene, DCN 63040000018B).

EPA uses a variety of numerical identification systems for tracking chemicals. These include CAS numbers assigned to non-confidential chemicals, premanufacture notice (PMN) numbers assigned by EPA when chemicals enter EPA's new chemical review process, document control numbers (DCN) assigned by the EPA OPPT's Confidential Business Information Center for EPA tracking, and Accession (ACC) numbers provided by EPA when a chemical identity listed on the TSCA Inventory has been claimed as TSCA CBI. In addition, chemicals that qualify for a reporting exemption under the Polymer Exemption Rule (40 CFR 723.250) may have a commercial trade identity or an IES Method I (CAS Inventory Expert Service) name assigned.

C. What Testing Does the ECA for FP Chemicals Require?

The ECA for laboratory-scale incineration testing of four composites of FP chemicals requires environmental testing, as described in Table 1 of this unit, which sets forth the required testing, test standards, and reporting requirements for testing to be conducted under the ECA.

The testing included in the ECA will be conducted in two segments, as follows: Phase I--PFOA Transport Testing (Phase I) and Phase II--Fluorotelomer Incineration Testing (Phase II). Phase I will consist of quantitative transport efficiency testing for PFOA. At the conclusion of Phase I, the Companies will provide EPA with a letter report summarizing the results. In the event that the transport efficiency of PFOA or total fluorine is equal to or greater than 70%, testing will proceed to Phase II. In the event that the transport efficiency of PFOA and total fluorine are both individually less than 70%, the Companies will initiate a technical consultation with EPA to reach agreement on how to proceed. The various outcomes of such a technical consultation are laid out in Part VIII. of the ECA.

Under Phase II, elemental analysis, combustion stoichiometry, thermogravimetric analysis, laboratory-scale combustion testing, and, if required under the ECA (see Table 1, footnote 9 of this unit), release assessment reporting will be performed for the four composites of FP chemicals that are the subject of the ECA.

[[Page 39633]]

Table 1.--Required Testing, Test Standards, Reporting Requirements: Phases of the Testing Program for the Incineration of FP Composites

Phase I		
PFOA Transport Testing	Test standard/ Reporting requirements	Deadline \1\ (Days)
Phase I Study Plan(s)	40 CFR 790.62 (b) as annotated by Part X. of the ECA	60 \3\
Phase I Quality Assurance	EPA Requirements	90 \3\

St. Gobain Performance Plastics
Merrimack Plant
Merrimack, NH

Barr Engineering Co.
June 19, 2007

LABORATORY DATA REDUCTION

	Test 1
Run 1	10,508
Run 2	9,635
Run 3	9,839

Sample ID		Sample Fraction	APFO, ng/ml	Sample Vol., ml	Mass APFO in Sample Fraction, ng	Total For Test Run, ng
SG-A-001	Reagent Blank	DI	0	100	0	0
SG-A-002	Reagent Blank	NaOH	0	100	0	
SG-A-003	Reagent Blank	NaOH	0	100	0	
SG-A-004	Reagent Blank	Buffer	0	100	0	
SG-A-005	Field Blank	DI Impinger	0	100	0	88
SG-A-006	Field Blank	NaOH Impinger	0	100	0	
SG-A-007	Field Blank	Buffer Impinger	0	100	0	
SG-A-008	Field Blank	Train Rinse/Filter	0.876	100	87.6	
SG-A-009	T1R1	DI Impinger	26.9	100	2,690	10,508
SG-A-009*	T1R1	DI Impinger	27.7			
SG-A-010	T1R1	NaOH Impinger	14.9	100	1,490	
SG-A-011	T1R1	Buffer Impinger	2.52	100	252	
SG-A-011*	T1R1	Buffer Impinger	2.08			
SG-A-012	T1R1	Train Rinse/Filter	19.6	310	6,076	
SG-A-013	T1R2	DI Impinger	27.6	100	2760	9,635
SG-A-014	T1R2	NaOH Impinger	15.7	100	1,570	
SG-A-014*	T1R2	NaOH Impinger	16.1			
SG-A-015	T1R2	Buffer Impinger	1.95	100	195	
SG-A-016	T1R2	Train Rinse/Filter	14.6	350	5110	
SG-A-017	T2R3	DI Impinger	26.7	100	2,670	9,839
SG-A-018	T2R3	NaOH Impinger	16.2	100	1,620	
SG-A-019	T2R3	Buffer Impinger	2.45	100	245	
SG-A-020	T2R3	Train Rinse/Filter	13.6	390	5,304	
SG-A-020*	T2R3	Train Rinse/Filter	13.8			

* Duplicate results are for laboratory Quality Assurance--results are averaged for reporting

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Analytical Report

Determination of Ammonium Perfluorooctanoate (APFO) in Air Impinger and Dispersion Samples

Exygen Report No. L0011423

Testing Laboratory

Exygen Research
A division of MPI Research, Inc.
3058 Research Drive
State College, PA 16801

Requester

Tim Russell
BARR Engineering Co.
4700 West 77th Street
Minneapolis, MN 55435-4803
Phone: 952-832-2630

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1 Introduction

Results are reported for the analysis of ammonium perfluorooctanoate (APFO) in air impinger and dispersion samples received at Exygen from BARR Engineering. The Exygen project number assigned to the samples is L0011423. Table I lists the analytes quantitated for the samples.

Table I. Target Analytes

Parameter	Acronym
Ammonium Perfluorooctanoate	APFO

2 Sample Receipt

Twenty-two samples were received at Exygen. A copy of all sample log-in information is presented in Attachment C.

The samples were received on April 27, 2007. The samples were hand-delivered on wet ice by Tim Russell of BARR Engineering. The samples were stored refrigerated from time of receipt until analysis.

3 Holding Times

The maximum holding time for completion of analysis is sixty days from sample collection. All holding times were met for this data set.

4 Methods

4.1 Air Impingers

Solid phase extraction (SPE) was used to prepare the samples for LC/MS/MS analysis. A 4-mL portion of sample was diluted 10 times with water. Forty milliliters of the diluted sample was then transferred to a C_{18} SPE cartridge. The cartridge was eluted with 5 mL of 100% methanol. This treatment resulted in an eight-fold concentration of the samples prior to analysis. A portion of the extract was diluted further, transferred to autosampler vials and analyzed using electrospray LC/MS/MS.

4.2 Dispersion Samples

Solid phase extraction (SPE) was used to prepare the samples for LC/MS/MS analysis. A 100-µL portion of sample was diluted 10,000 times with water. Forty milliliters of the diluted sample was then transferred to a C_{18} SPE cartridge. The cartridge was eluted with 5 mL of 100% methanol. This treatment resulted in an eight-fold concentration of the samples prior to analysis. A portion of the extract was diluted further, transferred to autosampler vials and analyzed using electrospray LC/MS/MS.

5 Analysis

In High Pressure Liquid Chromatography (HPLC), an aliquot of extract is injected and passed through a liquid-phase chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. Following HPLC separation, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against standards.

An HP1100 system interfaced to a Micromass Quattro Ultima was used to analyze the sample extracts. Analysis details can be found associated with each set in the raw data in Attachments D and E.

5.1 Calibration

A 7-point calibration curve was analyzed throughout the analytical sequence for PFOA. The calibration points were prepared at 0, 25, 50, 100, 250, 500, and 1000 ng/L (ppt) for LC/MS/MS analysis. Calibration standards are prepared using the same SPE procedure used for samples. The instrument response versus the concentration was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r^2 \geq 0.985$.

For the results reported here, calibration criteria were met. The calibration curves are included in the raw data in Attachments D and E.

5.2 Surrogates

^{13}C labeled-perfluorooctanoic acid (^{13}C PFOA) is used as a surrogate.

For air impinger samples, ^{13}C PFOA was added into the sampling bottle before the bottle was shipped to the field. In the field, the sample was poured into the pre-spiked bottle.

For dispersion samples, ^{13}C PFOA was added to the samples in the laboratory before extraction.

^{13}C PFOA recoveries can be found in Attachment B.

5.3 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared by adding a known concentration of the analyte to reagent methanol. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range.

6 Data Summary

Please see Attachment A for a detailed listing of the analytical results. Results are reported in parts per billion (ng/mL) for the analyte for air impinger samples on an as-received basis Results are reported in parts per million (µg/mL) for the analyte for dispersion samples on an as-received basis.

7 Attachments

7.1 Attachment A: Results

7.2 Attachment B: Quality Control Data

7.3 Attachment C: Chain of Custody

7.4 Attachment D: Air Impinger Analytical Data

7.5 Attachment E: Dispersion Analytical Data

8 Signatures

Karen Risha, Manager, Analytical — Date 05/25/07

Kevin Lloyd, General Manager, Analytical Sciences — Date 25-May-07

7.1 Attachment A: Results

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Analytical Report

Summary of APFO in Impinger Samples

Sample ID	Analyte Found (ng/mL) APFO	Sample Volume (mL)
SG-A-001	ND	100
SG-A-002	ND	100
SG-A-003	ND	100
SG-A-004	ND	100
SG-A-005	ND	100
SG-A-006	ND	100
SG-A-007	ND	100
SG-A-008	0.876	100
SG-A-009	26.9	100
SG-A-009*	27.7	100
SG-A-010	14.9	100
SG-A-011	2.52	100
SG-A-011*	2.08	100
SG-A-012	19.6	310
SG-A-013	27.6	100
SG-A-014	15.7	100
SG-A-014*	16.1	100
SG-A-015	1.95	100
SG-A-016	14.6	350
SG-A-017	26.7	100
SG-A-018	16.2	100
SG-A-019	2.45	100
SG-A-020	13.6	390
SG-A-020*	13.8	390

ND = Not Detected. Response between 0 and 0.5 ng/mL.
*Laboratory Duplicate

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Summary of APFO in Dispersion Samples

Sample ID	Analyte Found (µg/mL) APFO	Sample Volume (mL)
SG-L-001	8.46	225
SG-L-001*	8.86	225
SG-L-002	8.69	235

*Laboratory Duplicate

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7.2 Attachment B: Quality Control Data

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Recovery Summary for PFOA in Impinger Samples

Sample ID	PFOA Added (ng/mL)	PFOA Found in Sample (ng/mL)	PFOA Found in Spiked Sample (ng/mL)	Recovery (%)
SG-A-009 Spk C	50.0	25.8	71.1	91
SG-A-011 Spk C	5.00	2.42	7.95	111
SG-A-014 Spk C	50.0	15.1	59.7	89
SG-A-020 Spk C	50.0	13.1	64.6	103

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Recovery Summary for ^{13}C-PFOA in Impinger Samples

Sample ID	Analyte Added (ng/mL) ^{13}C-PFOA	Analyte Found (ng/mL) ^{13}C-PFOA	Recovery (%)
SG-A-001	5.00	6.40	128
SG-A-002	5.00	4.69	94
SG-A-003	5.00	4.48	90
SG-A-004	5.00	4.49	90
SG-A-005	5.00	5.32	106
SG-A-006	5.00	4.74	95
SG-A-007	5.00	4.39	88
SG-A-008	5.00	4.22	84
SG-A-009	5.00	5.14	103
SG-A-009*	5.00	4.82	96
SG-A-010	5.00	4.61	92
SG-A-011	5.00	5.10	102
SG-A-011*	5.00	4.28	86
SG-A-012	1.61	1.49	93
SG-A-013	5.00	5.55	111
SG-A-014	5.00	4.15	83
SG-A-014	5.00	4.07	81
SG-A-015	5.00	4.62	92
SG-A-016	1.43	1.33	93
SG-A-017	5.00	5.06	101
SG-A-018	5.00	4.51	90
SG-A-019	5.00	5.03	101
SG-A-020	1.28	1.23	96
SG-A-020*	1.28	1.26	98

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Recovery Summary for PFOA in Dispersion Samples

Sample ID	PFOA Added (µg/mL)	PFOA Found in Sample (µg/mL)	PFOA Found in Spiked Sample (µg/mL)	Recovery (%)
SG-L-001 Spk C	10	8.13	19.9	118

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Recovery Summary for ^{13}C-PFOA in Dispersion Samples

Sample ID	Analyte Added (µg/mL) ^{13}C-PFOA	Analyte Found (µg/mL) ^{13}C-PFOA	Recovery (%)
SG-L-001 Spk C	10	9.82	98
SG-L-001	5.0	5.07	101
SG-L-001*	5.0	5.05	101
SG-L-002	5.0	4.98	100

*Laboratory Duplicate

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7.3 Attachment C: Chain of Custody

MPI RESEARCH

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State College, PA 16801

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

Login

Login Group: L0011423

Login #: 11534
Project: P0003133
Company Name: Barr Engineering
Submitted By: Tim Russell
Login Type: Immediate Receipt of Samples
Started: True
Date Start: 04/27/2007
Due Date: 05/07/2007
Login Initiated*: 04/27/2007

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

* Dates entered into "Login Initiated" field prior to 1/5/06 reflect dates of receipt. The field was formerly called "Received Date"

Received By: Ammerman, Mark
Spread Sample:
Label:
Exygen SD/PI: Risha, Karen
Project Title/Type: Analysis of Samples for PFOA/APFO by LCMSMS / ROUTINE
Login Notes:

Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By
PK0013603		Received Date: 4/27/07 9:00 Package & Contents Uncompromised		Wet Ice 2.0	RECEIVED Ammerman, Mark

Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0250357	139.30 g		500 ml Clear Plastic Narrow	NONE		
C0250358	151.60 g		500 ml Clear Plastic Narrow	NONE		
C0250359	158.10 g		500 ml Clear Plastic Narrow	NONE		
C0250360	121.40 g		500 ml Clear Plastic Narrow	NONE		
C0250361	155.20 g		500 ml Clear Plastic Narrow	NONE		
C0250362	156.10 g		500 ml Clear Plastic Narrow	NONE		
C0250363	156.60 g		500 ml Clear Plastic Narrow	NONE		
C0250364	129.50 g		500 ml Clear Plastic Narrow	NONE		
C0250365	158.00 g		500 ml Clear Plastic Narrow	NONE		
C0250366	158.40 g		500 ml Clear Plastic Narrow	NONE		
C0250367	156.70 g		500 ml Clear Plastic Narrow	NONE		
C0250368	305.40 g		500 ml Clear Plastic Narrow	NONE		
C 369	154.80 g		500 ml Clear Plastic Narrow	NONE		

Login

Login Group: L0011423

Login #:	11534	Conform COC Sample:	True
Project:	P0003133	Conform COC:	True
Company Name:	Barr Engineering	Conform Sample:	True
Submitted By:	Tim Russell	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	04/27/2007		
Due Date:	05/07/2007		
Login Initiated*:	04/27/2007		

* Dates entered into "Login Initiated" field prior to 1/5/06 reflect dates of receipt. The field was formerly called "Received Date"

Received By: Ammerman, Mark
Spread Sample:
Label:
Exygen SD/PI: Risha, Karen
Project Title/Type: Analysis of Samples for PFOA/APFO by LCMSMS / ROUTINE
Login Notes:

Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By
PK0013603		Received Date: 4/27/07 9:00 Package & Contents Uncompromised		Wet Ice 2.0	RECEIVED Ammerman, Mark

Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0250370	160.40 g		500 ml Clear Plastic Narrow	NONE		
C0250371	158.10 g		500 ml Clear Plastic Narrow	NONE		
C0250372	341.10 g		500 ml Clear Plastic Narrow	NONE		
C0250373	156.40 g		500 ml Clear Plastic Narrow	NONE		
C0250374	160.00 g		500 ml Clear Plastic Narrow	NONE		
C0250375	155.90 g		500 ml Clear Plastic Narrow	NONE		
C0250376	368.30 g		500 ml Clear Plastic Narrow	NONE		
C0250377	380.10 g		500 ml Clear Plastic Narrow	NONE		
C0250378	387.30 g		500 ml Clear Plastic Narrow	NONE		

Login

Samples

Sample ID	Container	Matrix	Fraction	Sample	Date Sampled	Date Due
L0011423-0001	C0250357	LIQUID	Impinger solution	SG-A-001	04/26/2007	05/07/2007
L0011423-0002	C0250358	LIQUID	Impinger solution	SG-A-002	04/26/2007	05/07/2007
L0011423-0003	C0250359	LIQUID	Impinger solution	SG-A-003	04/26/2007	05/07/2007
L0011423-0004	C0250360	LIQUID	Impinger solution	SG-A-004	04/26/2007	05/07/2007
L0011423-0005	C0250361	LIQUID	Impinger solution	SG-A-005	04/26/2007	05/07/2007
L0011423-0006	C0250362	LIQUID	Impinger solution	SG-A-006	04/26/2007	05/07/2007
L0011423-0007	C0250363	LIQUID	Impinger solution	SG-A-007	04/26/2007	05/07/2007
L0011423-0008	C0250364	LIQUID	Impinger solution	SG-A-008	04/26/2007	05/07/2007
L0011423-0009	C0250365	LIQUID	Impinger solution	SG-A-009	04/26/2007	05/07/2007
L0011423-0010	C0250366	LIQUID	Impinger solution	SG-A-010	04/26/2007	05/07/2007
L0011423-0011	C0250367	LIQUID	Impinger solution	SG-A-011	04/26/2007	05/07/2007
L0011423-0012	C0250368	LIQUID	Impinger solution	SG-A-012	04/26/2007	05/07/2007
L0011423-0013	C0250369	LIQUID	Impinger solution	SG-A-013	04/26/2007	05/07/2007
L0011423-0014	C0250370	LIQUID	Impinger solution	SG-A-014	04/26/2007	05/07/2007
L0011423-0015	C0250371	LIQUID	Impinger solution	SG-A-015	04/26/2007	05/07/2007
L0011423-0016	C0250372	LIQUID	Impinger solution	SG-A-016	04/26/2007	05/07/2007

L0011423-0017	C0250373	LIQUID	Impinger solution	SG-A-017	04/26/2007	05/07/2007
L0011423-0018	C0250374	LIQUID	Impinger solution	SG-A-018	04/26/2007	05/07/2007
L0011423-0019	C0250375	LIQUID	Impinger solution	SG-A-019	04/26/2007	05/07/2007
L0011423-0020	C0250376	LIQUID	Impinger solution	SG-A-020	04/26/2007	05/07/2007
L0011423-0021	C0250377	LIQUID	Dispersion	SG-L-001	04/26/2007	05/07/2007
L0011423-0022	C0250378	LIQUID	Dispersion	SG-L-002	04/26/2007	05/07/2007

Login Reviewed By: Brian Stillman Date/Time: 4.30.07 945

Request for Laboratory Analytical Services

Page 1 of 3
№ 11803

Report Results To

Check One:

☐ *Barr Engineering Company*
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

☒ *Barr Engineering Company*
7390 Ohms Lane
Edina, MN 55439-2330
(952) 832-2600

Attention: Richard Berg (Print Name) 952-832-2787 (Direct Phone No.)

Send Invoice To

Project Number 29/06-004.001.004

Barr Engineering Company
4700 West 77th Street
Minneapolis, MN 55435-4803
Ph. (952) 832-2600 Fax (952) 832-2601

Attention: ACCOUNTS PAYABLE (Print Name)

Special instructions and/or specific regulatory requirements:
(method, limit of detection, etc.)

STANDARD TURNAROUND (10 working DAYS)
Include sample results, Case narrative, QC summary, calibration summary, RAW DATA

Sample Identification	Date/Time Collected	~~Media~~ I.D. #	Type: Grab	Type: Comp.	Type: QC	~~EPA Method 5~~	~~EPA Method 202~~	APFO LC/MS/MS				D.I. Impinger	NaOH Impinger	Buffer Impinger	MeOH/Filter	Filter	Acetone Rinse	Wet Catch	MeCl2 Rinse	Total No. of Containers	Remarks
1. SG-A-001	4-26-07/0800	TIRO			X			X				X								3	Reagent Blanks
2. SG-A-002	/	TIRO			X			X					X							3	
3. SG-A-003	/	TIRO			X			X						X						3	
4. SG-A-004	/	TIRO			X			X							X					3	
5. SG-A-005	/0900	TIR00			X			X				X								3	Field Blanks
6. SG-A-006	/	TIR00			X			X					X							3	
7. SG-A-007	/	TIR00			X			X						X						3	
8. SG-A-008	/	TIR00			X			X							X					3	
9. SG-A-009	/1200	TIR1		X				X				X								1	
10. SG-A-010	/1200	TIR1		X				X					X							1	

Custody

Collected by (Print Name): Tim Russell / Chris Lee

Relinquished by: Tim Russell — Date/Time 4-27-07/0900

Relinquished by: — Date/Time

Method of Shipment: ☒ Sampler ☐ FedEx ☐ UPS Other: _____

Collectors Signature: _____ Date: 4-26-07

LABORATORY: Exygen Research

Received by: _____ Date/Time 04/27/07 09a

Received by: — Date/Time

Received at Lab by: — Date/Time

Sample Condition upon Receipt: ☐ Acceptable ☐ Other (explain)

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy

Rev. 03/01

BARR

Request for Laboratory Analytical Services

№ 11804

Report Results To

Check One:

☐ *Barr Engineering Company*
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

☒ *Barr Engineering Company*
7390 Ohms Lane
Edina, MN 55439-2330
(952) 832-2600

Attention: Richard Berg (Print Name) 952-832-2787 (Direct Phone No.)

Send Invoice To

Project Number 29/06-004.001.004

Barr Engineering Company
4700 West 77th Street
Minneapolis, MN 55435-4803
Ph. (952) 832-2600 Fax (952) 832-2601

Attention: Accounts Payable (Print Name)

Special instructions and/or specific regulatory requirements:
(method, limit of detection, etc.)

STANDARD TURNAROUND (10 working DAYS)
Include sample results, case narrative, QC summary, calibration summary, RAW DATA

Sample Identification	Date/Time Collected	~~Media~~ I.D. #	Type: Grab	Type: Comp.	Type: QC	EPA ~~Method~~ 5	EPA ~~Method~~ 202	APTO LC/MS/MS	D.I. Impinger	NaOH Impinger	Buffer Impinger	MeOH/Filter	Filter	Acetone Rinse	Wet Catch	MEC Rinse	Total No. of Containers	Remarks
1. SG-A-01ϕ (011)	4-26-07/1200	TIR1		X				X										1	
2. SG-A-012	/1200	TIR1		X				X										1	
3. SG-A-013	/1330	TIR2		X				X			X							1	
4. SG-A-014	/	TIR2		X				X				X						1	
5. SG-A-015	/	TIR2		X				X										1	
6. SG-A-016	/	TIR2		X				X										1	
7. SG-A-017	/1500	TIR3		X				X			X	X						1	
8. SG-A-018	/	TIR3		X				X				X						1	
9. SG-A-019	/	TIR3		X				X										1	
10. SG-A-020	/	TIR3		X				X										1	

Chain of Custody

Collected by (Print Name): Tim Russell / Chris Lee

Relinquished by: Tim Russell — Date/Time 4-27-07/0900

Relinquished by: — Date/Time

Method of Shipment: ☒ Sampler ☐ FedEx ☐ UPS Other: _____

Collectors Signature: _____ Date: 4-26-07

LABORATORY: Exxyen Research

Received by: _____ Date/Time 4/27/07 09a

Received by: — Date/Time

Received at Lab by: — Date/Time

Sample Condition upon Receipt: ☐ Acceptable ☐ Other (explain)

P:\ZAIRSAMPLING\DATASHEETS\OTHER\CHAINOFCUSTODY(EXAMPLE2).CDR 03-08-01

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy

Rev. 03/01

ARR

Request for Laboratory Analytical Services

PAI 3 of 3
№ 11805

Results To

Check One:

☐ ***Barr Engineering Company***
4700 West 77th Street
Minneapolis, MN 55435-4803
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Attention: Richard Berg (Print Name) 952-832-2787 (Direct Phone No.)

Send Invoice To

Project Number 29/06-004.001.004

Barr Engineering Company
4700 West 77th Street
Minneapolis, MN 55435-4803
Ph. (952) 832-2600 Fax (952) 832-2601

Attention: Accounts PAYABLE (Print Name)

Special instructions and/or specific regulatory requirements:
(method, limit of detection, etc.)

See Pages 1 & 2 of 3

Sample Identification	Date/Time Collected	Media I.D. #	Type: Grab	Type: Comp.	Type: QC	EPA Method 5	~~EPA Method 202~~	APFO LL/MS/MS	Dispersion	~~Filter~~	~~Acetone Rinse~~	~~Wet Catch~~	~~MEOH Rinse~~	Total No. of Containers	Remarks
1. SG-L-001	4.26.07 / 1415						X	X	X					1	
2. SG-L-002	↓ / ↓						X	X	X					1	
3.															
4.															
5.															
6.															
7.															
8.															
9.															
10.															

Custody

Collected by (Print Name): Tim Russell

Relinquished by: Tim Russell — Date/Time 4-27-07 / 0900

Relinquished by: — Date/Time

Method of Shipment: ☒ Sampler ☐ FedEx ☐ UPS Other: _____

Collectors Signature: [signature] Date: 4-26-07

LABORATORY: Oxygen Research

Received by: [signature] — Date/Time 04/27/07 0900

Received by: — Date/Time

Received at Lab by: — Date/Time

Sample Condition upon Receipt: ☐ Acceptable ☐ Other (explain)

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy

Rev. 03/01

7.4 Attachment D: Air Impinger Analytical Data

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050407E
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	Water	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	4.0 mL	Extraction Date:	5/4/2007
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	5/6/2007

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050407E-501	0	-	-	0	-	-	-	-
XC050107-1	-	CS	050407E-502	25	-	-	2883	-	-	-	-
XC050107-2	-	CS	050407E-503	50	-	-	5111	-	-	-	-
XC050107-3	-	CS	050407E-504	100	-	-	10568	-	-	-	-
XC050107-4	-	CS	050407E-505	250	-	-	25389	-	-	-	-
XC050107-5	-	CS	050407E-506	500	-	-	49545	-	-	-	-
XC050107-6	-	CS	050407E-507	1000	-	-	96470	-	-	-	-
Methanol Wash	-	C	050407E-508	-	-	-	1217	-	-	-	-
Reagent Control	NA	C	050407E-509	-	-	1	0	ND	ND	-	-
Reagent Spk A	NA	LCS	050407E-510	-	-	1	5104	48.5	50.4	50	97
Reagent Spk B	NA	LCS	050407E-511	-	-	1	49471	517	538	500	103
L11423-9 Spk C	SG-A-009	LF	050407E-512	-	100	1000	7245	71100	74000	50000	91
L11423-9 Spk C	SG-A-009	LF	050407E-513	-	100	10	663384	*	-	50000	-
Methanol Wash	-	C	050407E-514	-	-	-	1064	-	-	-	-
XC050107-1	-	CS	050407E-515	25	-	-	2607	-	-	-	-
XC050107-2	-	CS	050407E-516	50	-	-	5210	-	-	-	-
L11423-1	SG-A-001	S	050407E-517	-	100	10	106	ND	ND	-	-
L11423-5	SG-A-005	S	050407E-518	-	100	10	301	ND	ND	-	-
L11423-9	SG-A-009	S	050407E-519	-	100	100	24895	25800	26900	-	-
L11423-9 Rep	SG-A-009	S	050407E-520	-	100	100	25691	26600	27700	-	-
L11423-9	SG-A-009	S	050407E-521	-	100	10	271981	*	-	-	-
L11423-9 Rep	SG-A-009	S	050407E-522	-	100	10	246368	*	-	-	-
L11423-13	SG-A-013	S	050407E-523	-	100	10	264544	**	-	-	-
L11423-17	SG-A-017	S	050407E-524	-	100	10	238032	**	-	-	-
XC050107-3	-	CS	050407E-525	100	-	-	10540	-	-	-	-
XC050107-4	-	CS	050407E-526	250	-	-	23863	-	-	-	-
XC050107-5	-	CS	050407E-527	500	-	-	47488	-	-	-	-
XC050107-6	-	CS	050407E-528	1000	-	-	90944	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 517.909
Slope = 94.6499
Coef. Of Det. = 0.997985

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.
**Sample requires dilution. See data set 050407ER.

Spreadsheet prepared by: MLC / 5/10/07

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050407E
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	^{13}C-PFOA	Matrix:	Water	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	415 -> 370	Sample Volume Extracted:	4.0 mL	Extraction Date:	05/04/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/06/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	13C-PFOA Found (ng/L)	Amount 13C-PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050407E-501	0	-	-	0	-	-	-
XC050107-1	-	CS	050407E-502	25	-	-	2629	-	-	-
XC050107-2	-	CS	050407E-503	50	-	-	4544	-	-	-
XC050107-3	-	CS	050407E-504	100	-	-	9588	-	-	-
XC050107-4	-	CS	050407E-505	250	-	-	24191	-	-	-
XC050107-5	-	CS	050407E-506	500	-	-	48270	-	-	-
XC050107-6	-	CS	050407E-507	1000	-	-	92849	-	-	-
Methanol Wash	-	C	050407E-508	-	-	-	0	-	-	-
Reagent Control	NA	C	050407E-509	-	-	1	0	ND	-	-
Reagent Spk A	NA	LCS	050407E-510	-	-	1	5003	51.3	50	103
Reagent Spk B	NA	LCS	050407E-511	-	-	1	49132	530	500	106
L11423-9 Spk C	SG-A-009	LF	050407E-512	-	100	1000	418	*	5000	-
L11423-9 Spk C	SG-A-009	LF	050407E-513	-	100	10	42399	4570	5000	91
Methanol Wash	-	C	050407E-514	-	-	-	0	-	-	-
XC050107-1	-	CS	050407E-515	25	-	-	2458	-	-	-
XC050107-2	-	CS	050407E-516	50	-	-	4896	-	-	-
L11423-1	SG-A-001	S	050407E-517	-	100	10	59224	6400	5000	128
L11423-5	SG-A-005	S	050407E-518	-	100	10	49285	5320	5000	106
L11423-9	SG-A-009	S	050407E-519	-	100	100	4503	*	5000	-
L11423-9 Rep	SG-A-009	S	050407E-520	-	100	100	4779	*	5000	-
L11423-9	SG-A-009	S	050407E-521	-	100	10	47648	5140	5000	103
L11423-9 Rep	SG-A-009	S	050407E-522	-	100	10	44653	4820	5000	96
L11423-13	SG-A-013	S	050407E-523	-	100	10	51389	5550	5000	111
L11423-17	SG-A-017	S	050407E-524	-	100	10	46879	5060	5000	101
XC050107-3	-	CS	050407E-525	100	-	-	10015	-	-	-
XC050107-4	-	CS	050407E-526	250	-	-	23043	-	-	-
XC050107-5	-	CS	050407E-527	500	-	-	45495	-	-	-
XC050107-6	-	CS	050407E-528	1000	-	-	90171	-	-	-

13C-PFOA Found (ng/L) = (peak area - intercept) / slope x DF

Recovery (%) = $\frac{\text{13C-PFOA found (ng/L)}}{\text{amount 13C-PFOA added (ng/L)}} \times 100$

Standard Curve : Linear (1/x weighted)
Intercept = 269.952
Slope = 92.1846
Coef. Of Det. = 0.999069

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.

Spreadsheet prepared by: MLC / 5/10/07

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

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423
Sponsor Study/Protocol No: NA
Matrix: DI Water Impinger Sample
The samples listed below were removed from refrigerator No. 34
Time 8:16 AM Date 5/4/2007 Initials SDD

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-A-009	L11423-9* Spk C	100 mL	40.0	200.0
SG-A-001	L11423-1*	100 mL	40.0	-
SG-A-005	L11423-5*	100 mL	40.0	-
SG-A-009	L11423-9*	100 mL	40.0	-
SG-A-009	L11423-9 Rep*	100 mL	40.0	-
SG-A-013	L11423-13*	100 mL	40.0	-
SG-A-017	L11423-17*	100 mL	40.0	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
L11423-9* Spk C	SS20796 (1000 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-

All samples were measured:
Time 10:10 AM Date 5/4/2007 Initials SDD

After measuring samples were returned to refrigerator No. 34
Time 5:30 PM Date 5/4/2007 Initials SDD

Comments: *Sample is pre-diluted ten times by taking 4 mL of sample and bringing the volume up to 40 mL with HPLC water

Analysis Summary:
Data Set: 050407E Initials/Date: MLC / 5/4/07
Data Set: 050407ER Initials/Date: MLC / 5/10/07
Data Set: - Initials/Date: - / -

Set extraction/analysis data verified by: CEE Date: 05/23/07

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

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007C[$$] PROTOCOL NUMBER: NA

MATRIX: DI Water Impinger Sample ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	Reagent Control			-		-	-	Methanol	46314
na	Reagent Spk A			-		-	-	C18 SPE	005837081B
na	Reagent Spk B			-		-	-	HPLC Water	46335
SG-A-009	L11423-9* Spk C			0.1/10		-	-	-	-
SG-A-001	L11423-1*			-		-	-	-	-
SG-A-005	L11423-5*			-		-	-	-	-
SG-A-009	L11423-9*			0.1/1.6		-	-	-	-
SG-A-009	L11423-9 Rep*			0.1/1.0		-	-	-	-
SG-A-013	L11423-13*			-		0.1/1.0		-	-
SG-A-017	L11423-17*	↓	↓	-	↓	0.1/1.0	↓	*Initials/Date*	EDO 5/4/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C44404
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date	-	EDO 5/4/07	EDO 5/4/07	MLC 5/4/07	MLC 5/4/07	MLC 5/10/07	MLC 5/10/07	*Initials/Date*	MLC 5/4/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

*Sample was diluted ten times prior to extraction.
**Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:

Final extracts stored in refrigerator 32 Initials: EDO Date: 5/4/2007

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

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Exygen STUDY NO: L11423

Protocol No.: None
Method No.: $$
Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA
Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC050107-0	Methanol	46303
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC050107-1	C18 SPE	005337081B
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC050107-2	Water	46335
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC050107-3	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC050107-4	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC050107-5	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC050107-6	-	-
-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/01/07

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) freezer # 34 Time: 9:10 AM — Initials/Date: EDD 1 5/01/07

40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD 1 5/01/07

After measuring, samples returned to (refrigerator) freezer # 34 Time: 11:10 AM — Initials/Date: EDD 1 5/01/07

Samples fortified: Initials/Date: AMB 1 5/01/07

SPE clean-up: Initials/Date: EDD 1 5/01/07

Final volume adjusted to 5 mL: Initials/Date: EDD 1 5/01/07

Extracts placed in refrigerator # 32 Initials/Date: EDD 1 5/01/07

Comments:

STANDARD EXPIRATION DATE: 5/15/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/10/07

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050407E DI Water.SPL
Printed: Fri May 04 15:22:11 2007 MLC 5/4/07

Page Position: (1, 1)

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/mL)	Conc B	Conc C	Test ID	DF
1	94	050407E-501	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---	0	1
2	95	050407E-502	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
3	96	050407E-503	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
4	97	050407E-504	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
5	98	050407E-505	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
6	99	050407E-506	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
7	100	050407E-507	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1
8	92	050407E-508	---	---	Methanol Wash	---	Blank	---	---	---	0	1
9	73	050407E-509	---	---	Reagent Control	---	Blank	---	---	---	0	1
10	74	050407E-510	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---	0	1
11	75	050407E-511	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500	---	0	1
12	76	050407E-512	---	---	L11423-9 Spk C, 50,000 ng/L, DF=1,000	---	QC	50000	5000	---	0	1000
13	77	050407E-513	---	---	L11423-9 Spk C, 50,000 ng/L, DF=10	---	QC	50000	5000	---	0	10
14	92	050407E-514	---	---	Methanol Wash	---	Blank	---	---	---	0	1
15	95	050407E-515	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
16	96	050407E-516	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
17	78	050407E-517	---	---	L11423-1, DF=10	---	Analyte	---	5000	---	0	10
18	79	050407E-518	---	---	L11423-5, DF=10	---	Analyte	---	5000	---	0	10
19	80	050407E-519	---	---	L11423-9, DF=100	---	Analyte	---	5000	---	0	100
20	81	050407E-520	---	---	L11423-9 Rep, DF=100	---	Analyte	---	5000	---	0	100
21	82	050407E-521	---	---	L11423-9, DF=10	---	Analyte	---	5000	---	0	10
22	83	050407E-522	---	---	L11423-9 Rep, DF=10	---	Analyte	---	5000	---	0	10
23	84	050407E-523	---	---	L11423-13, DF=10	---	Analyte	---	5000	---	0	10
24	85	050407E-524	---	---	L11423-17, DF=10	---	Analyte	---	5000	---	0	10
25	97	050407E-525	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
26	98	050407E-526	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
27	99	050407E-527	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
28	100	050407E-528	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1

	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
20	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
21	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
22	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
23	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
24	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
25	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
26	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
27	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
28	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA

Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/4/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/10/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Fri May 04 15:23:11 2007 MLC 5/4/07

Solvent Delay (mins) : 0.00

Function : 1 MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Friday, May 04, 2007 14:57:57

Printed: Friday, May 04, 2007 15:23:15 MLC 5/4/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

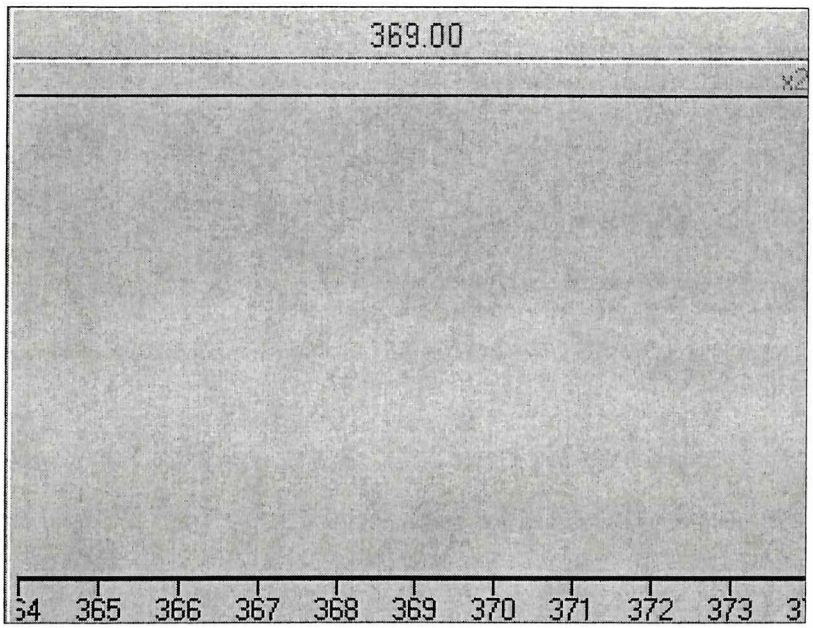
HP1100 Autosampler Initial Conditions

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	77

Tuning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Fri May 04 15:23:29 2007 MLC 5/4/07



Dau 413.00

SOURCE (ESP-)	Set	Rdbk	Analyser	Set	Rdbk
Capillary	3.00	-2.94	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	-696

Pressures	Rdbk	Gas Flows	Rdbk
Analyser Vacuum	OFF	Cone Gas	89.7
Gas Cell	2.3e-3	Desolvation	682.4

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050407E
Extraction Date: 05/04/07
Analyzed on: 05/06/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2883
XC050107-2	50	5111
XC050107-3	100	10568
XC050107-4	250	25389
XC050107-5	500	49545
XC050107-6	1000	96470

Coefficient of Determination: 0.999793219

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999896604
R Square	0.999793219
Adjusted R Square	0.999741524
Standard Error	581.3358145
Observations	6

MLC
5/10/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	6536020549	6536020549	19340.12381	1.60355E-08
Residual	4	1351805.317	337951.3292		
Total	5	6537372354			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	835.284243	324.7422376	2.572145371	0.061838379	-66.3447528	1736.913239	-66.3447528	1736.913239
X Variable 1	96.08015301	0.690882291	139.0687737	1.60355E-08	94.16195625	97.99834976	94.16195625	97.99834976

Initial Calibration Curve for ^{13}C-PFOA

Exygen Study No.: L11423
Set No: 050407E
Extraction Date: 05/04/07
Analyzed on: 05/06/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2629
XC050107-2	50	4544
XC050107-3	100	9588
XC050107-4	250	24191
XC050107-5	500	48270
XC050107-6	1000	92849

Coefficient of Determination: 0.999562742

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999781347
R Square	0.999562742
Adjusted R Square	0.999453428
Standard Error	818.6379256
Observations	6

MLC
5/10/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	6127967991	6127967991	9143.92735	7.17083E-08
Residual	4	2680672.213	670168.0532		
Total	5	6130648663			

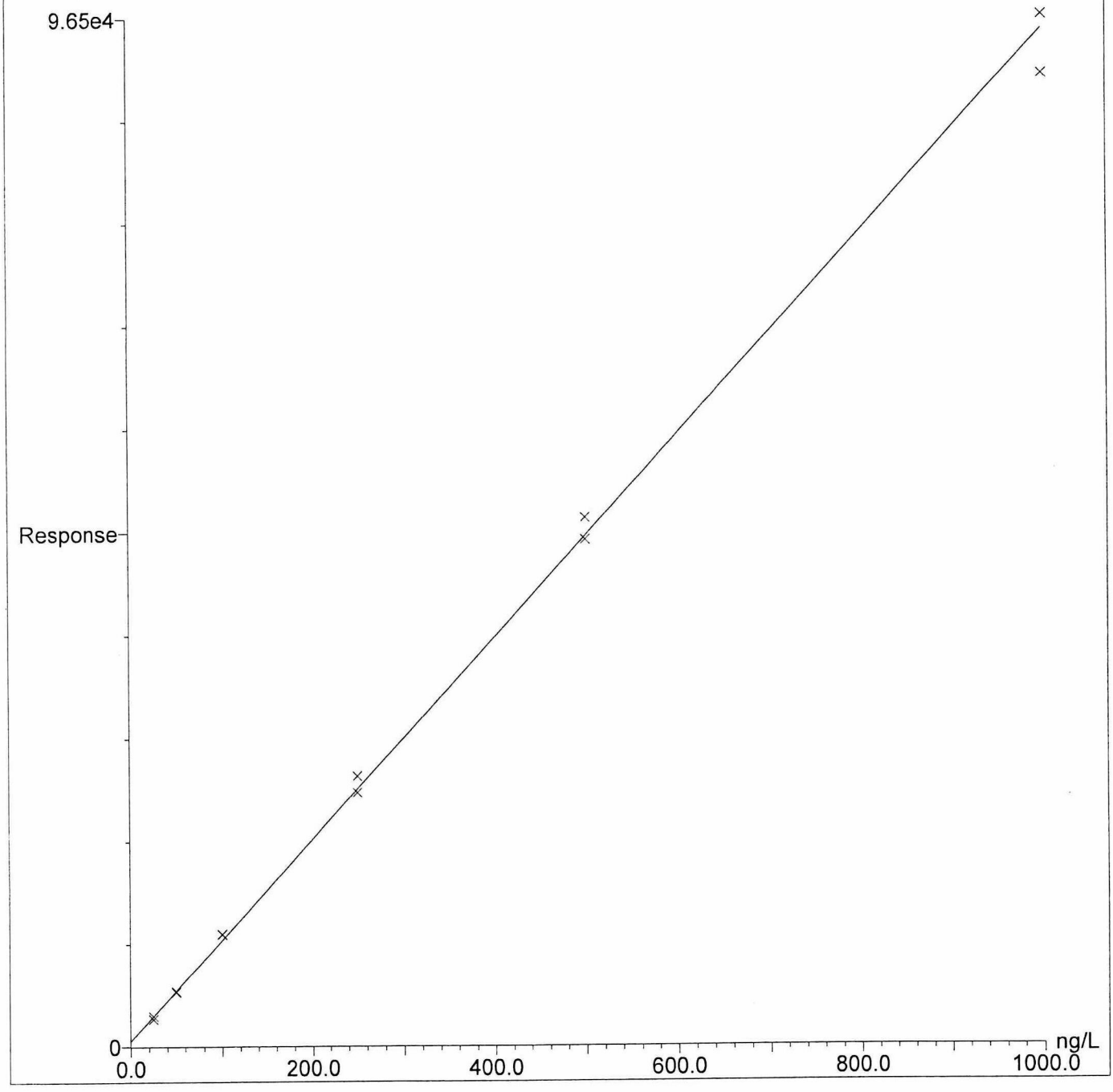
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	497.2028836	457.3024835	1.087251659	0.338055181	-772.4723582	1766.878125	-772.4723582	1766.878125
X Variable 1	93.03261439	0.972901431	95.62388483	7.17083E-08	90.33140697	95.7338218	90.33140697	95.7338218

Quantify Calibration Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050407E DI Water
Last modified: Thu May 10 12:47:08 2007
Printed: Thu May 10 12:48:23 2007

MLC 5/10/07

Compound 1 name: PFOA
Coefficient of Determination: 0.997985
Calibration curve: 94.6499 * x + 517.909
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

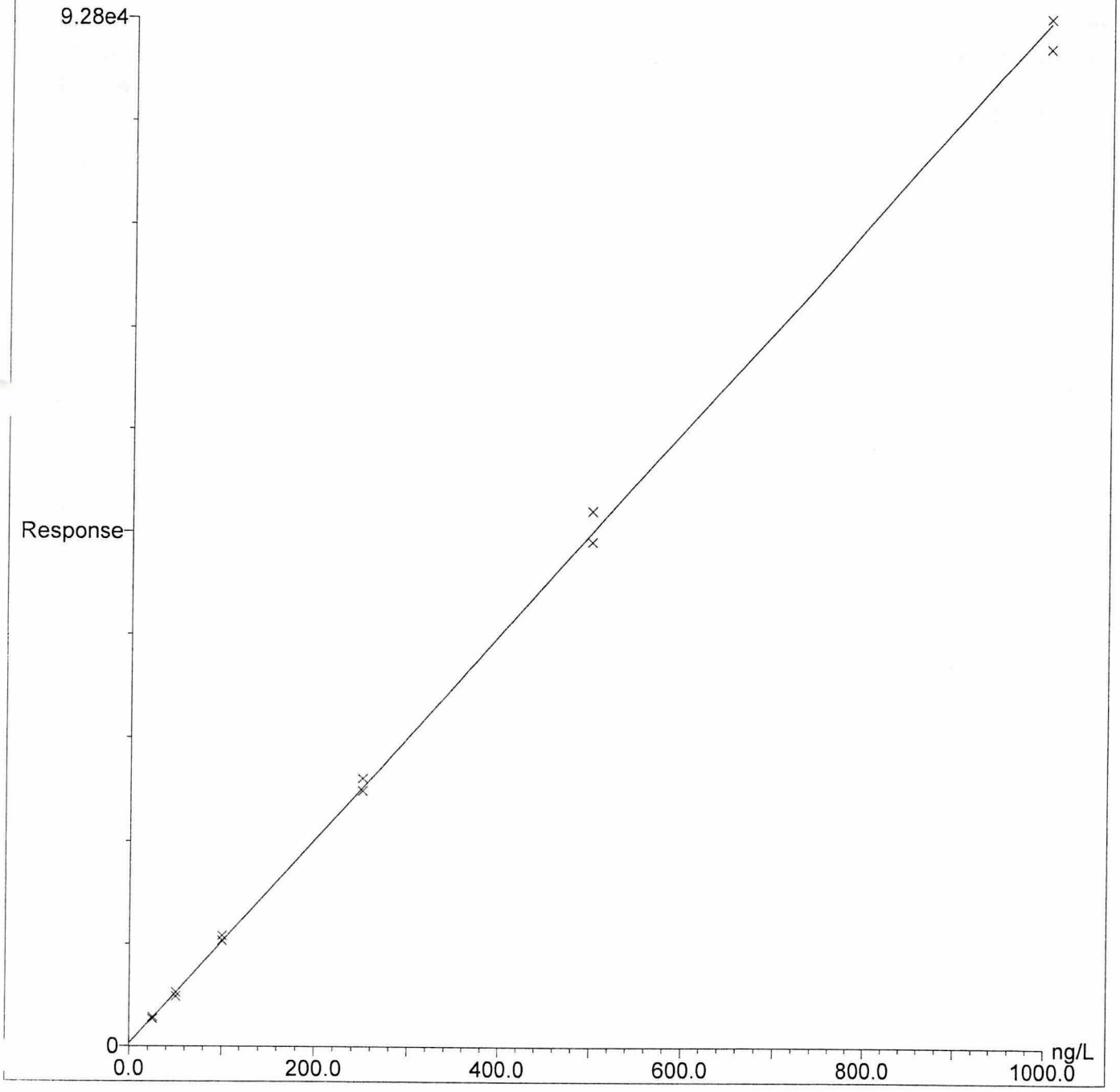


Quantify Calibration Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050407E DI Water
Last modified: Thu May 10 12:47:08 2007
Printed: Thu May 10 12:48:23 2007

MLC 5/10/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.999069
Calibration curve: 92.1846 * x + 269.952
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

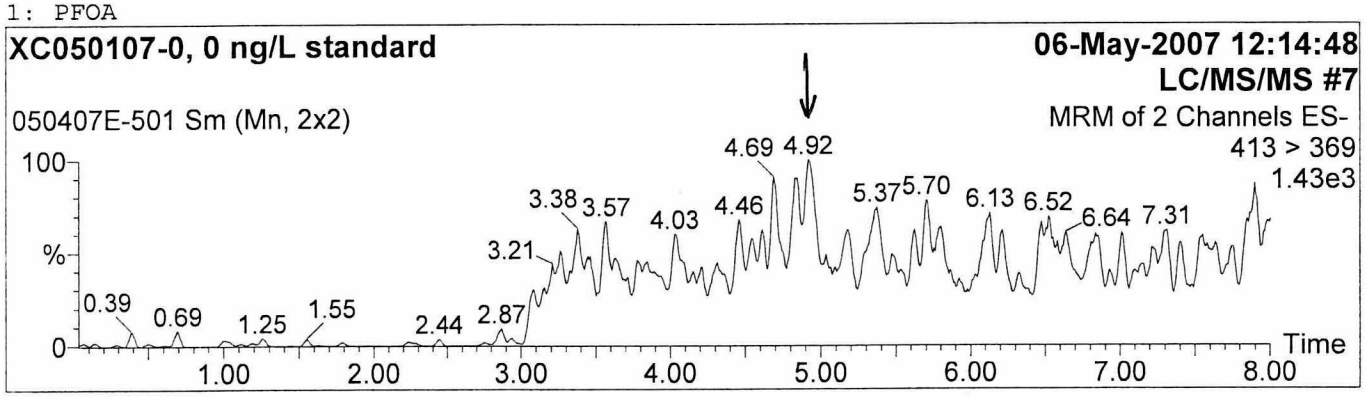
Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

Printed: Thu May 10 12:48:24 2007

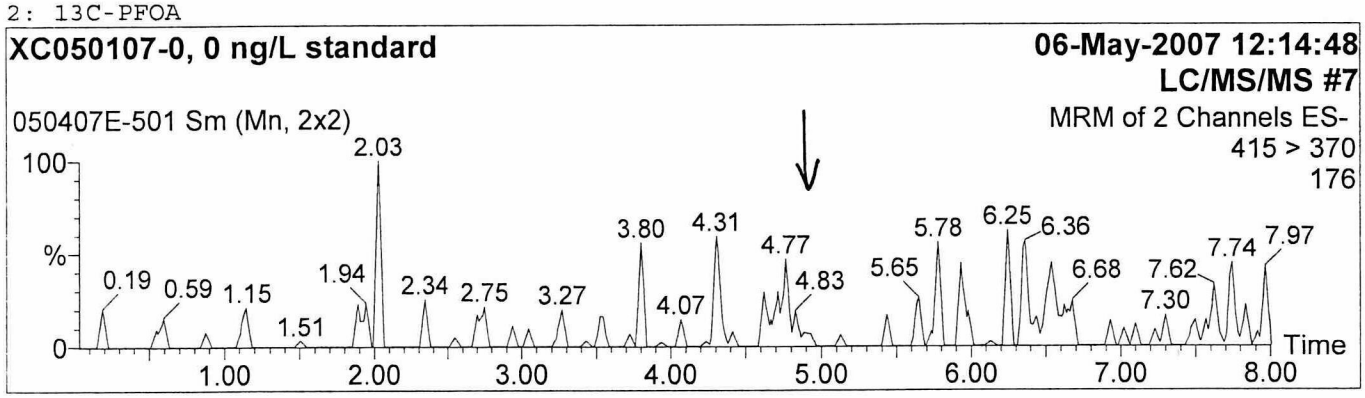
Initials MLC
Date 5/10/07
Run# 050407E-501 To 050407E-528

Name: 050407E-501
Text:

1: PFOA



2: 13C-PFOA



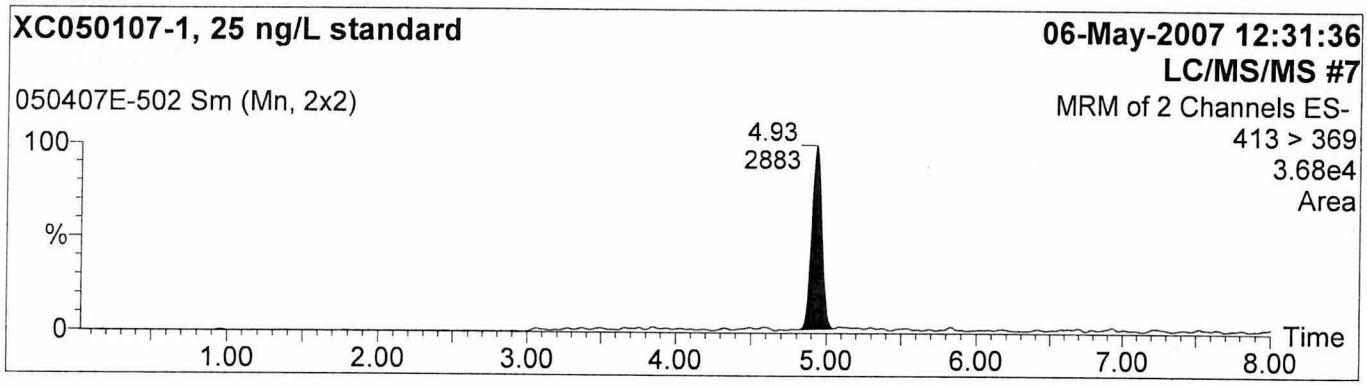
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

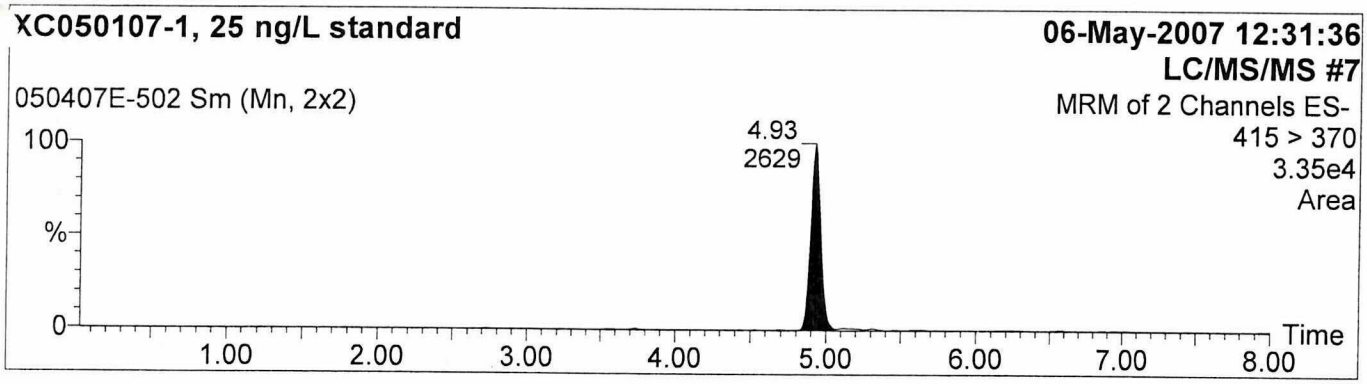
Printed: Thu May 10 12:48:24 2007

Name: 050407E-502
Text:

1: PFOA



2: 13C-PFOA



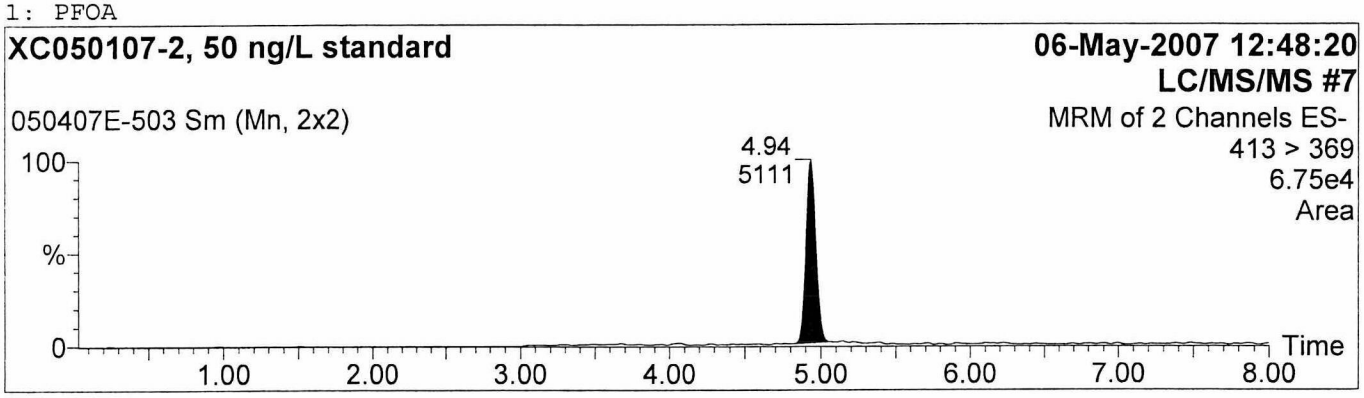
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

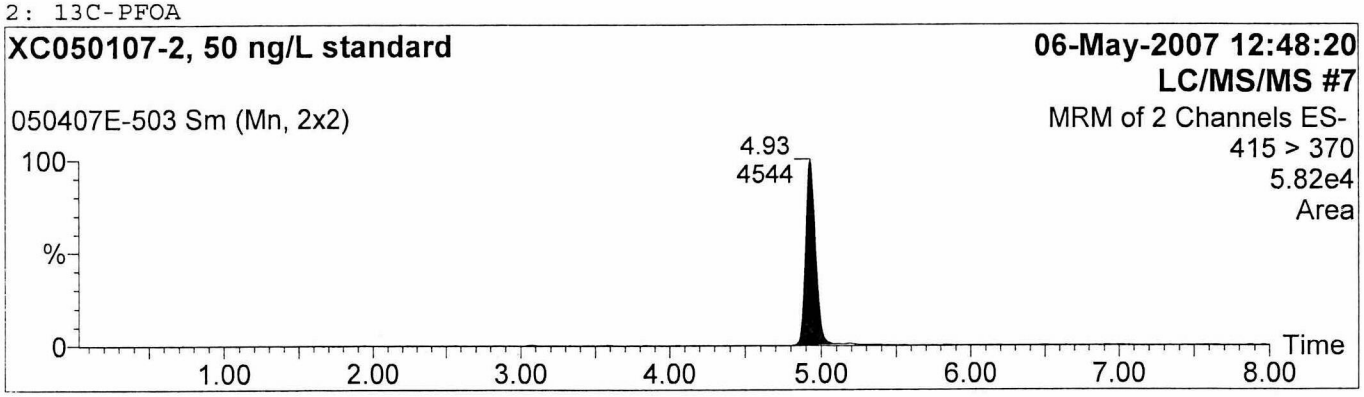
Printed: Thu May 10 12:48:24 2007

Name: 050407E-503
Text:

1: PFOA



2: 13C-PFOA



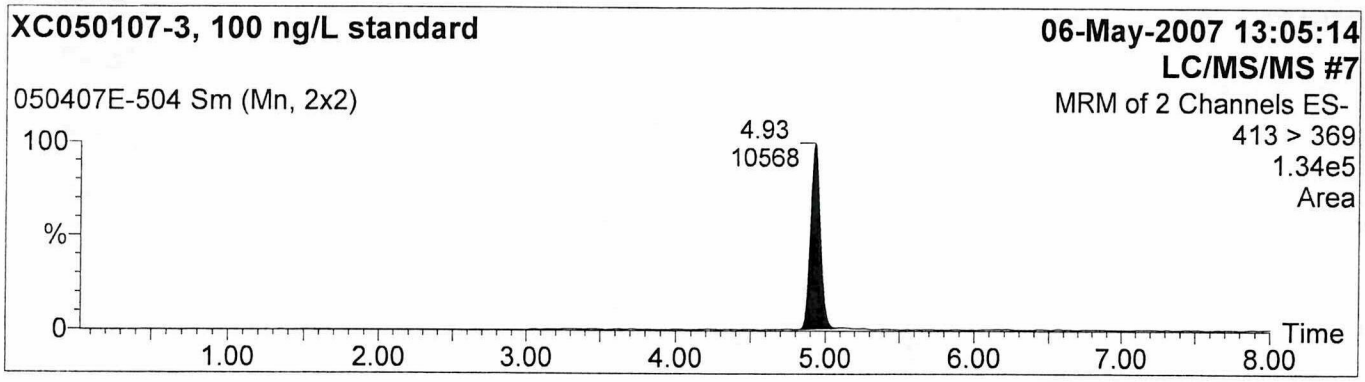
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

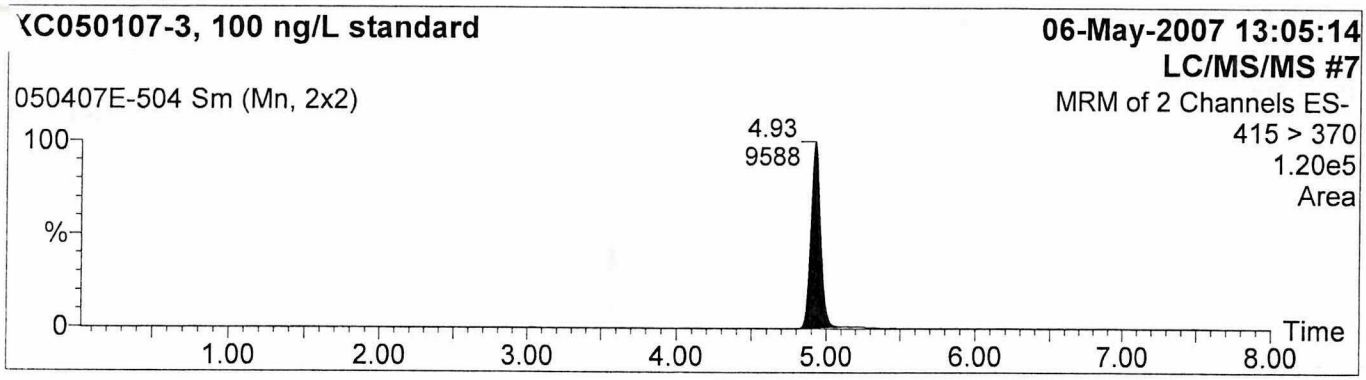
Printed: Thu May 10 12:48:24 2007

Name: 050407E-504
Text:

1: PFOA



2: 13C-PFOA



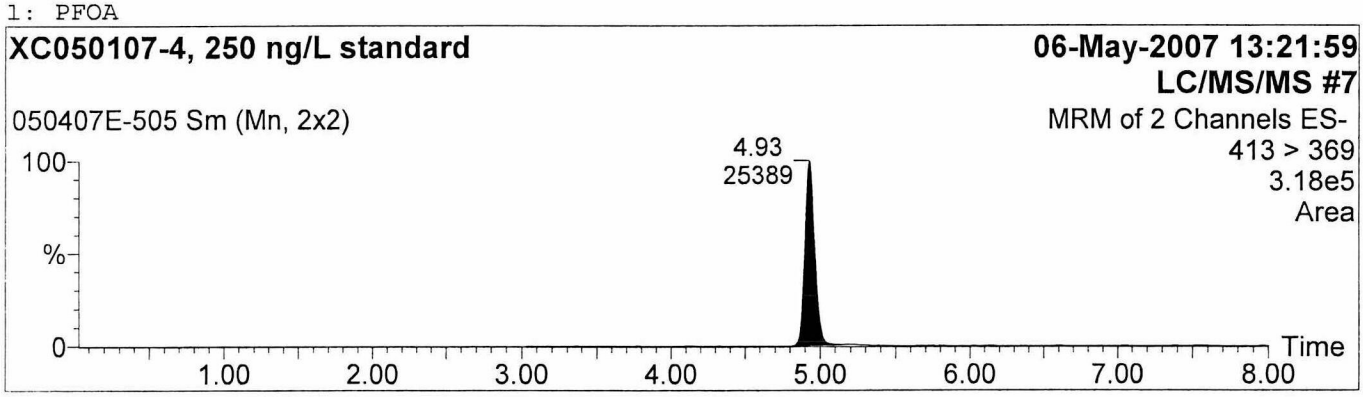
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

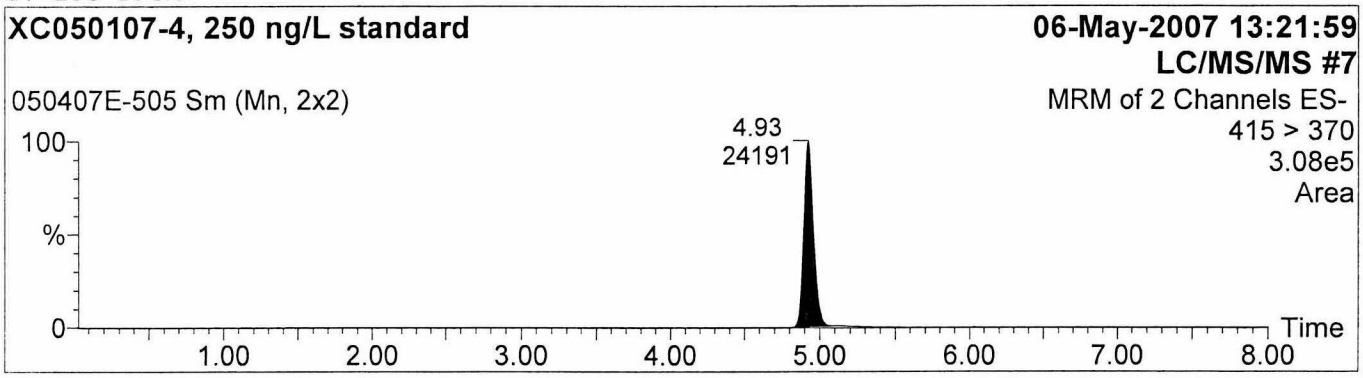
Printed: Thu May 10 12:48:24 2007

Name: 050407E-505
Text:

1: PFOA



2: 13C-PFOA



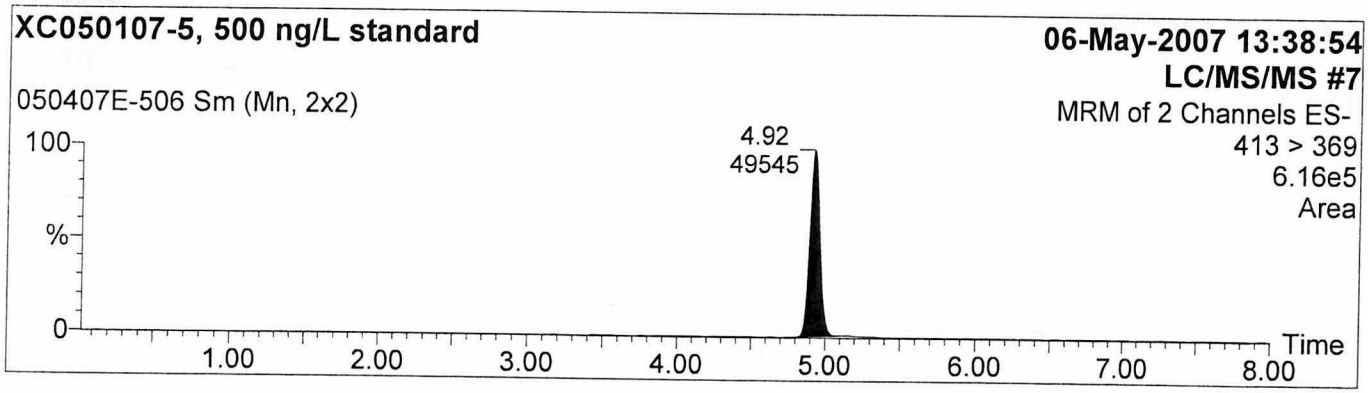
Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

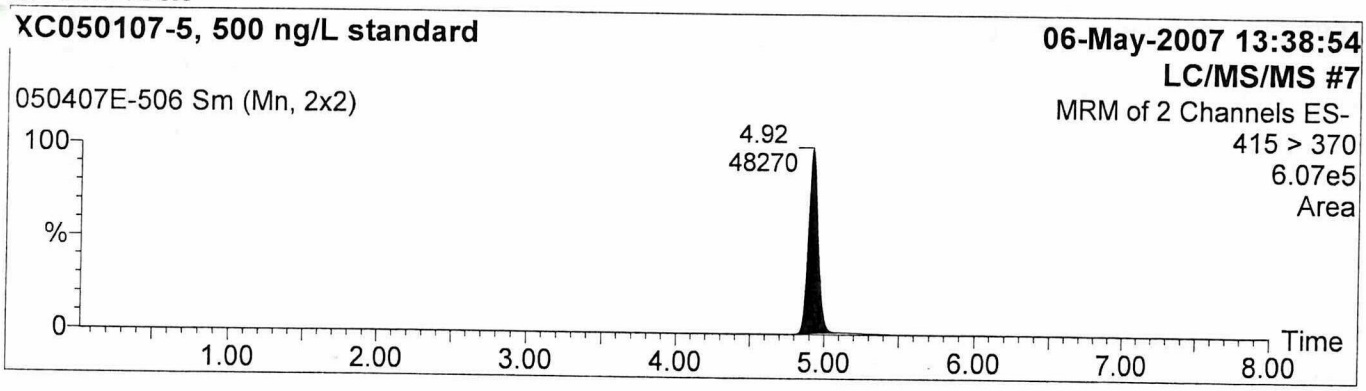
Printed: Thu May 10 12:48:24 2007

Name: 050407E-506
Text:

1: PFOA



2: 13C-PFOA



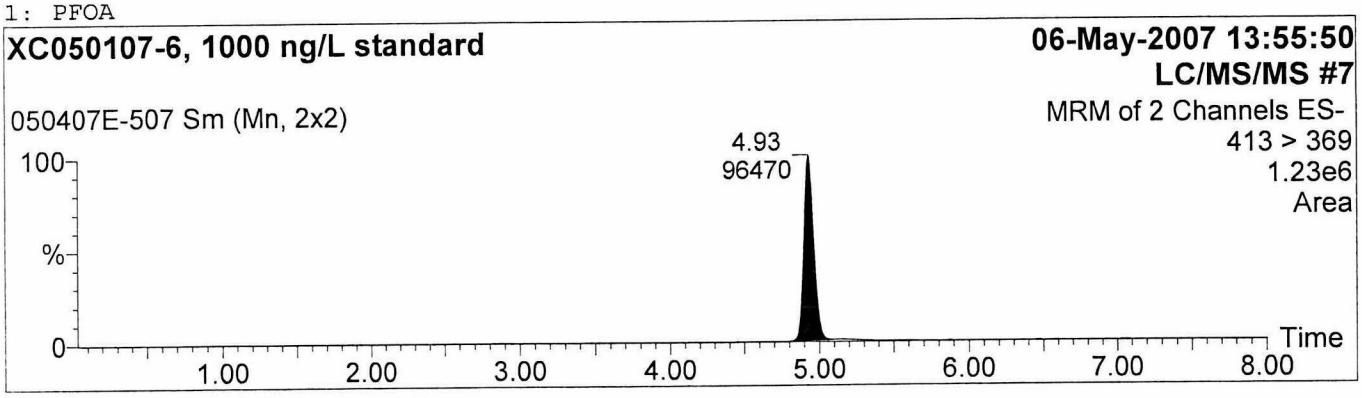
Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

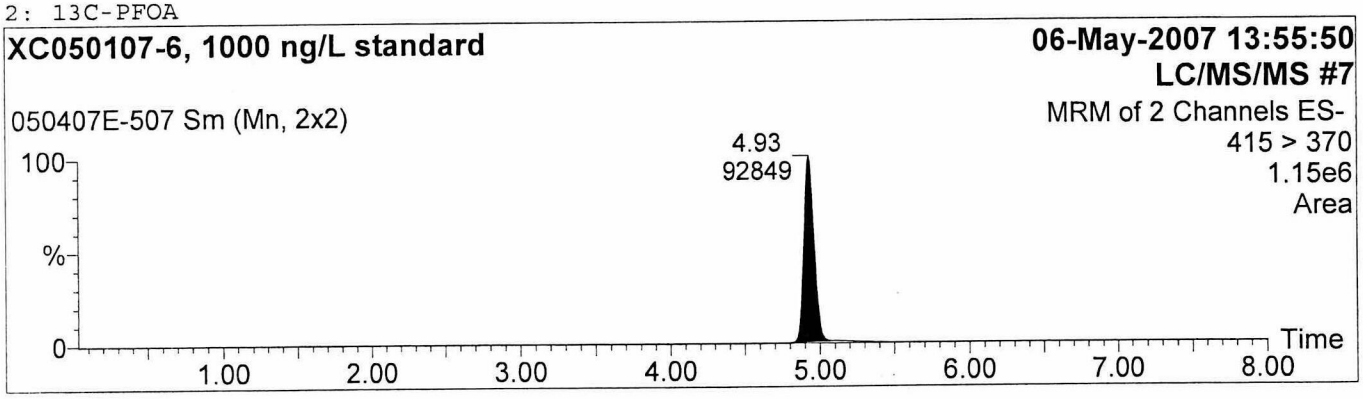
Printed: Thu May 10 12:48:24 2007

Name: 050407E-507
Text:

1: PFOA



2: 13C-PFOA



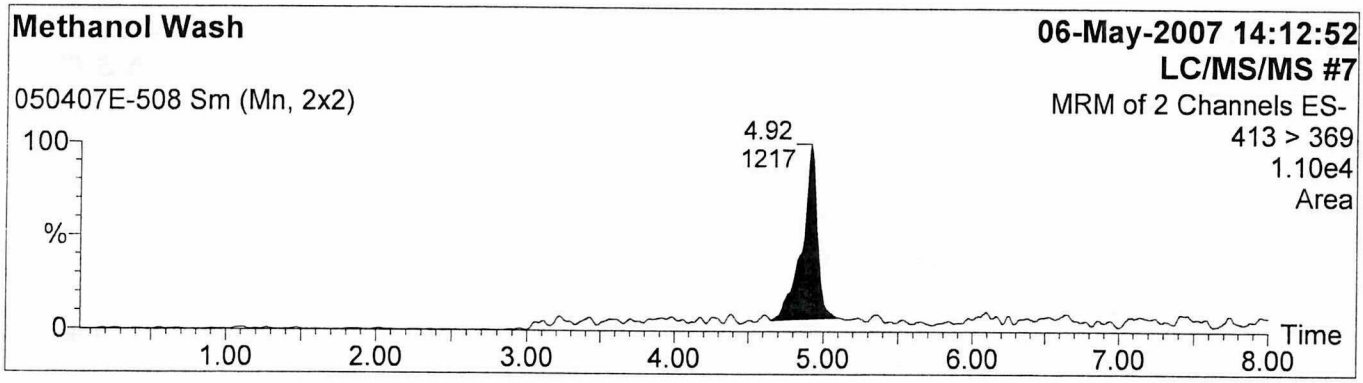
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

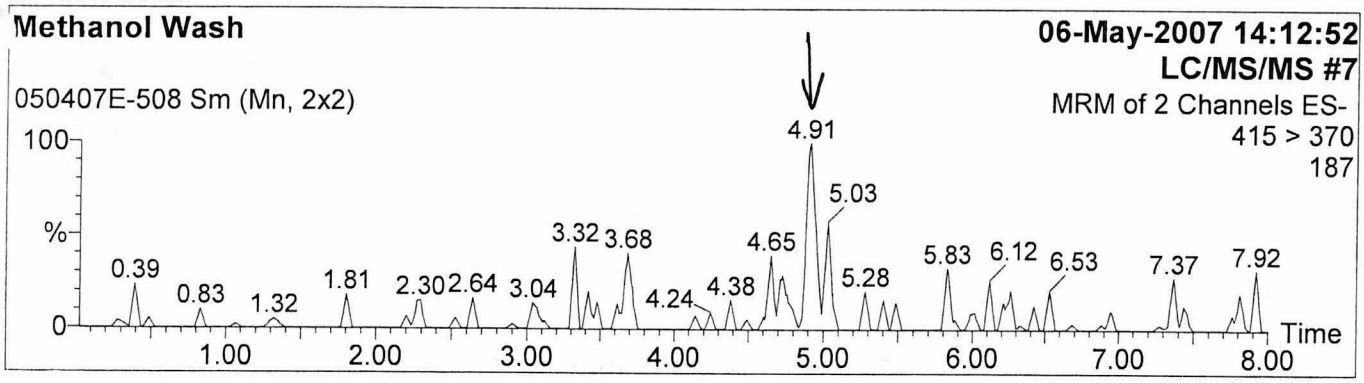
Printed: Thu May 10 12:48:24 2007

Name: 050407E-508
Text:

1: PFOA



2: 13C-PFOA



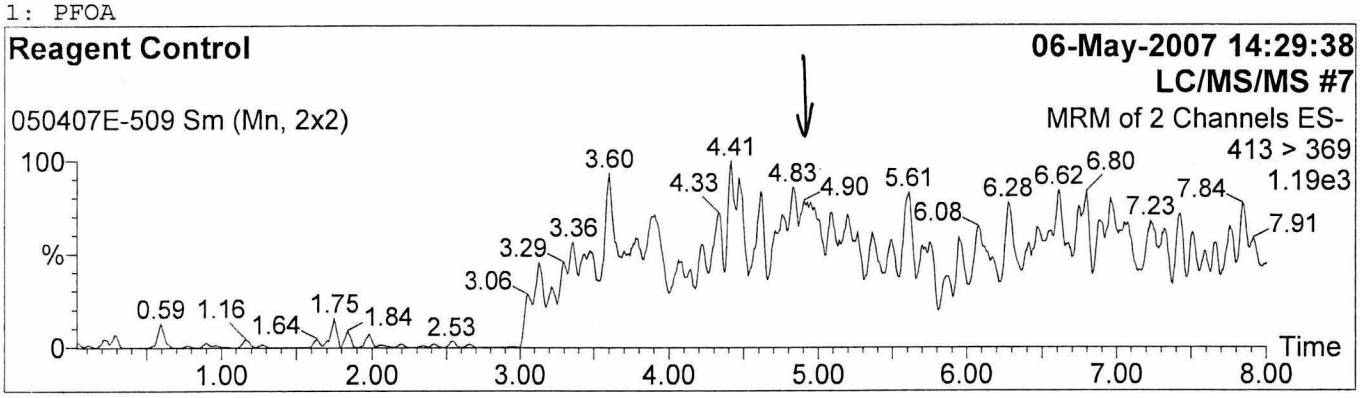
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

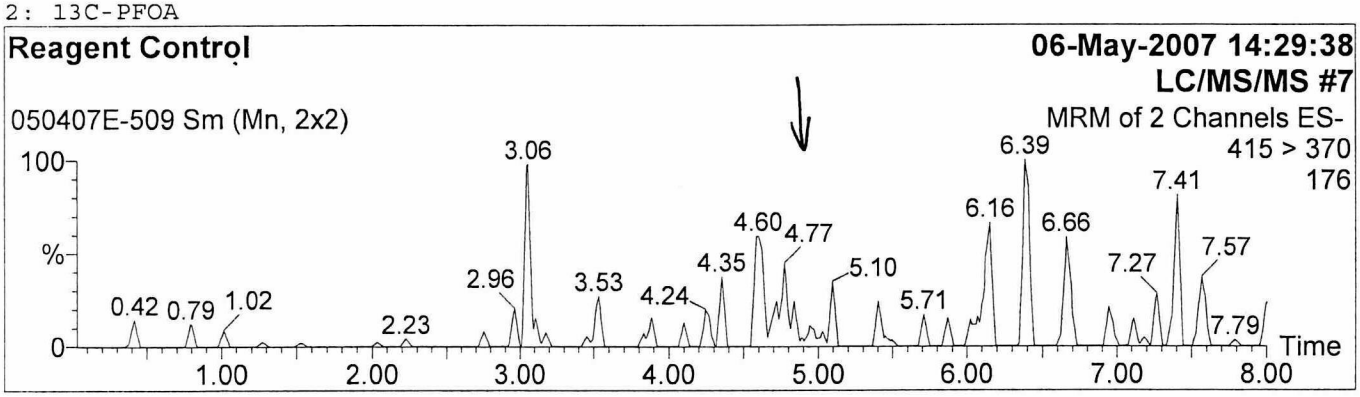
Printed: Thu May 10 12:48:24 2007

Name: 050407E-509
Text:

1: PFOA



2: 13C-PFOA



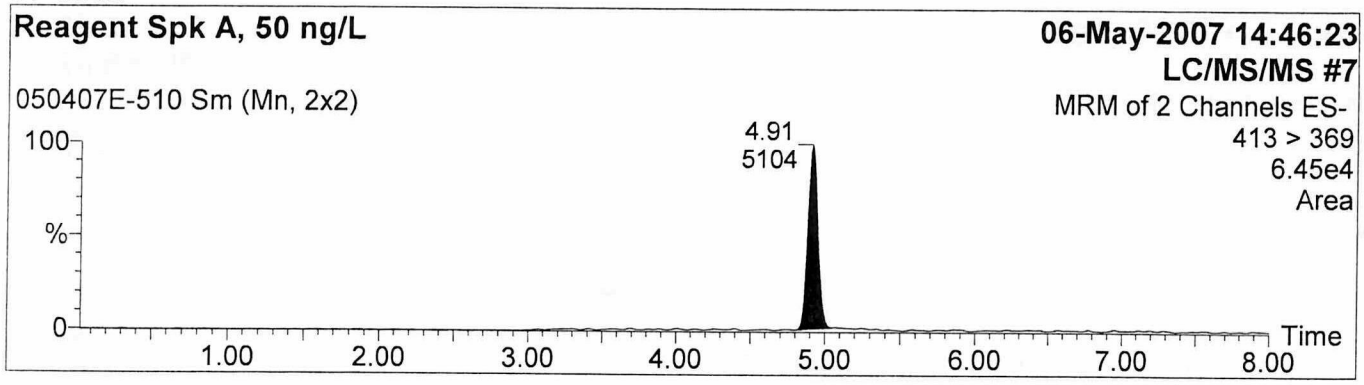
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

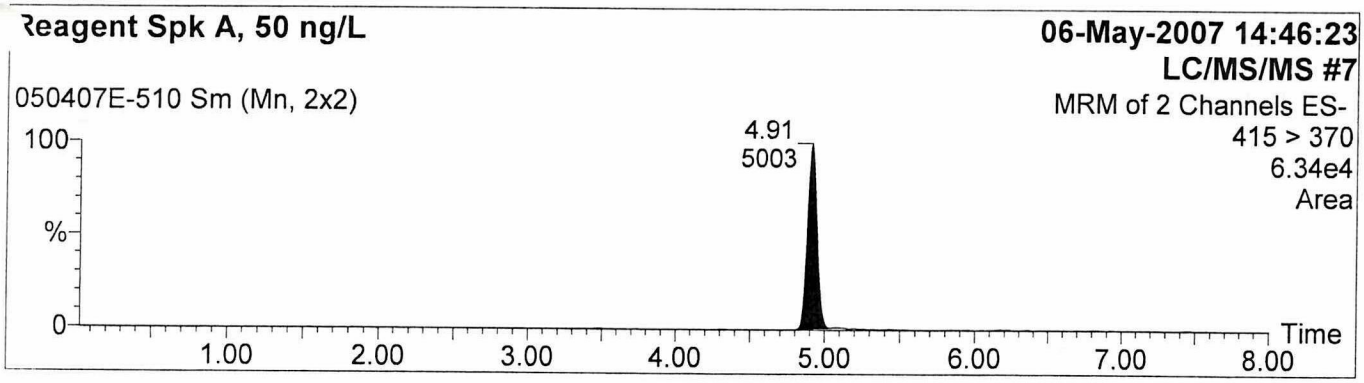
Printed: Thu May 10 12:48:24 2007

Name: 050407E-510
Text:

1: PFOA



2: 13C-PFOA



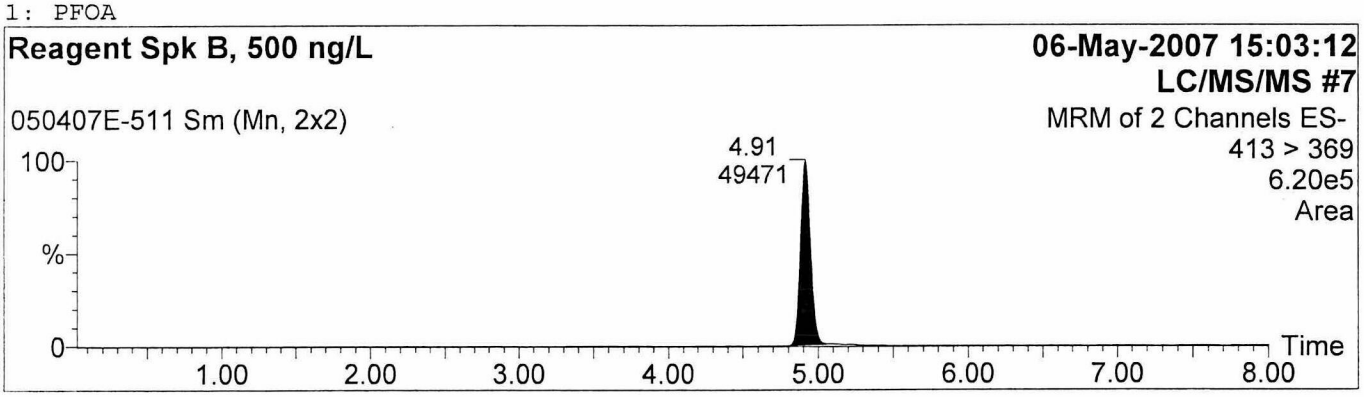
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

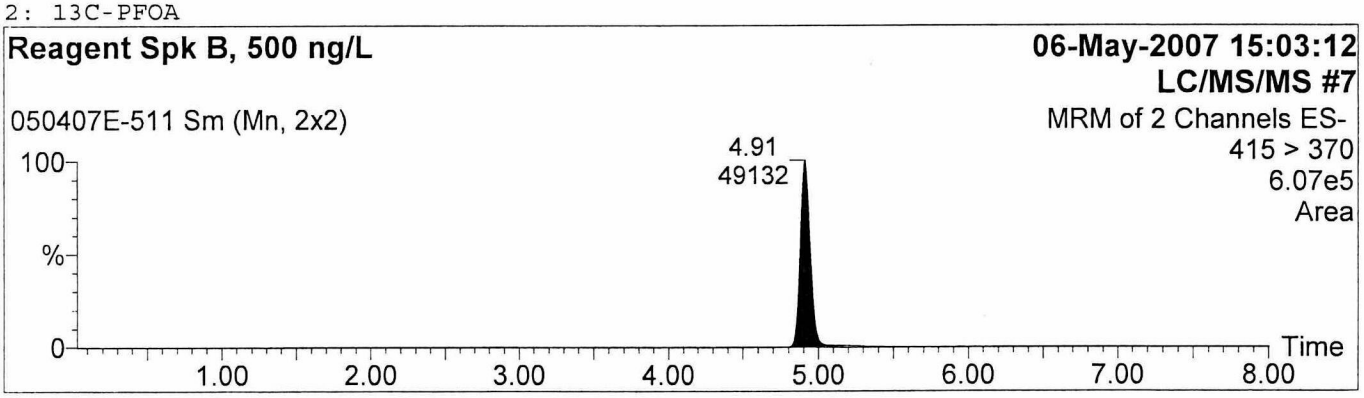
Printed: Thu May 10 12:48:24 2007

Name: 050407E-511
Text:

1: PFOA



2: 13C-PFOA



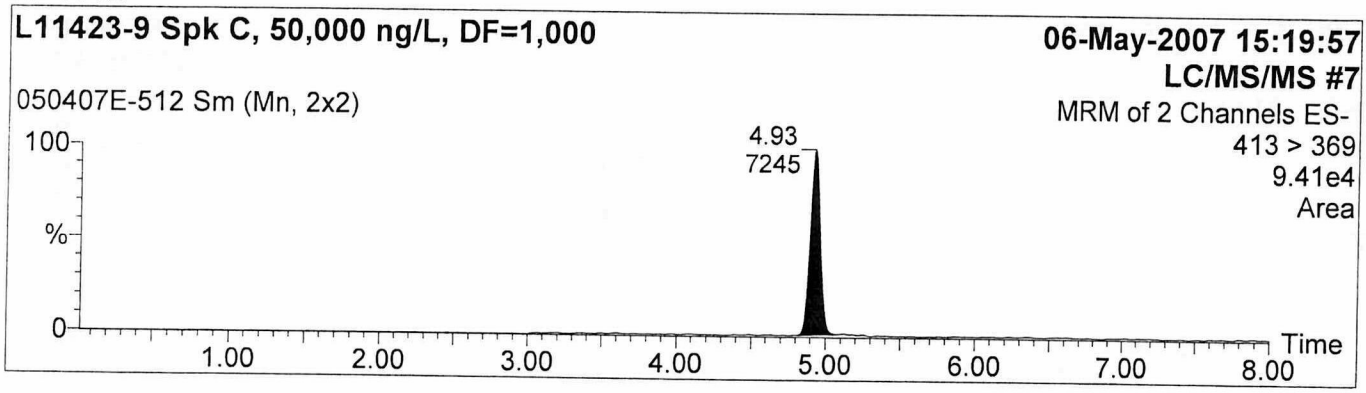
Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

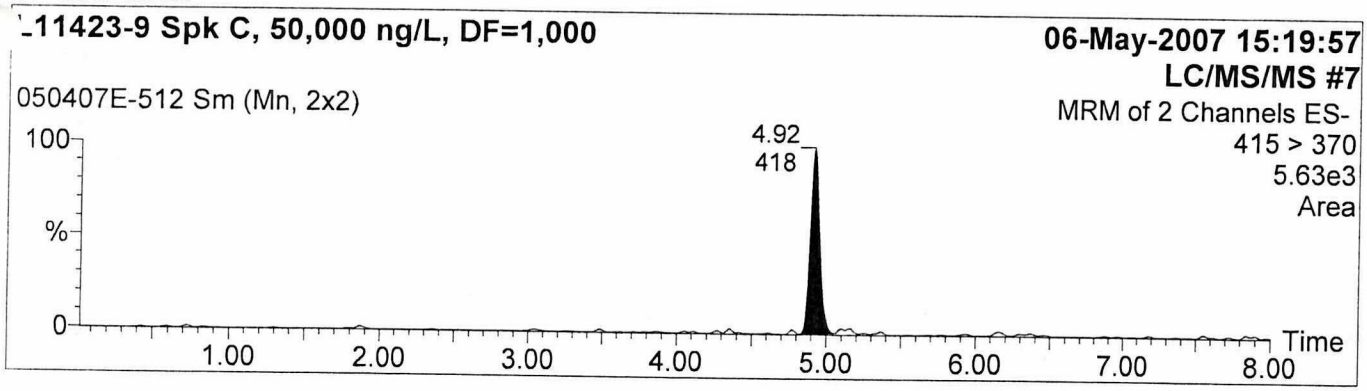
Printed: Thu May 10 12:48:24 2007

Name: 050407E-512
Text:

1: PFOA



2: 13C-PFOA



Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
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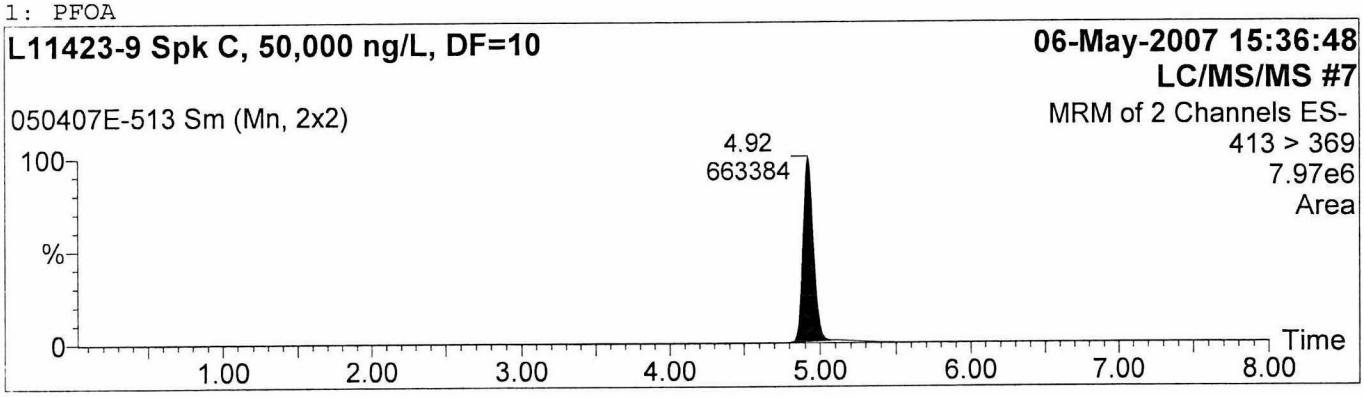
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

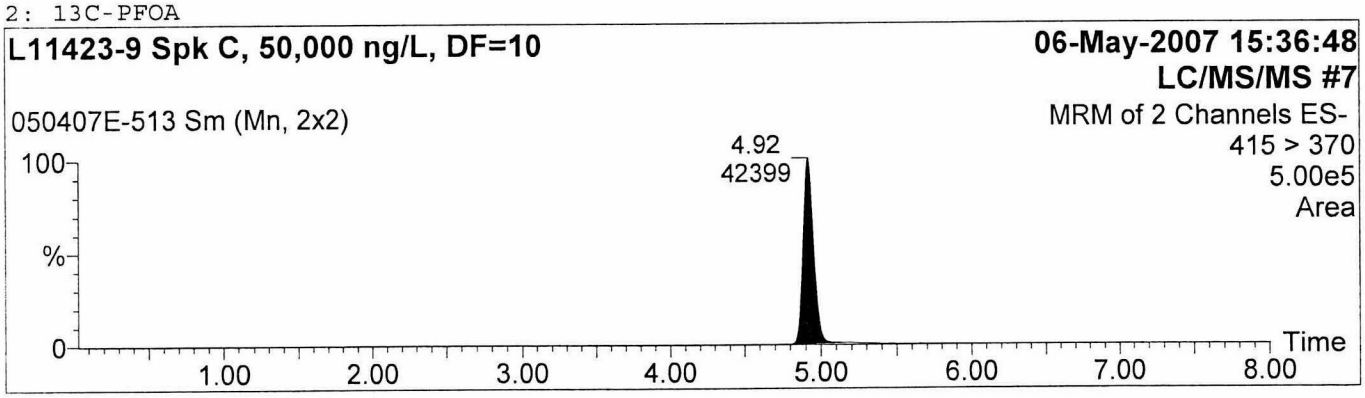
Printed: Thu May 10 12:48:24 2007

Name: 050407E-513
Text:

1: PFOA



2: 13C-PFOA



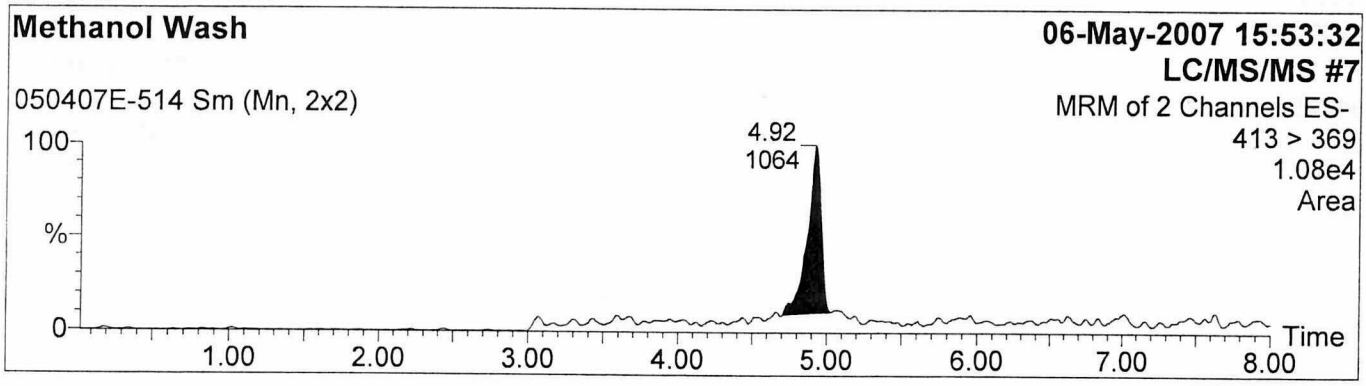
Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

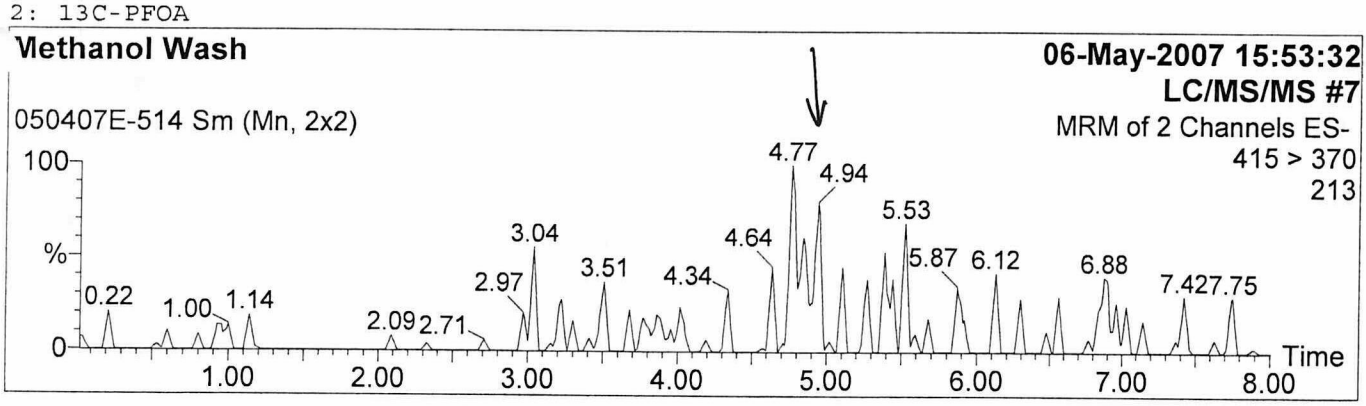
Printed: Thu May 10 12:48:24 2007

Name: 050407E-514
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
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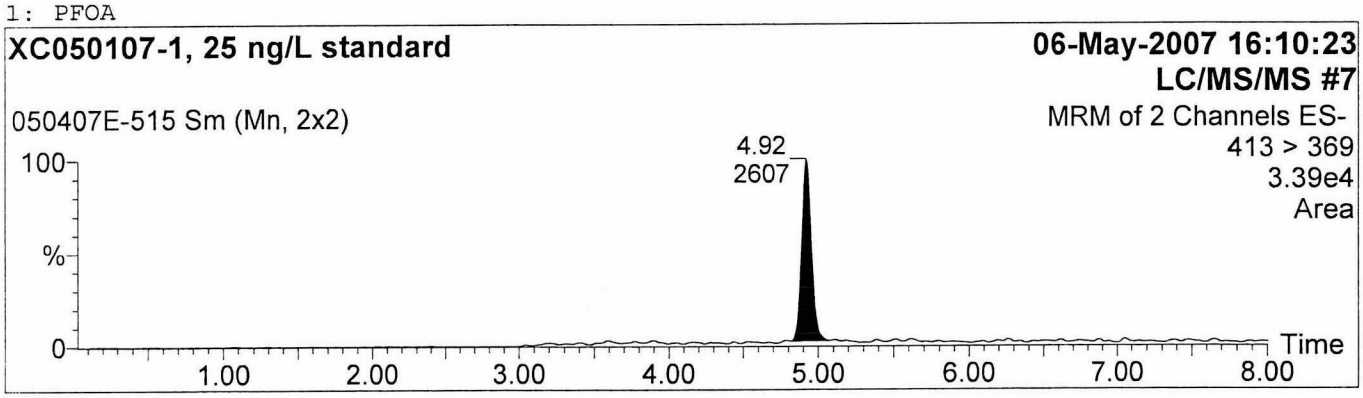
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

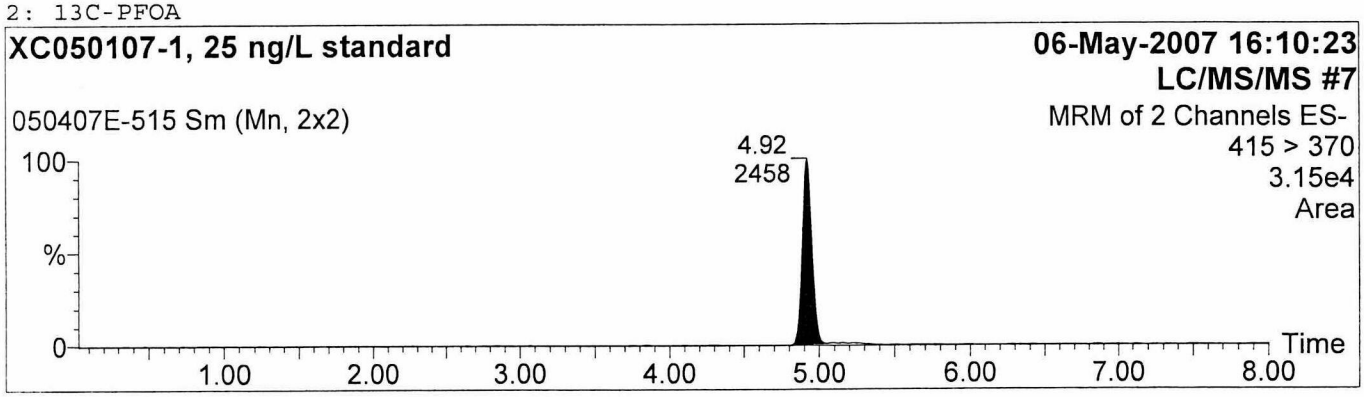
Printed: Thu May 10 12:48:24 2007

Name: 050407E-515
Text:

1: PFOA



2: 13C-PFOA



Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
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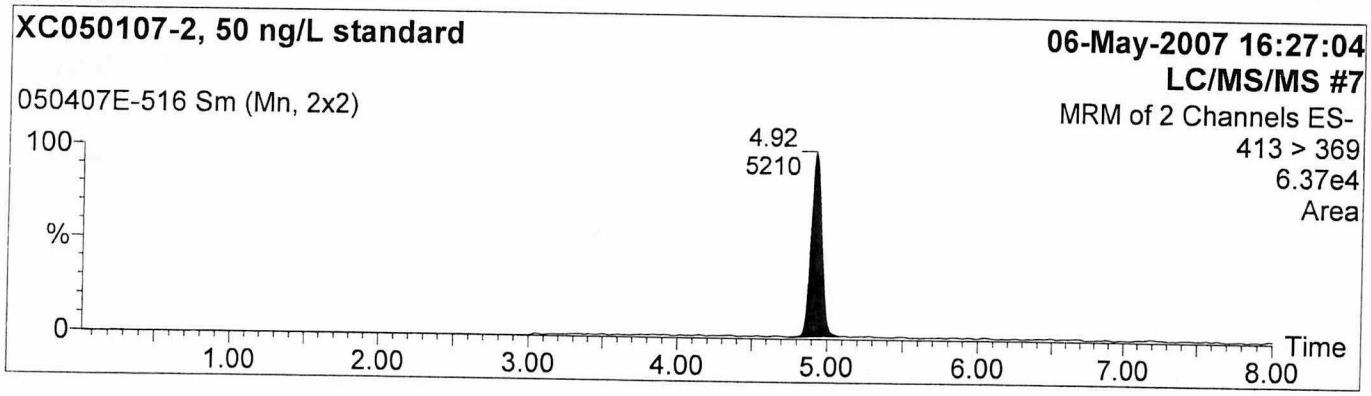
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

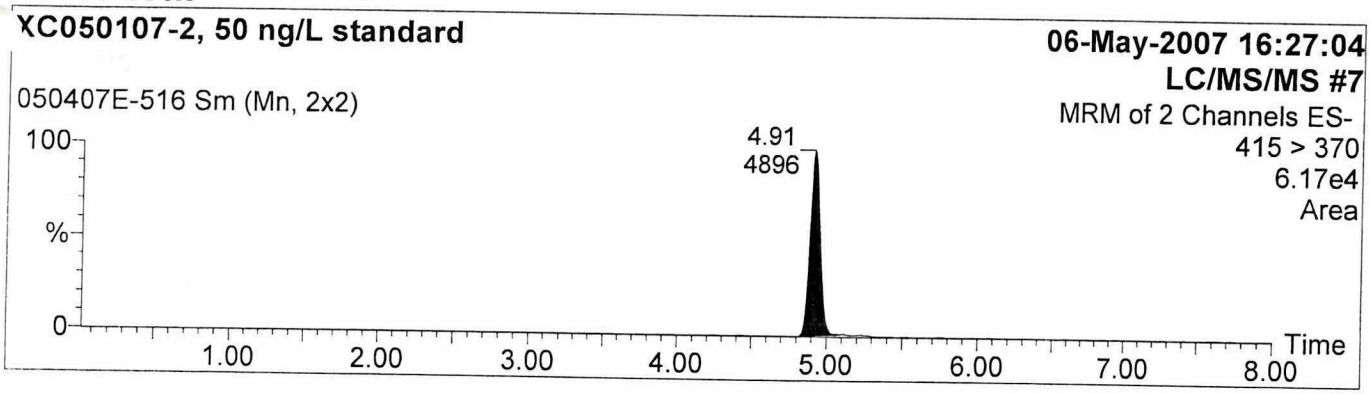
Printed: Thu May 10 12:48:24 2007

Name: 050407E-516
Text:

1: PFOA



2: 13C-PFOA



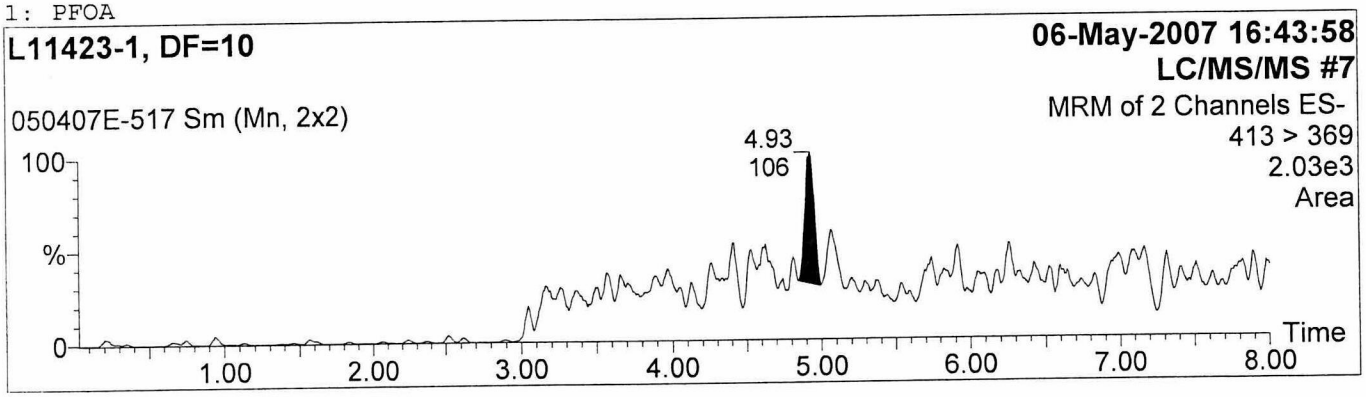
Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

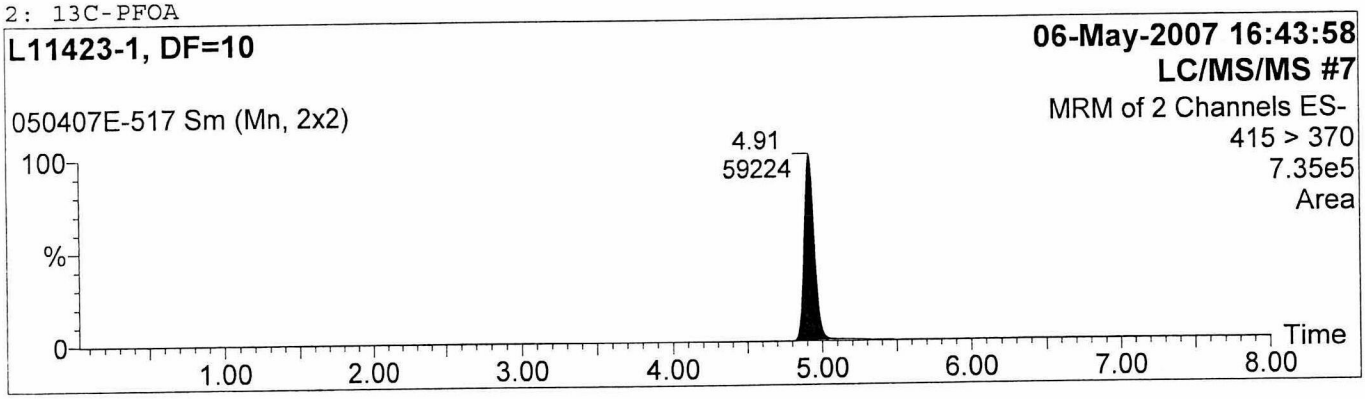
Printed: Thu May 10 12:48:24 2007

Name: 050407E-517
Text:

1: PFOA



2: 13C-PFOA



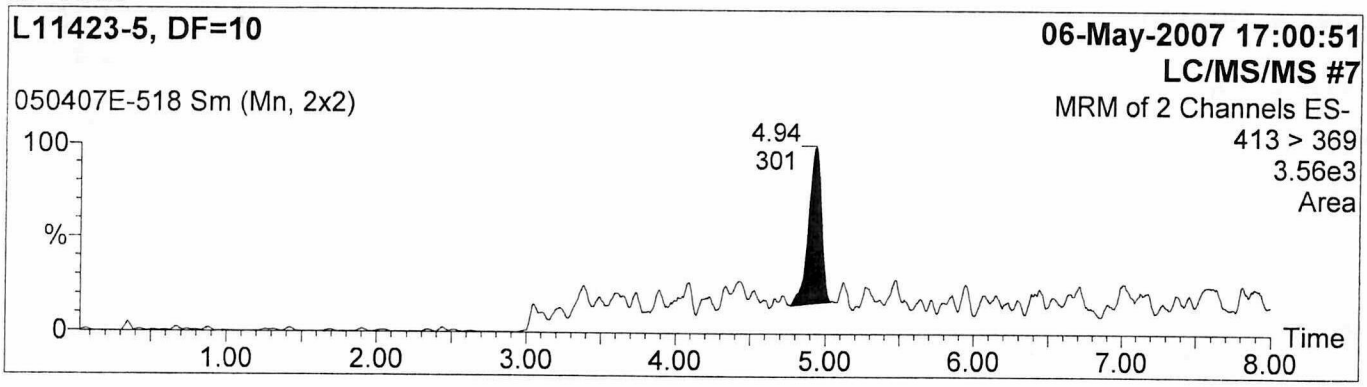
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

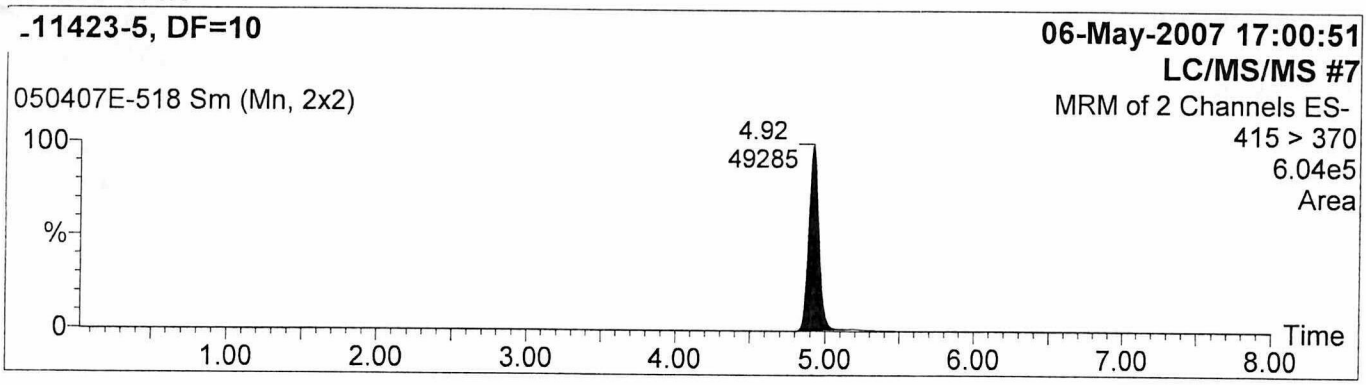
Printed: Thu May 10 12:48:24 2007

Name: 050407E-518
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
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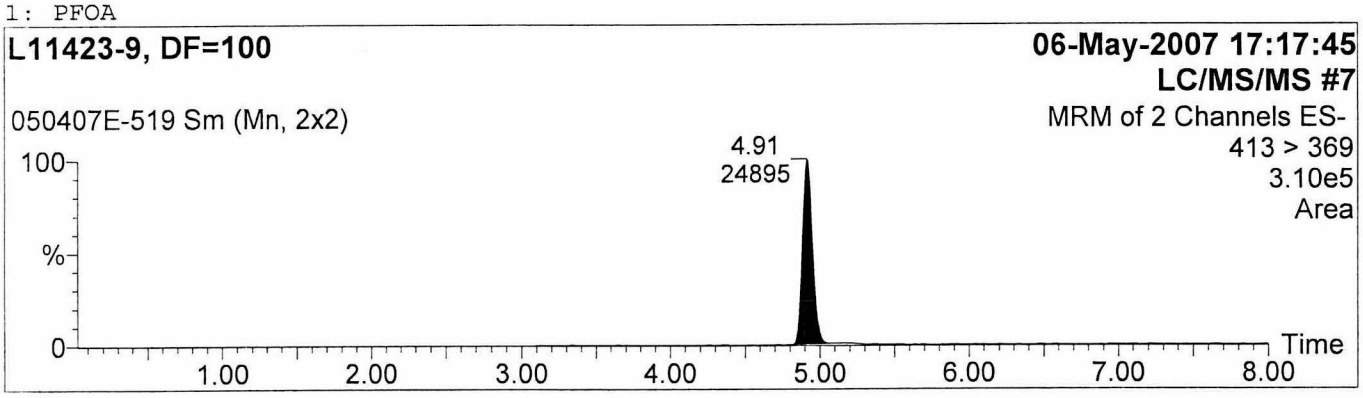
Last modified: Thu May 03 10:45:11 2007
Job Code:

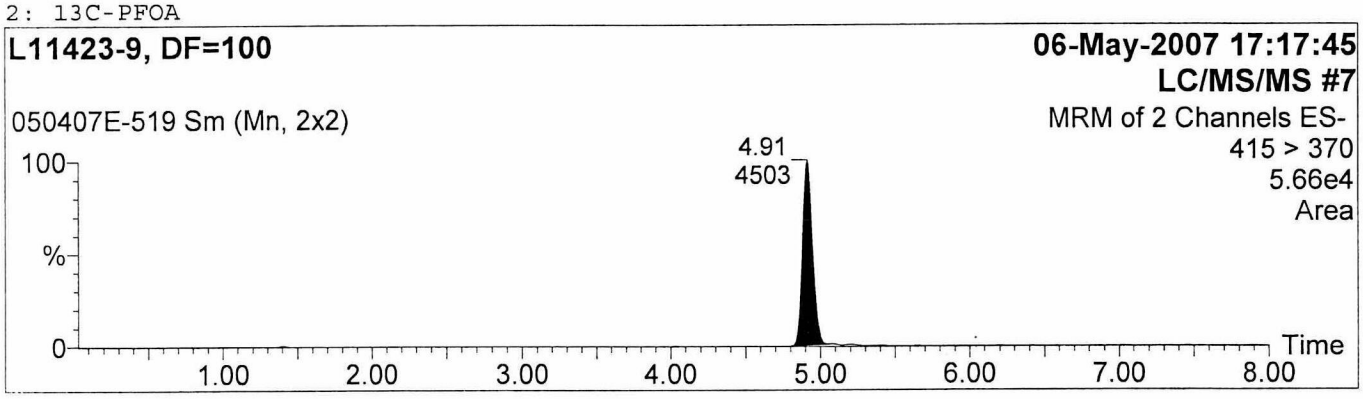
Printed: Thu May 10 12:48:24 2007

Name: 050407E-519
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
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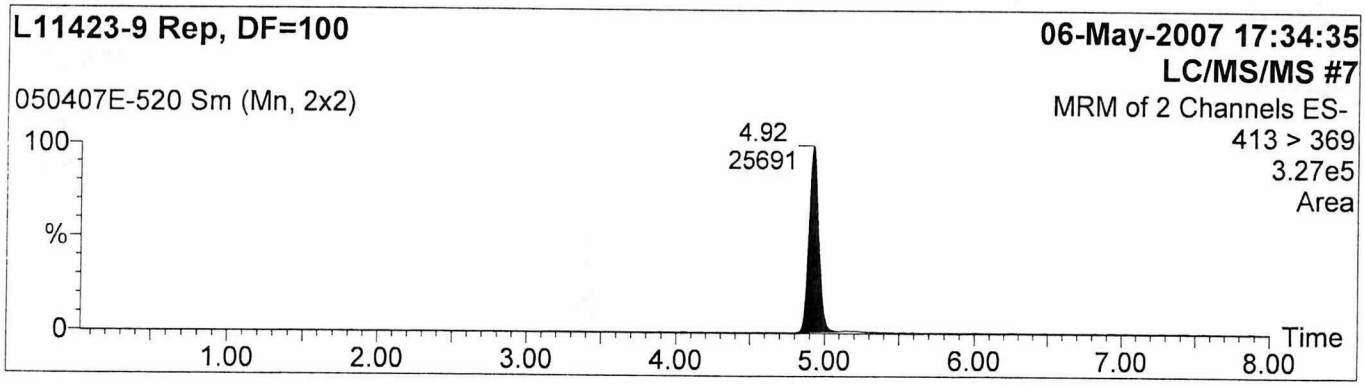
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

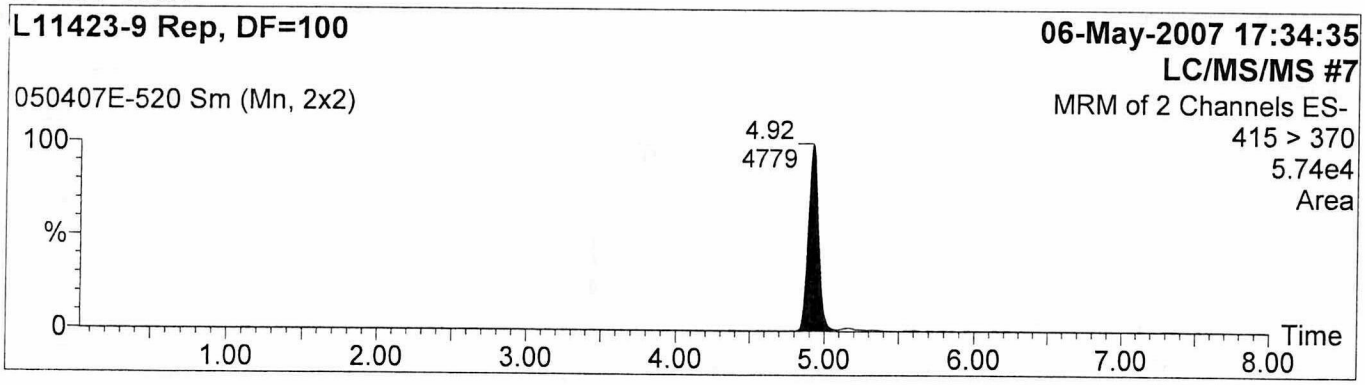
Printed: Thu May 10 12:48:24 2007

Name: 050407E-520
Text:

1: PFOA



2: 13C-PFOA



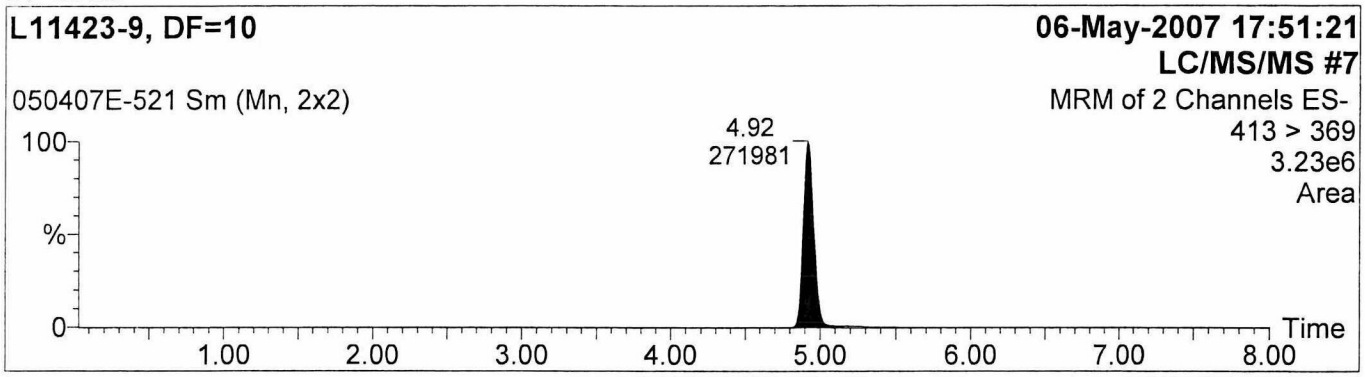
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

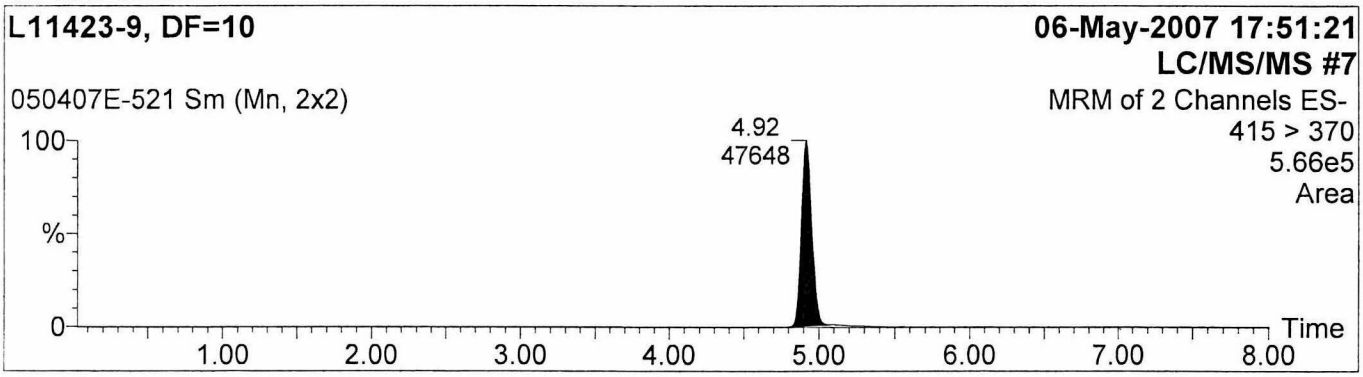
Printed: Thu May 10 12:48:24 2007

Name: 050407E-521
Text:

1: PFOA



2: 13C-PFOA



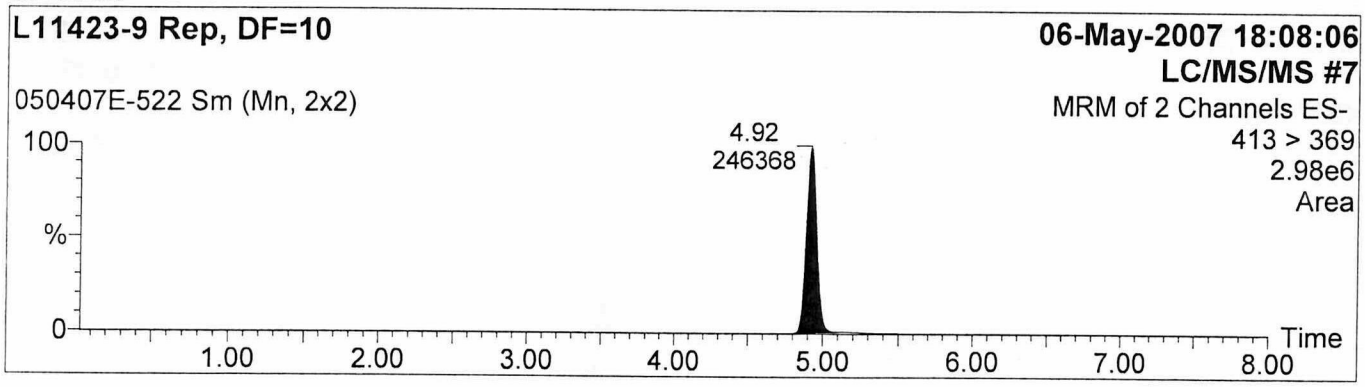
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

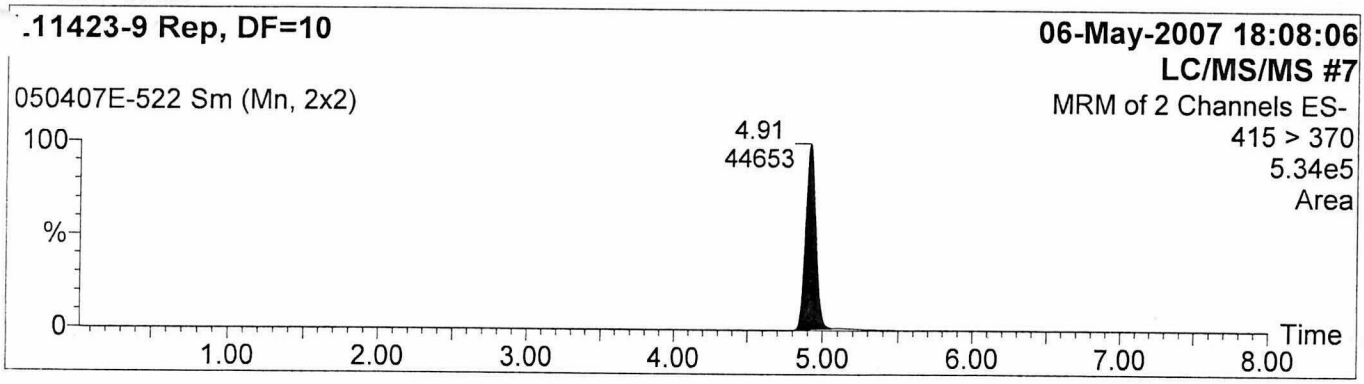
Printed: Thu May 10 12:48:24 2007

Name: 050407E-522
Text:

1: PFOA



2: 13C-PFOA



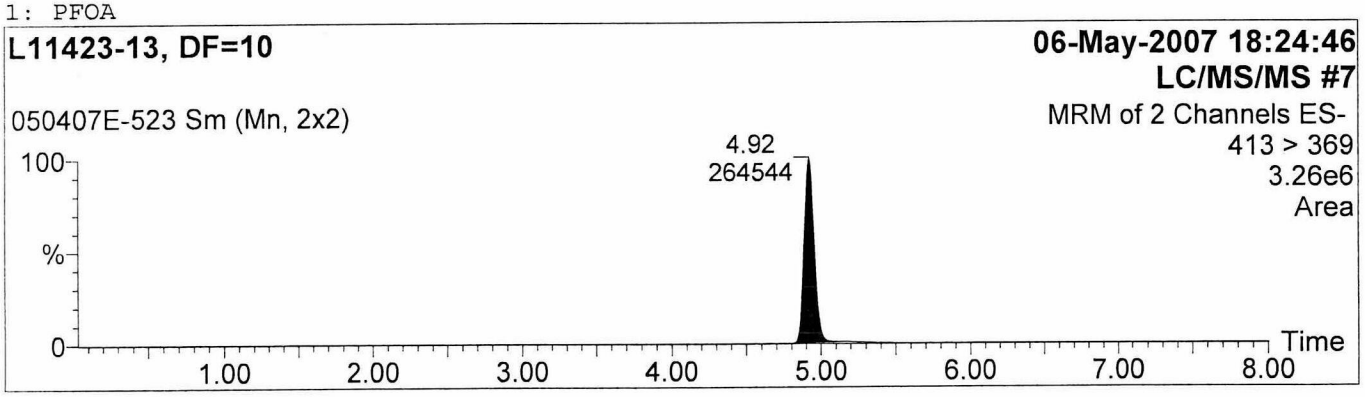
Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

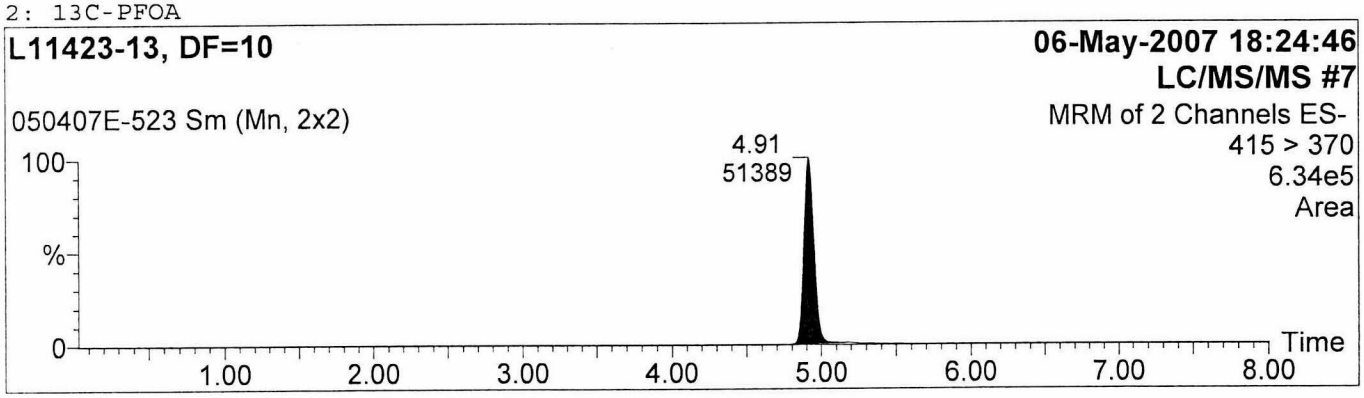
Printed: Thu May 10 12:48:24 2007

Name: 050407E-523
Text:

1: PFOA



2: 13C-PFOA



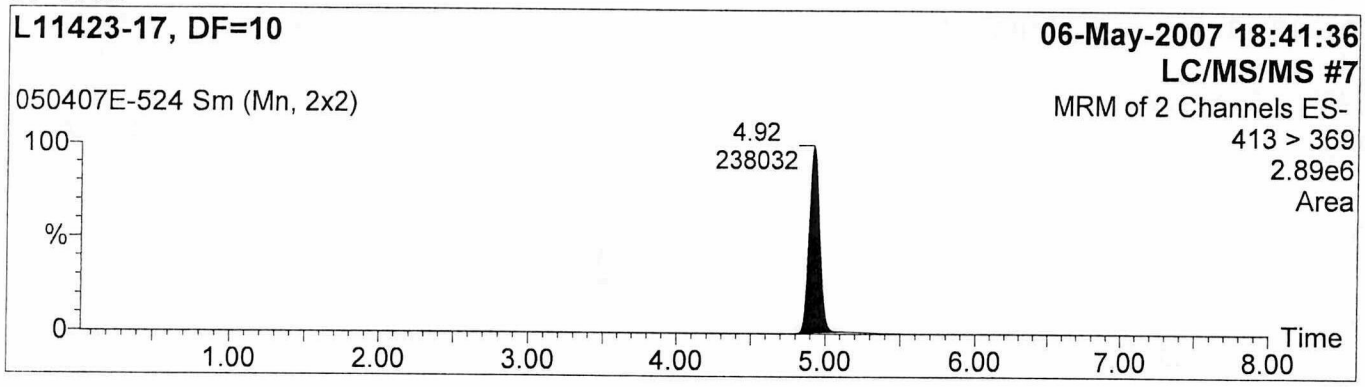
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

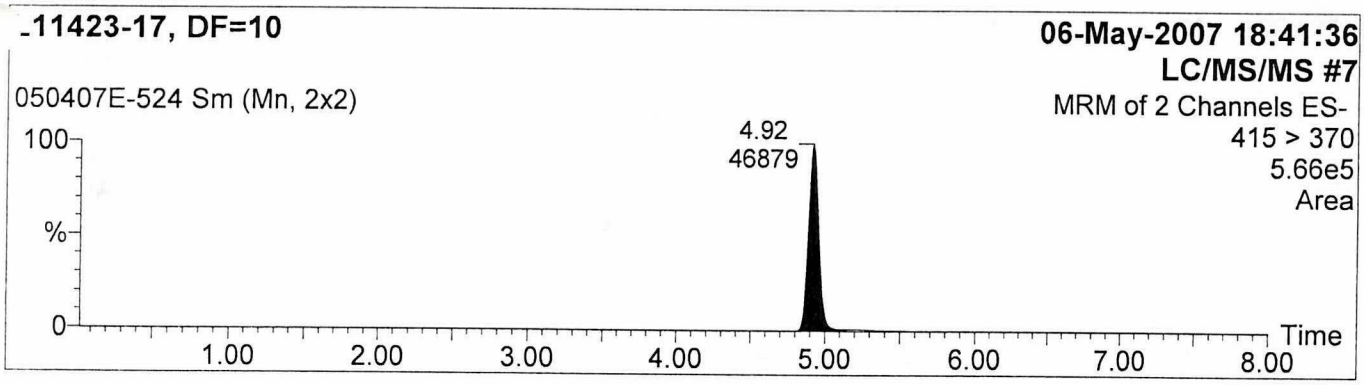
Printed: Thu May 10 12:48:24 2007

Name: 050407E-524
Text:

1: PFOA



2: 13C-PFOA



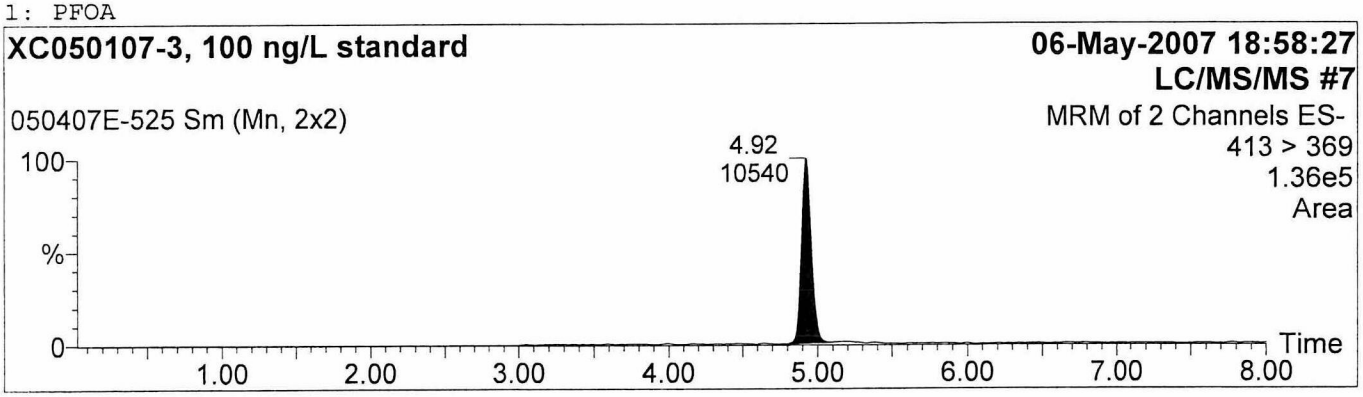
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

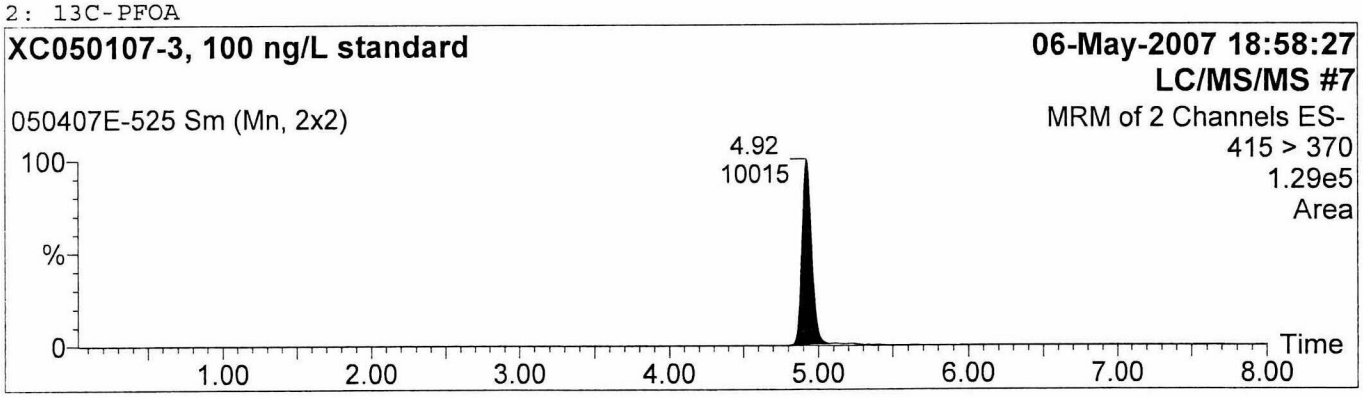
Printed: Thu May 10 12:48:24 2007

Name: 050407E-525
Text:

1: PFOA



2: 13C-PFOA



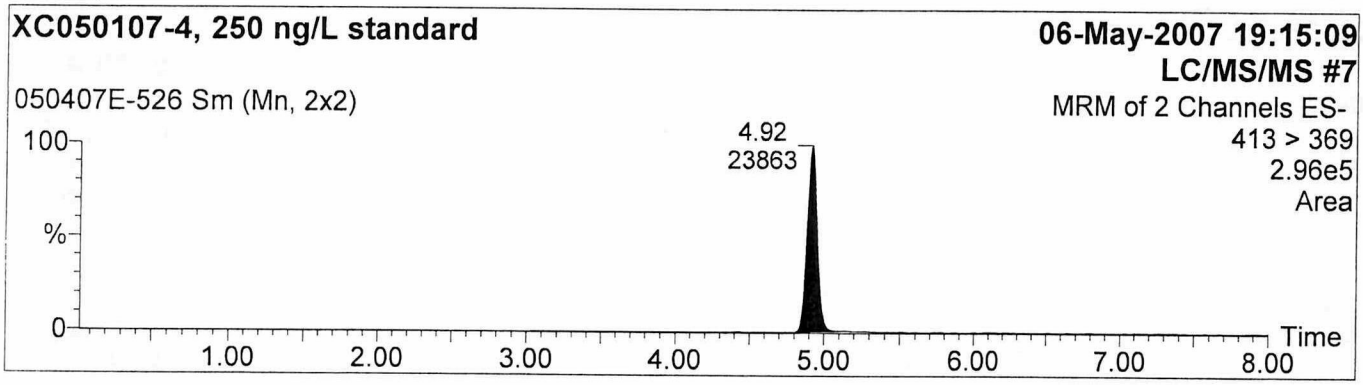
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

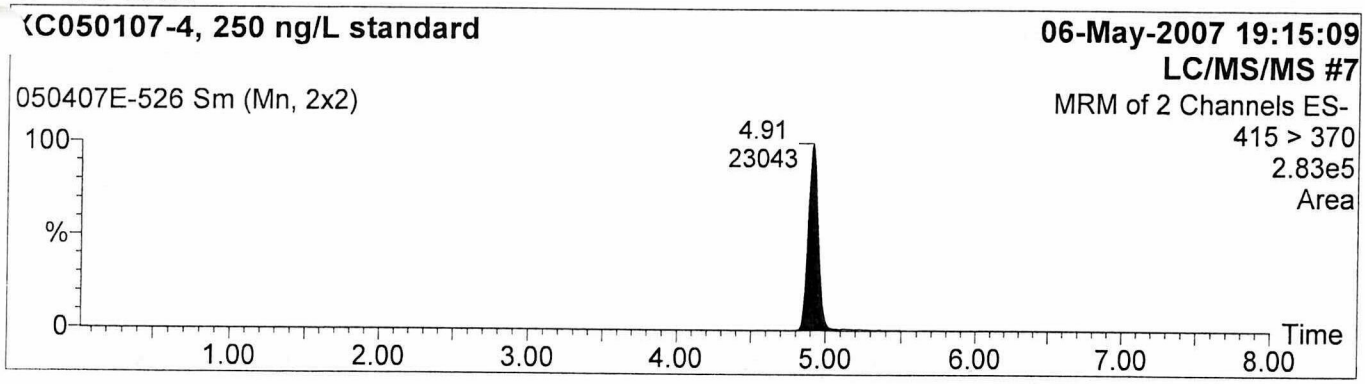
Printed: Thu May 10 12:48:24 2007

Name: 050407E-526
Text:

1: PFOA



2: 13C-PFOA



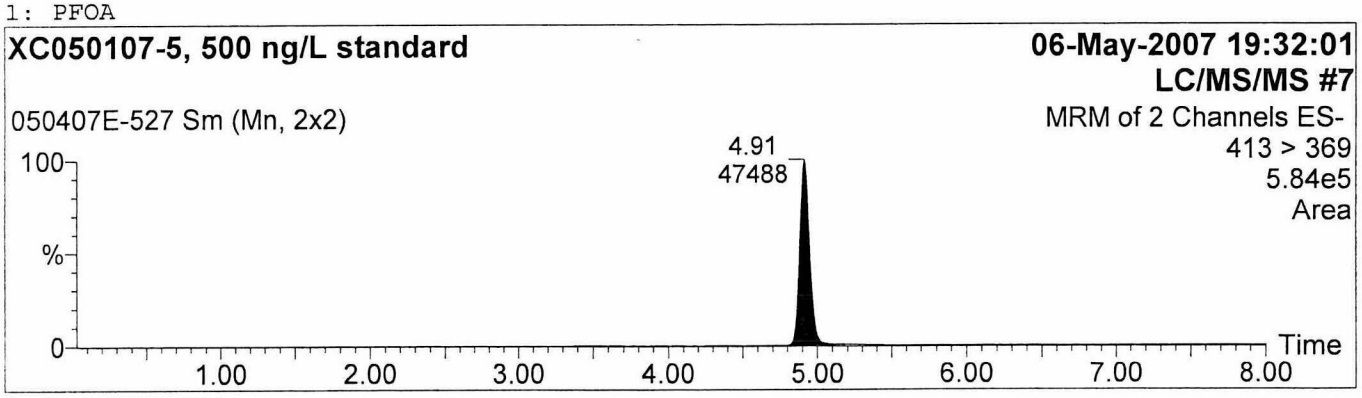
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

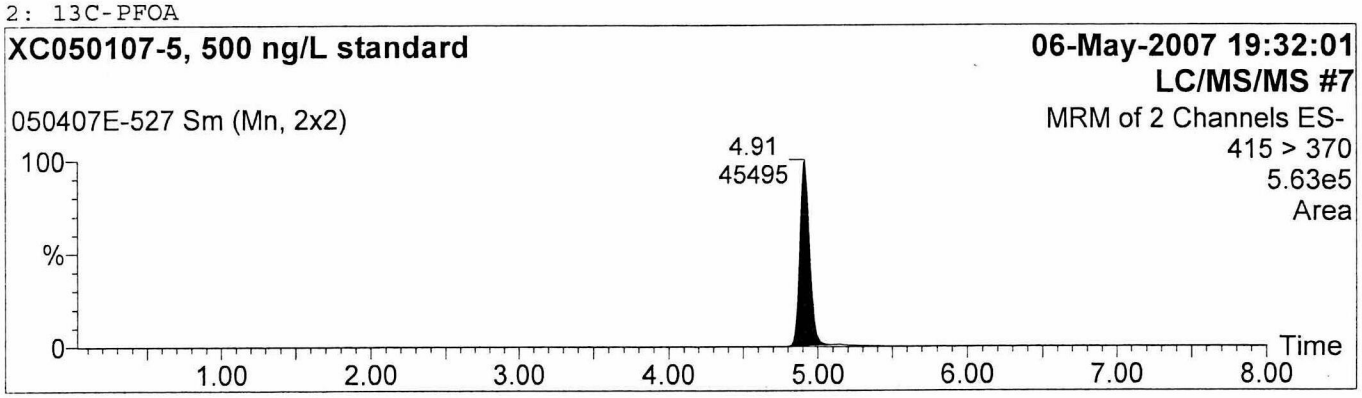
Printed: Thu May 10 12:48:24 2007

Name: 050407E-527
Text:

1: PFOA



2: 13C-PFOA



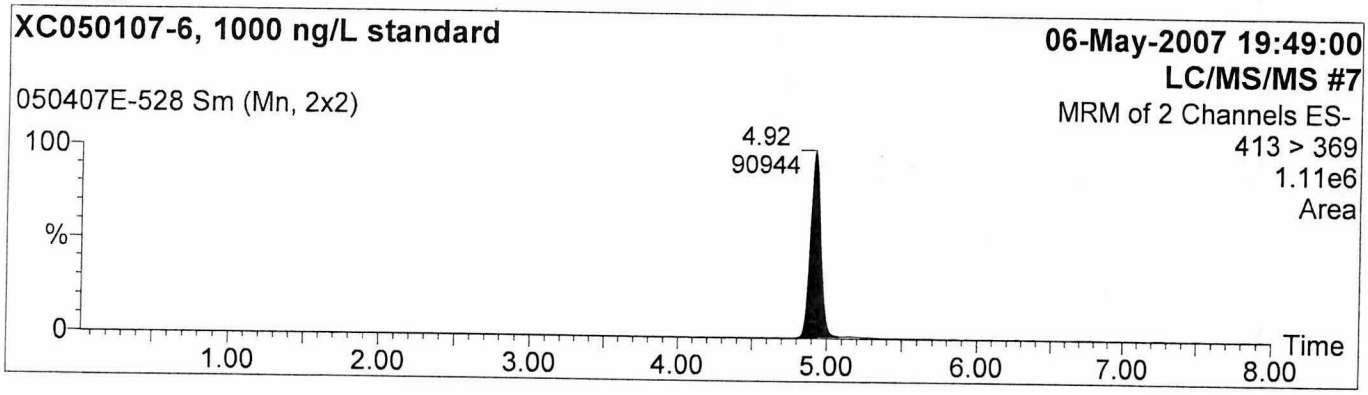
Quantify Sample Report
Study No: L11423, Set No: 050407E. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407E DI Water
Last modified: Thu May 10 12:39:49 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

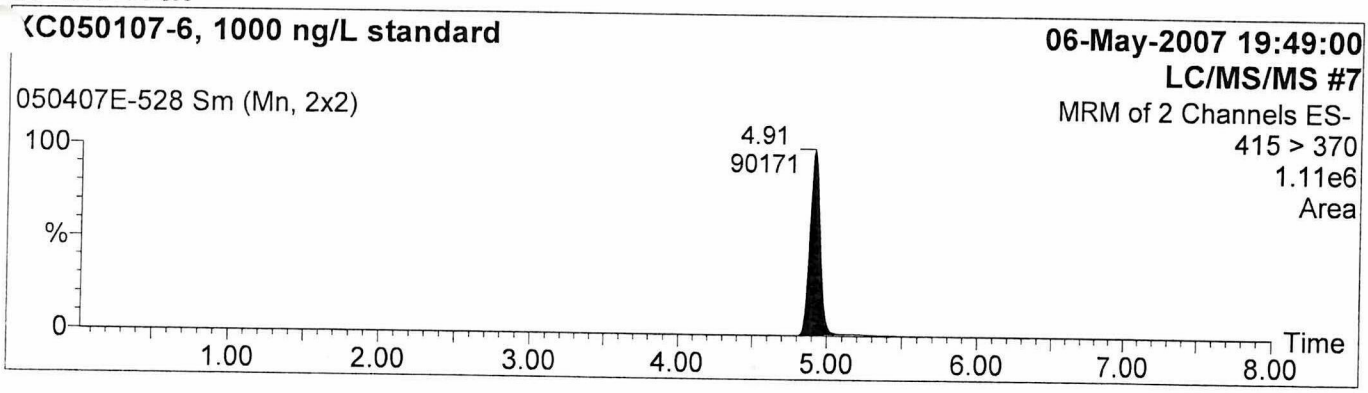
Printed: Thu May 10 12:48:24 2007

Name: 050407E-528
Text:

1: PFOA



2: 13C-PFOA



RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050407ER
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	Water	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	4.0 mL	Extraction Date:	5/4/2007
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	5/11/2007

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050407ER-501	0	-	-	0	-	-	-	-
XC050107-1	-	CS	050407ER-502	25	-	-	2865	-	-	-	-
XC050107-2	-	CS	050407ER-503	50	-	-	5729	-	-	-	-
XC050107-3	-	CS	050407ER-504	100	-	-	10906	-	-	-	-
XC050107-4	-	CS	050407ER-505	250	-	-	25049	-	-	-	-
XC050107-5	-	CS	050407ER-506	500	-	-	49703	-	-	-	-
XC050107-6	-	CS	050407ER-507	1000	-	-	95609	-	-	-	-
Methanol Wash	-	C	050407ER-508	-	-	-	0	-	-	-	-
L11423-13	SG-A-013	S	050407ER-509	-	100	100	26410	27600	-	-	-
L11423-17	SG-A-017	S	050407ER-510	-	100	100	25544	26700	-	-	-
Methanol Wash	-	C	050407ER-511	-	-	-	0	-	-	-	-
XC050107-1	-	CS	050407ER-512	25	-	-	2997	-	-	-	-
XC050107-2	-	CS	050407ER-513	50	-	-	5266	-	-	-	-
XC050107-3	-	CS	050407ER-514	100	-	-	9840	-	-	-	-
XC050107-4	-	CS	050407ER-515	250	-	-	24733	-	-	-	-
XC050107-5	-	CS	050407ER-516	500	-	-	44916	-	-	-	-
XC050107-6	-	CS	050407ER-517	1000	-	-	88824	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 793.284
Slope = 92.7058
Coef. Of Det. = 0.996584

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

Spreadsheet prepared by: MLC / 5/15/07

Masslynx - Sample List

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050407ER DI Water.SPL
Printed: Thu May 10 15:42:29 2007 MLC 5/10/07

Exygen STUDY NO. L11423

Page Position: (1, 1)

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/mL)	Conc B	Conc C	Test ID	DF
								L (RE) MLC 5/10/07				
1	94	050407ER-501	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---	0	1
2	95	050407ER-502	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
3	96	050407ER-503	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
4	97	050407ER-504	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
5	98	050407ER-505	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
6	99	050407ER-506	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
7	100	050407ER-507	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1
8	92	050407ER-508	---	---	Methanol Wash	---	Blank	---	---	---	0	1
9	21	050407ER-509	---	---	L11423-13, DF=100	---	Analyte	---	5000	---	0	100
10	22	050407ER-510	---	---	L11423-17, DF=100	---	Analyte	---	5000	---	0	100
11	92	050407ER-511	---	---	Methanol Wash	---	Blank	---	---	---	0	1
12	95	050407ER-512	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
13	96	050407ER-513	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
14	97	050407ER-514	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
15	98	050407ER-515	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
16	99	050407ER-516	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
17	100	050407ER-517	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1

	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA

Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/10/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/15/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Thu May 10 15:42:42 2007 MLC 6/6/07

Solvent Delay (mins) : 0.00

Function : 1 **MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Thursday, May 10, 2007 15:42:51

Printed: Thursday, May 10, 2007 15:42:53 MLC 5/10/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

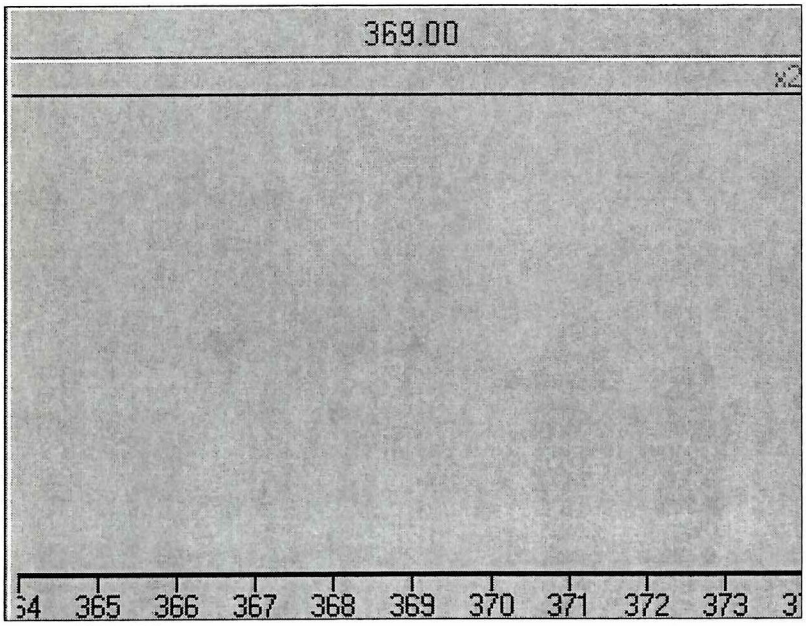
HP1100 Autosampler Initial Conditions

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	100

Tuning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Thu May 10 15:43:02 2007 MLC 5/10/07



Dau 413.00

SOURCE (ESP-)	Set	Rdbk	Analyser	Set	Rdbk
Capillary	3.00	-2.94	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	102	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	92.6
Gas Cell	2.3e-3	Desolvation	673.6

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050407ER
Extraction Date: 05/04/07
Analyzed on: 05/11/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2865
XC050107-2	50	5729
XC050107-3	100	10906
XC050107-4	250	25049
XC050107-5	500	49703
XC050107-6	1000	95609

Coefficient of Determination: 0.999694445

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999847211
R Square	0.999694445
Adjusted R Square	0.999618056
Standard Error	698.71477
Observations	6

MLC
5/15/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	6389072630	6389072630	13086.93597	3.5015E-08
Residual	4	1952809.319	488202.3299		
Total	5	6391025440			

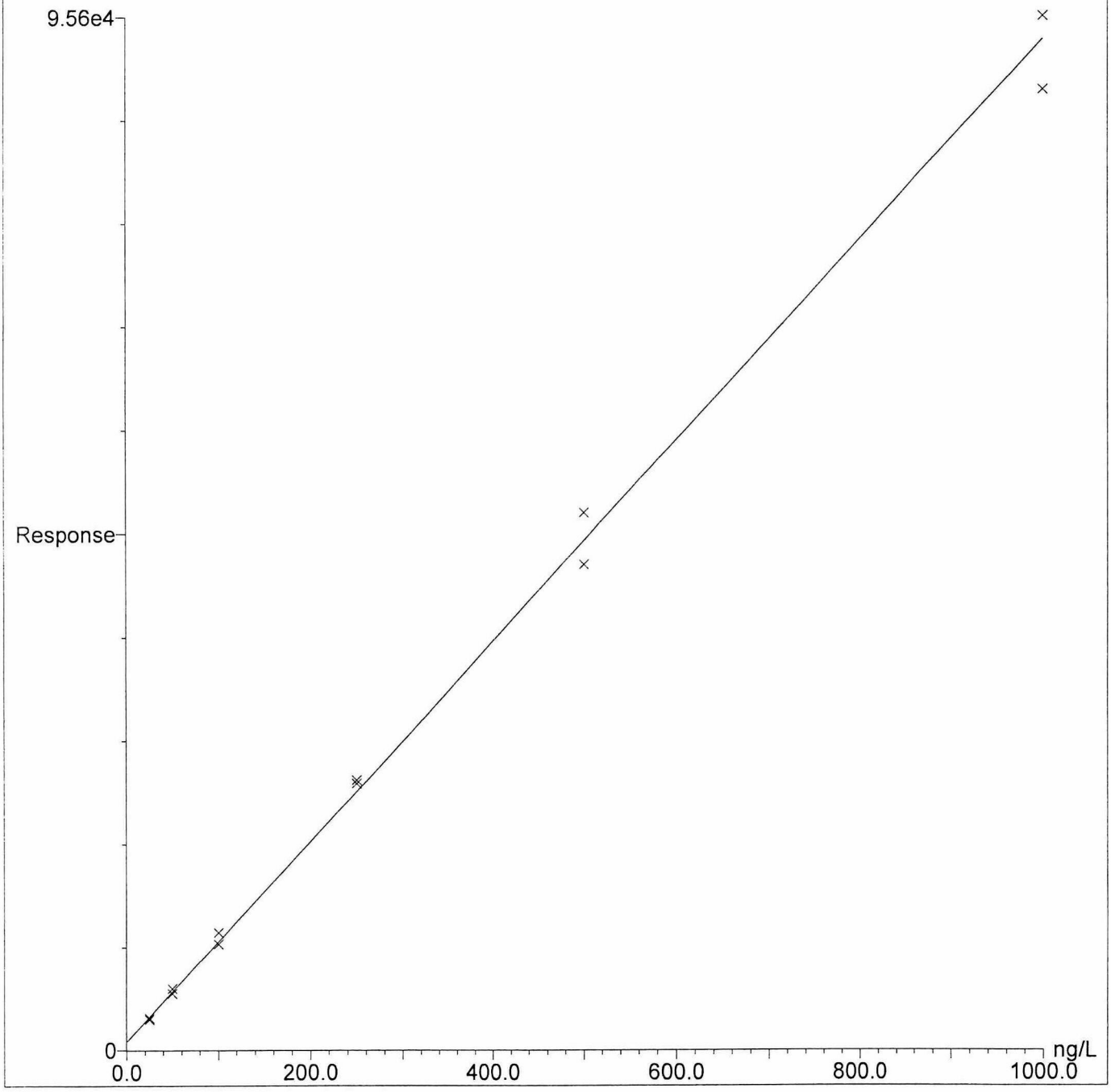
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	1166.278064	390.3117478	2.988068052	0.040415183	82.59892213	2249.957206	82.59892213	2249.957206
X Variable 1	94.9939385	0.830380048	114.3981467	3.5015E-08	92.68843388	97.29944312	92.68843388	97.29944312

Quantify Calibration Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050407ER DI Water
Last modified: Tue May 15 08:32:57 2007
Printed: Tue May 15 10:33:21 2007

MLC 5/15/07

Compound 1 name: PFOA
Coefficient of Determination: 0.996584
Calibration curve: 92.7058 * x + 793.284
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

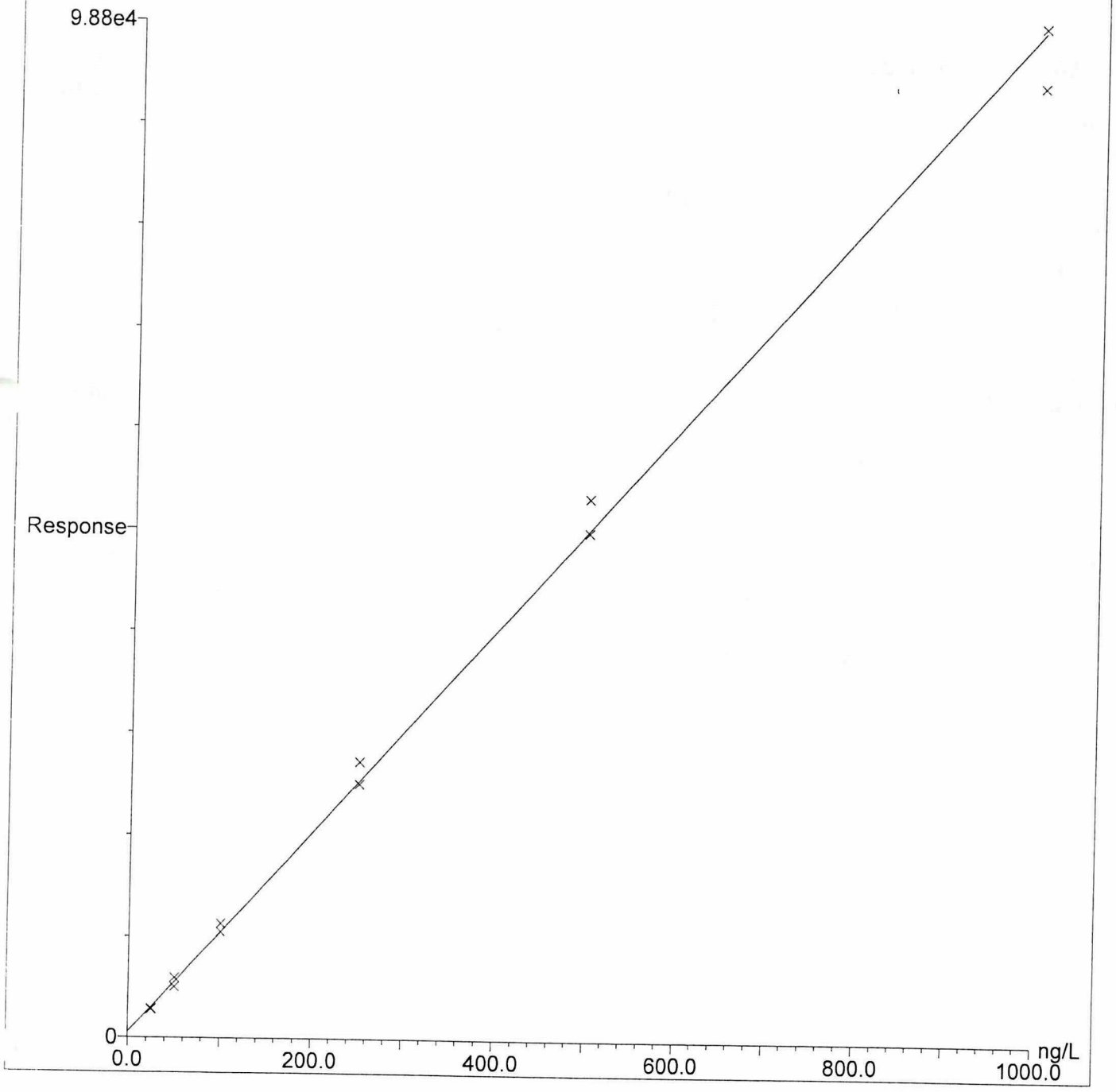


Quantify Calibration Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050407ER DI Water
Last modified: Tue May 15 08:32:57 2007
Printed: Tue May 15 10:33:21 2007

MLC 5/15/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.996671
Calibration curve: 97.8276 * x + 533.615
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

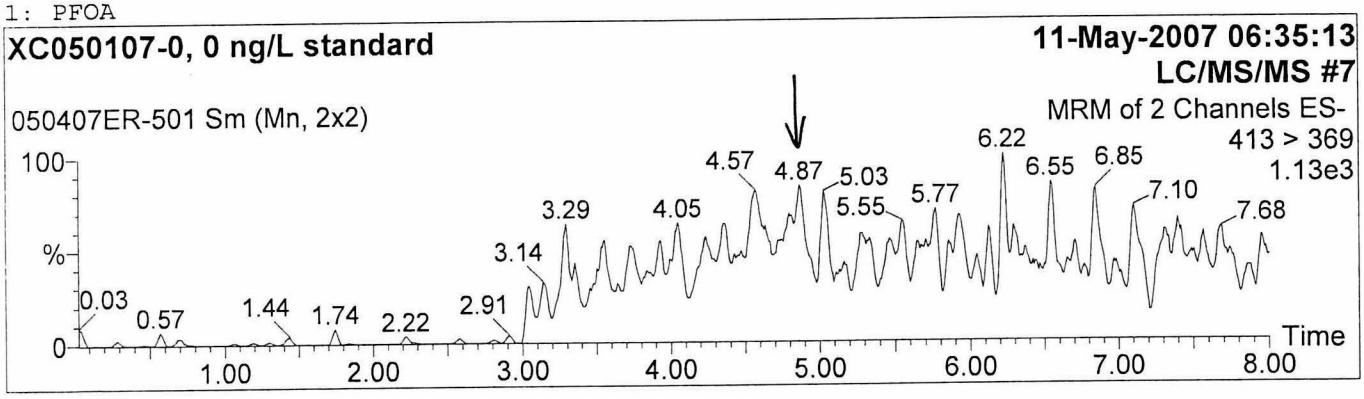
Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

Printed: Tue May 15 10:33:22 2007

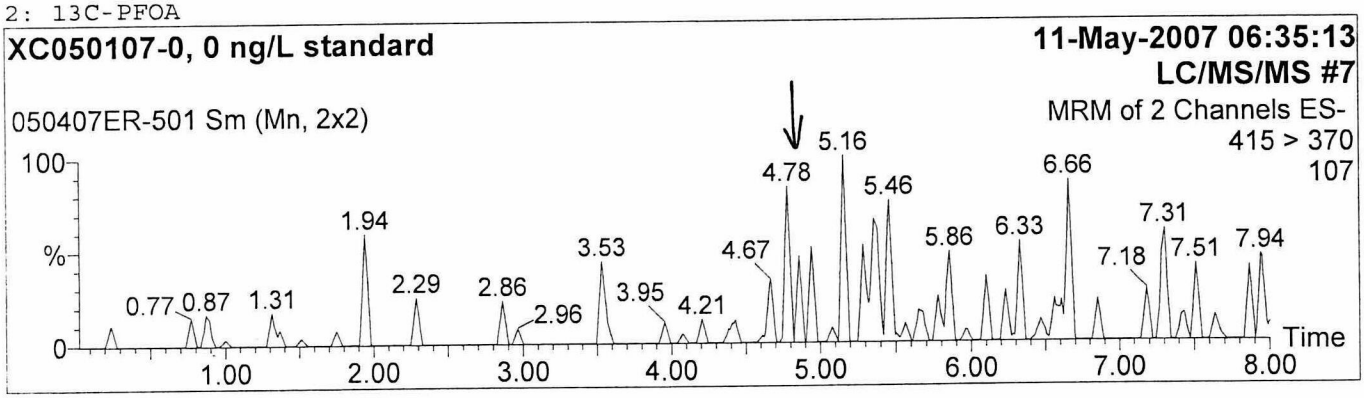
Initials MLC
Date 5/15/07
Run# 050407ER-501 To 050407ER-517

Name: 050407ER-501
Text:

1: PFOA



2: 13C-PFOA



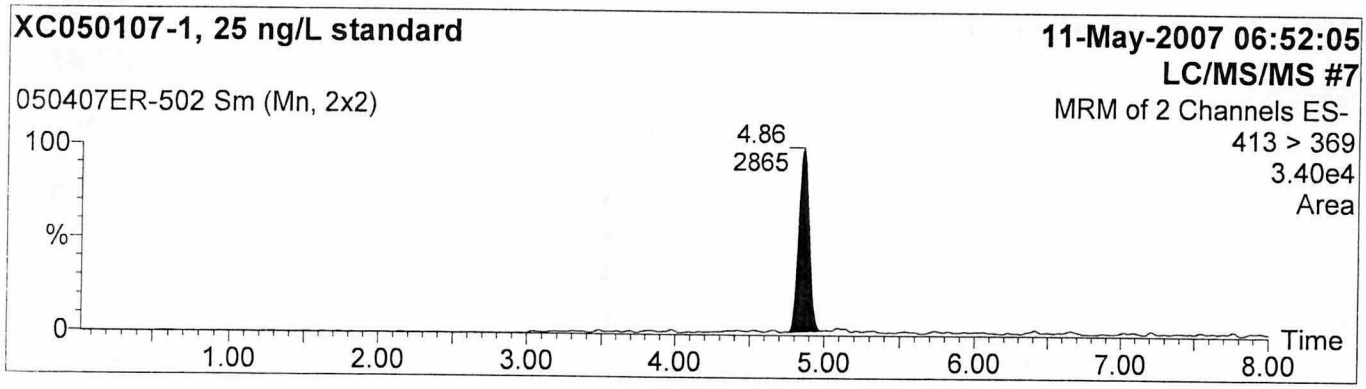
Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

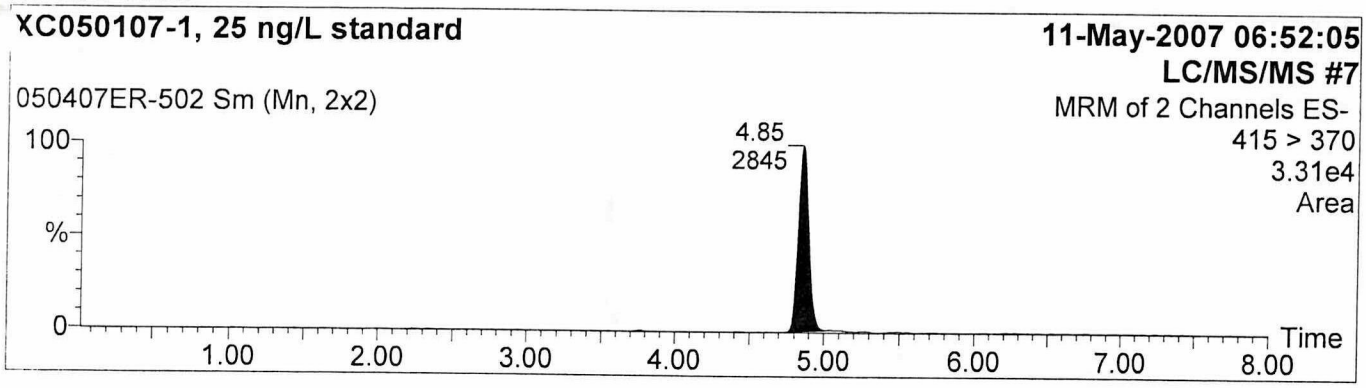
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-502
Text:

1: PFOA



2: 13C-PFOA



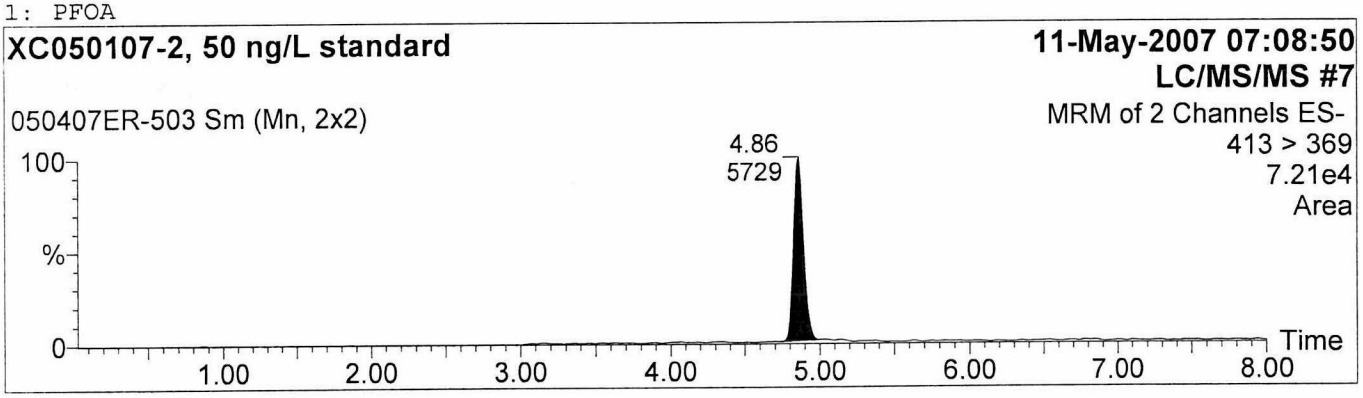
Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

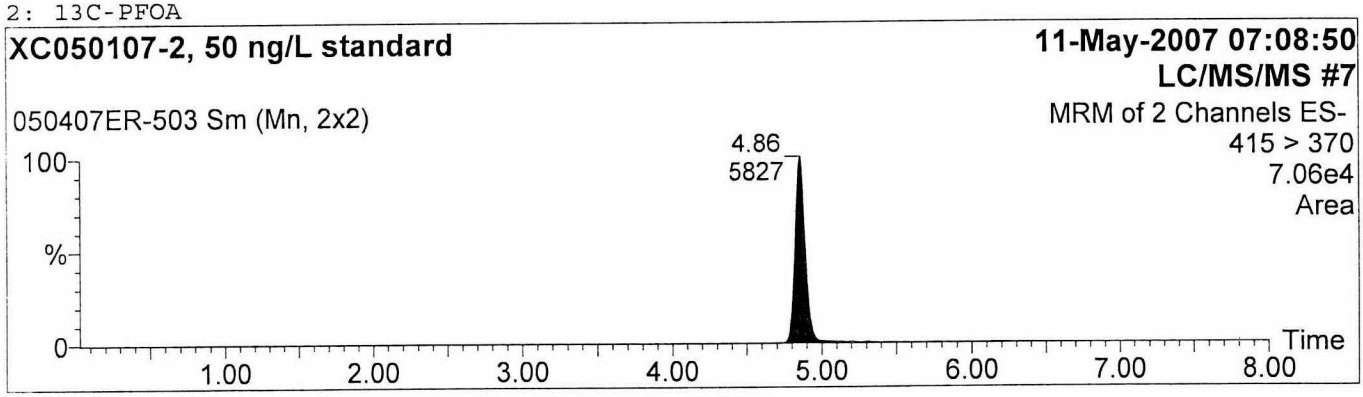
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-503
Text:

1: PFOA



2: 13C-PFOA



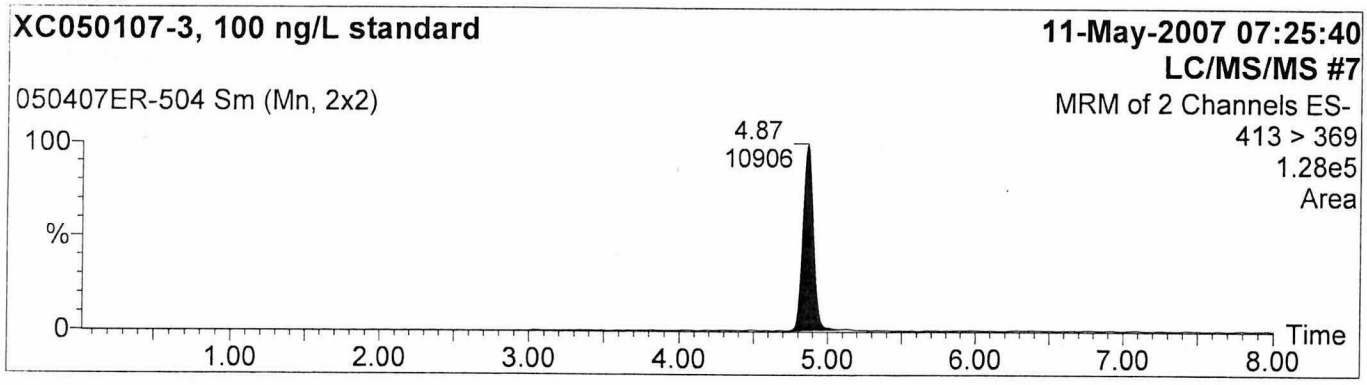
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

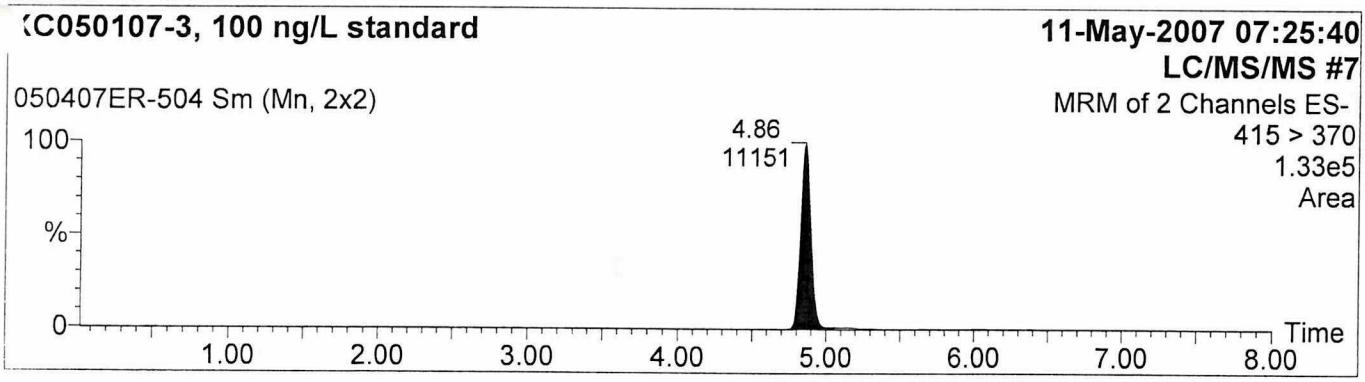
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-504
Text:

1: PFOA



2: 13C-PFOA

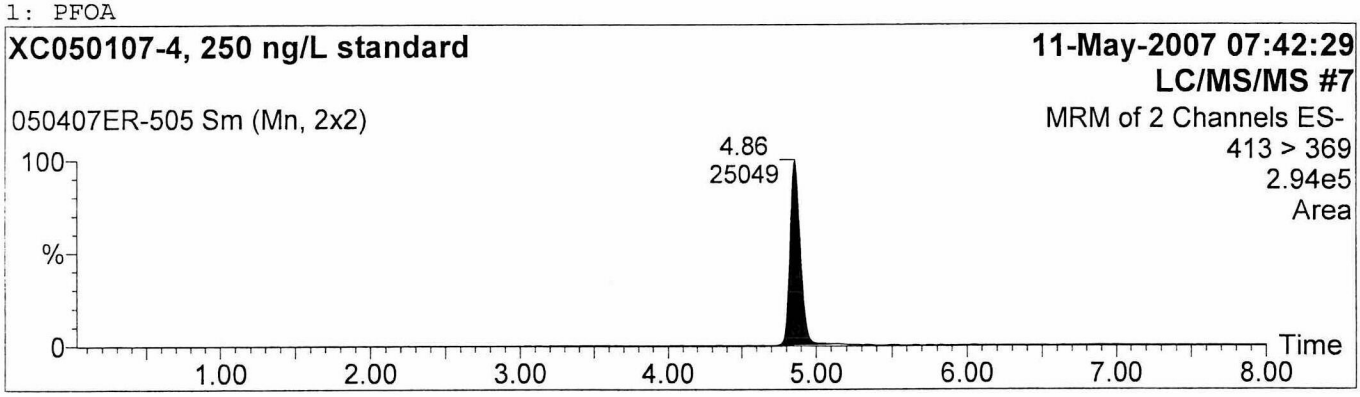


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

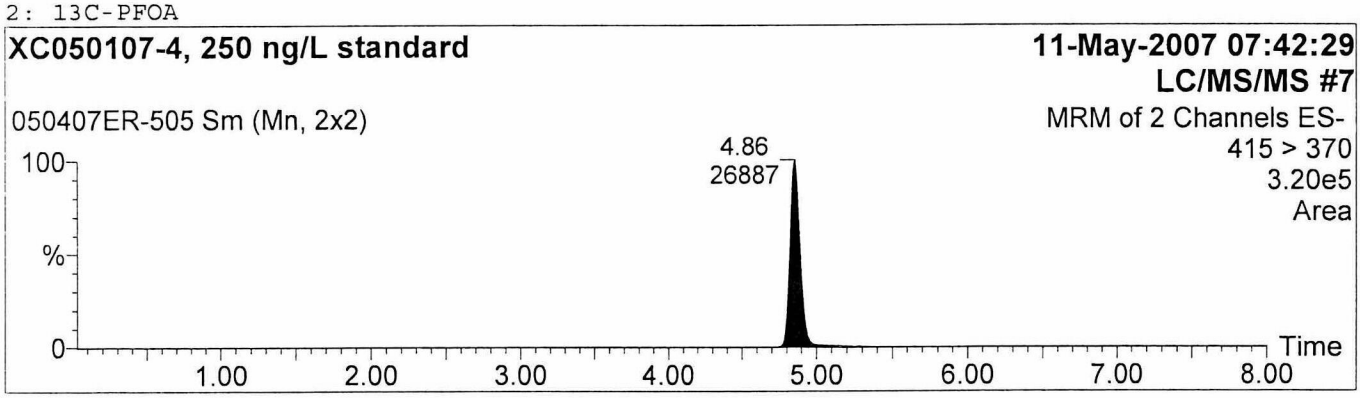
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-505
Text:

1: PFOA



2: 13C-PFOA



Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
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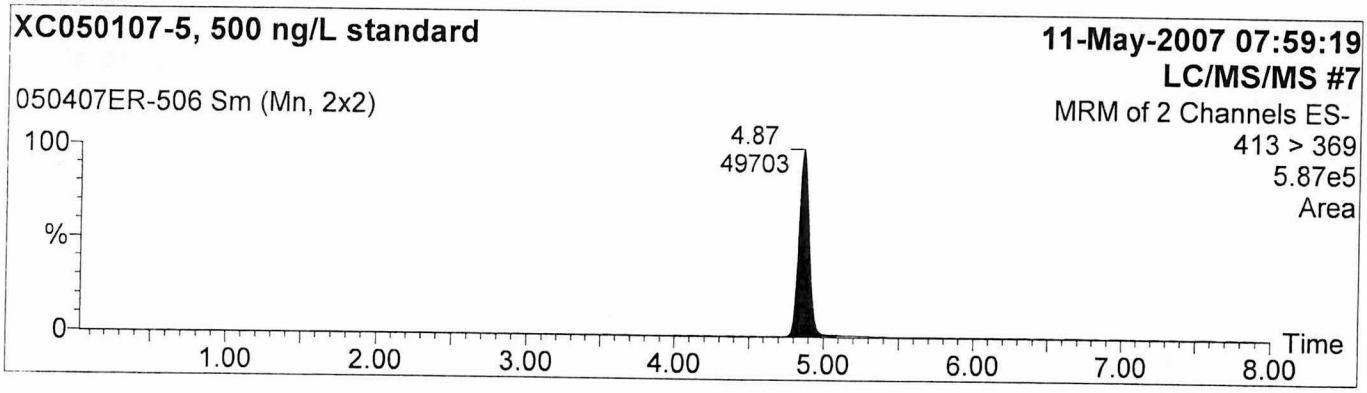
Last modified: Tue May 15 08:29:30 2007
Job Code:

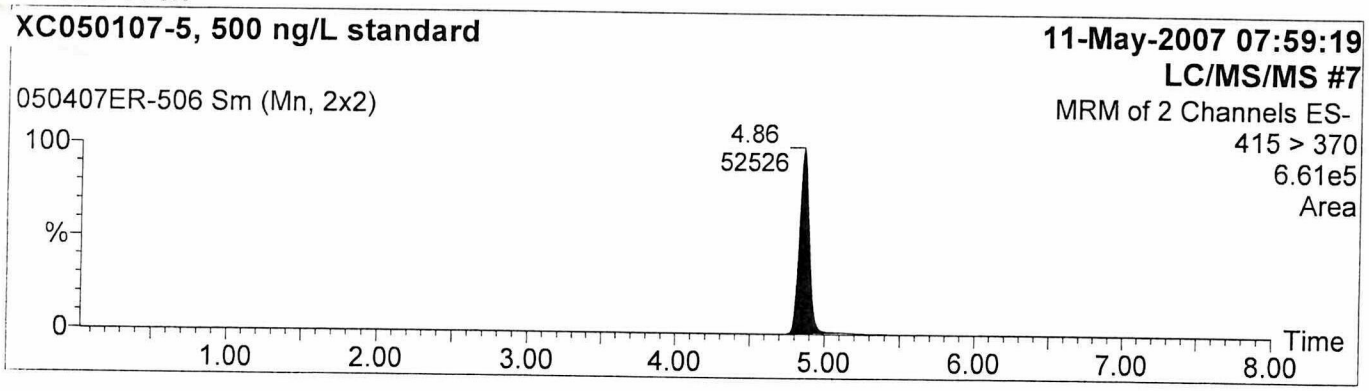
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-506
Text:

1: PFOA



2: 13C-PFOA



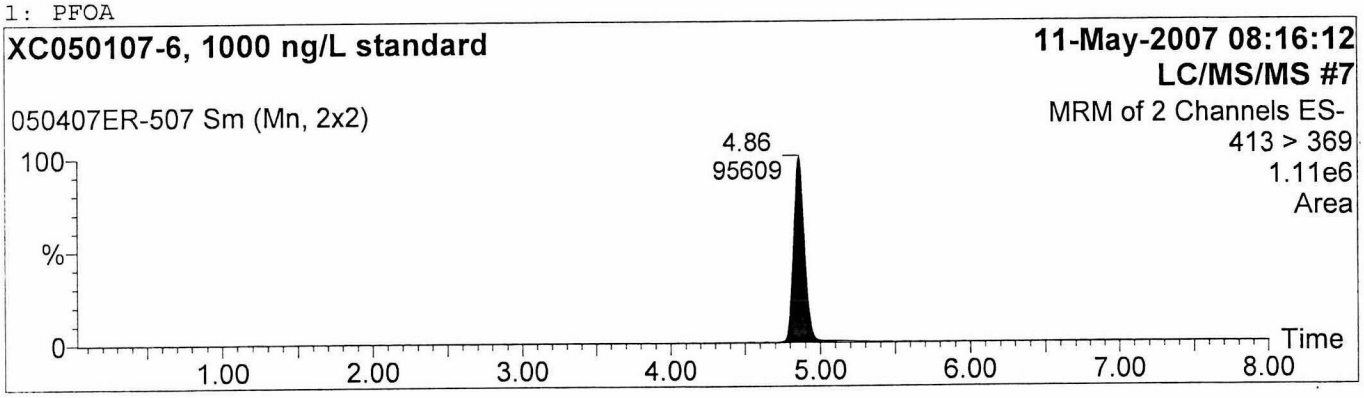
Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

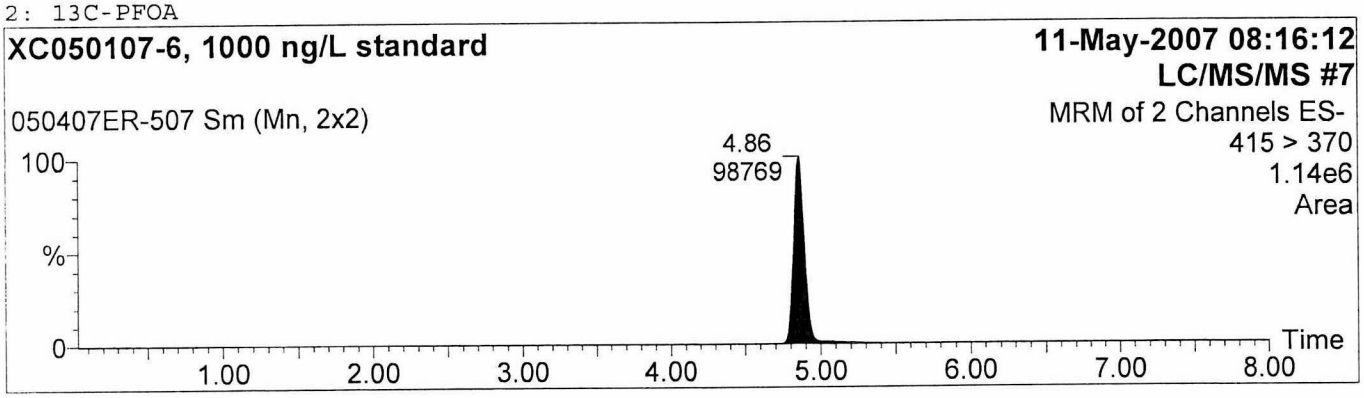
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-507
Text:

1: PFOA



2: 13C-PFOA



Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
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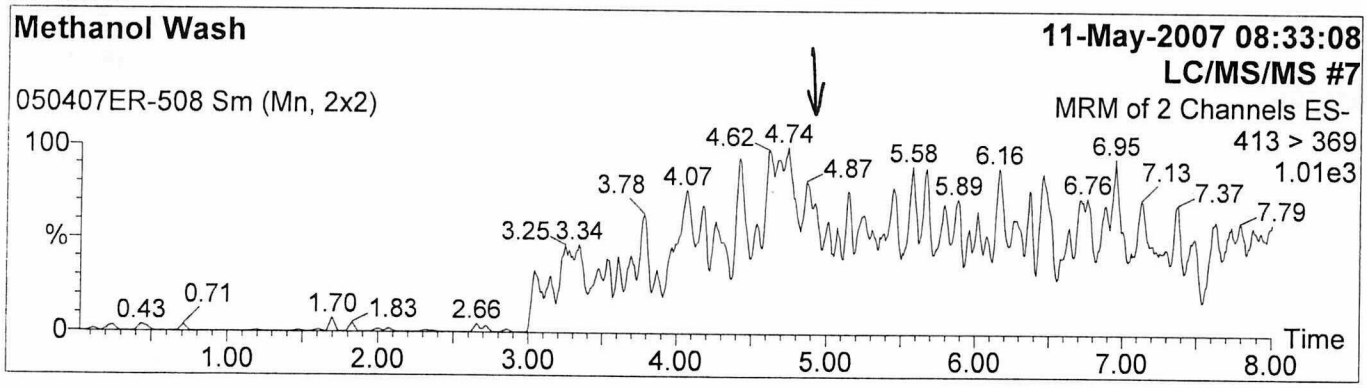
Last modified: Tue May 15 08:29:30 2007
Job Code:

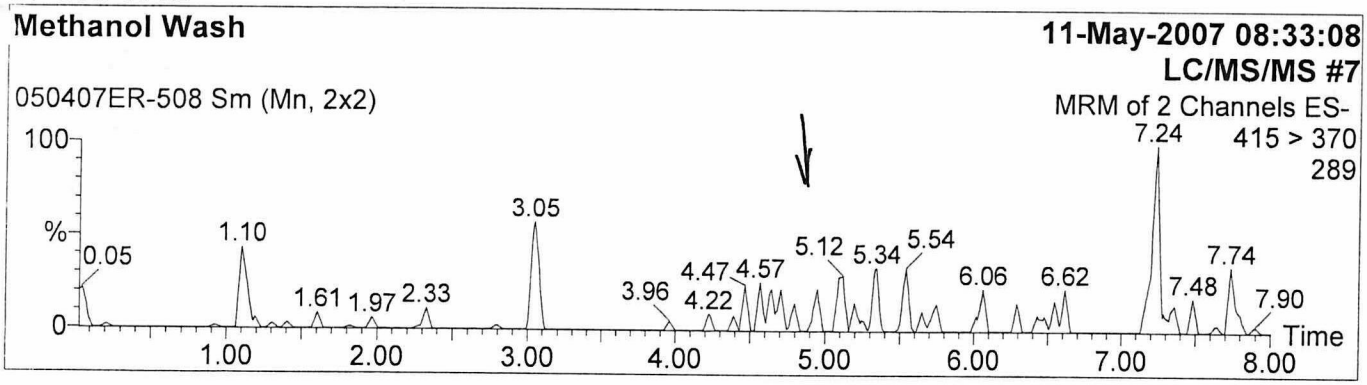
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-508
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
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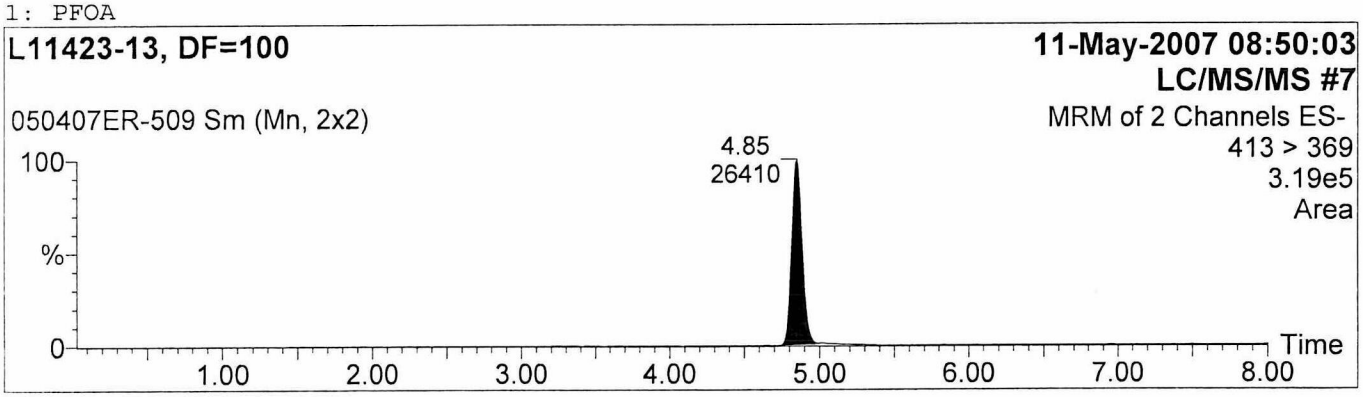
Last modified: Tue May 15 08:29:30 2007
Job Code:

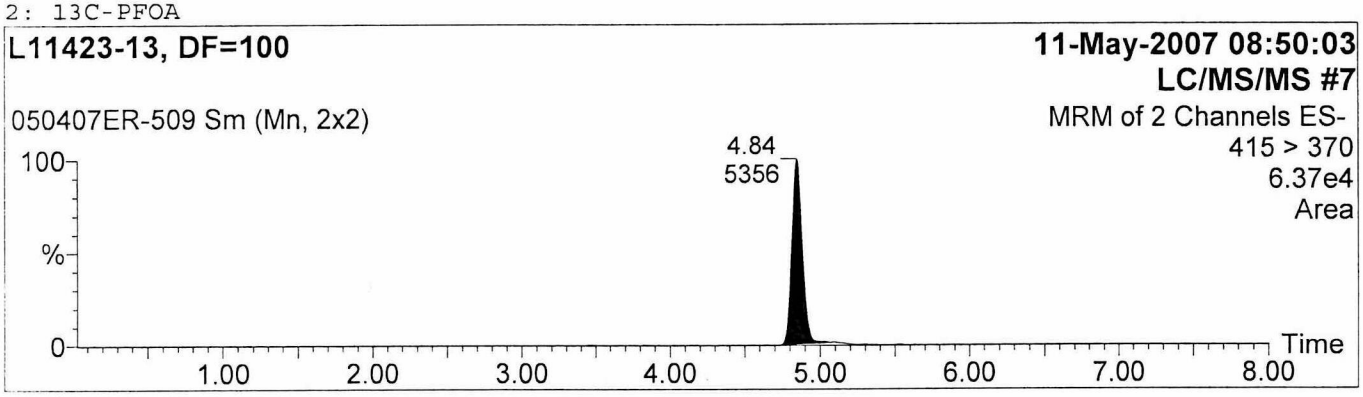
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-509
Text:

1: PFOA



2: 13C-PFOA



Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
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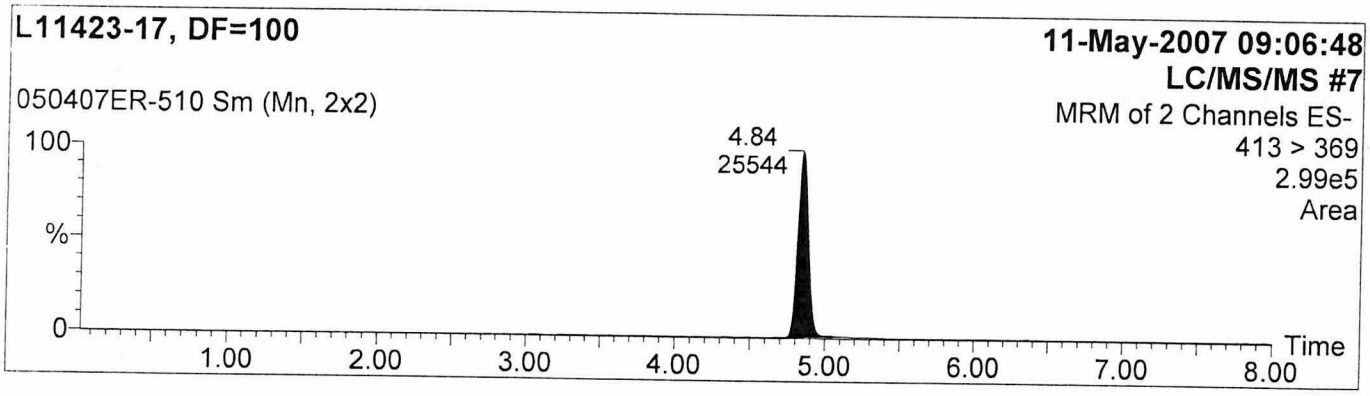
Last modified: Tue May 15 08:29:30 2007
Job Code:

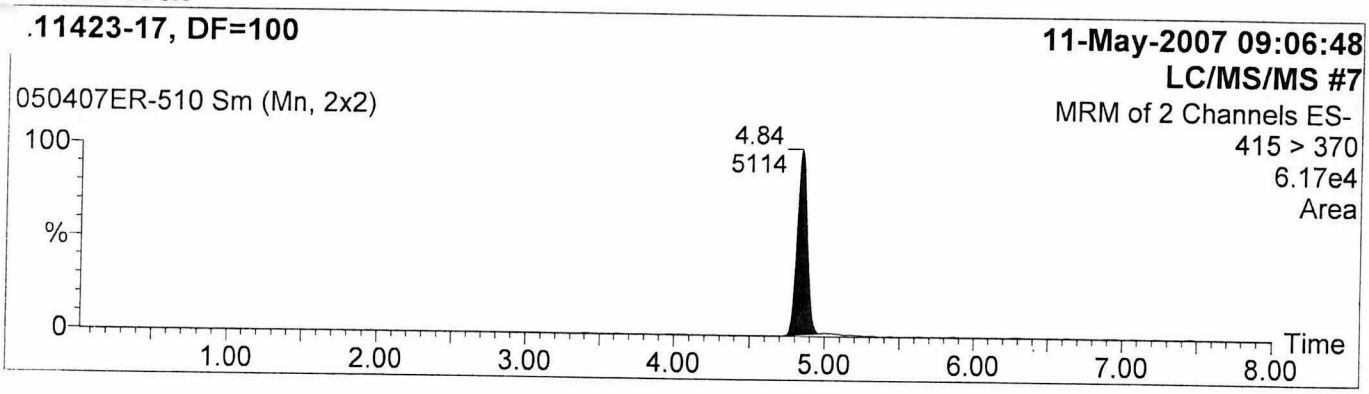
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-510
Text:

1: PFOA



2: 13C-PFOA



Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
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Last modified: Tue May 15 08:29:30 2007
Job Code:

Printed: Tue May 15 10:33:22 2007

Name: 050407ER-511
Text:

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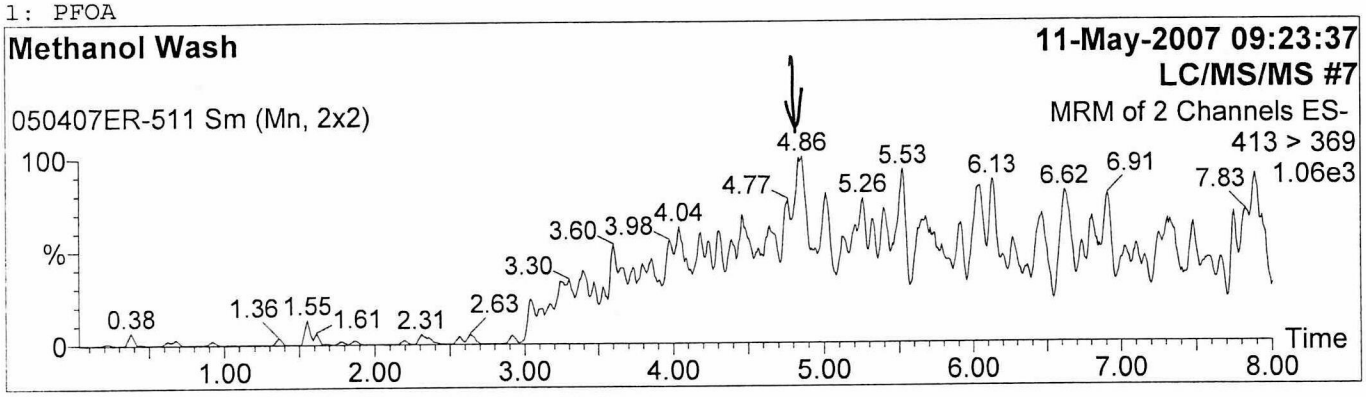
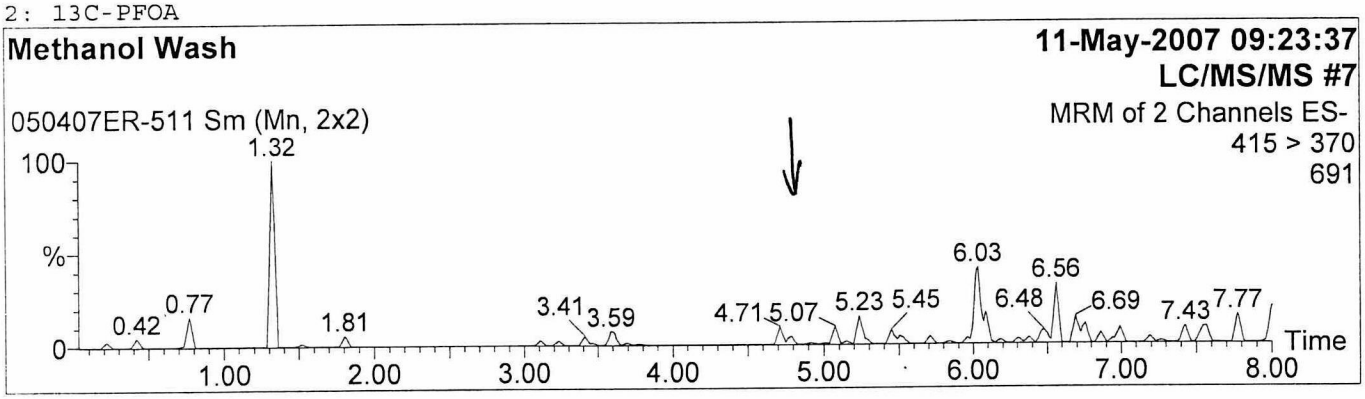


2: 13C-PFOA



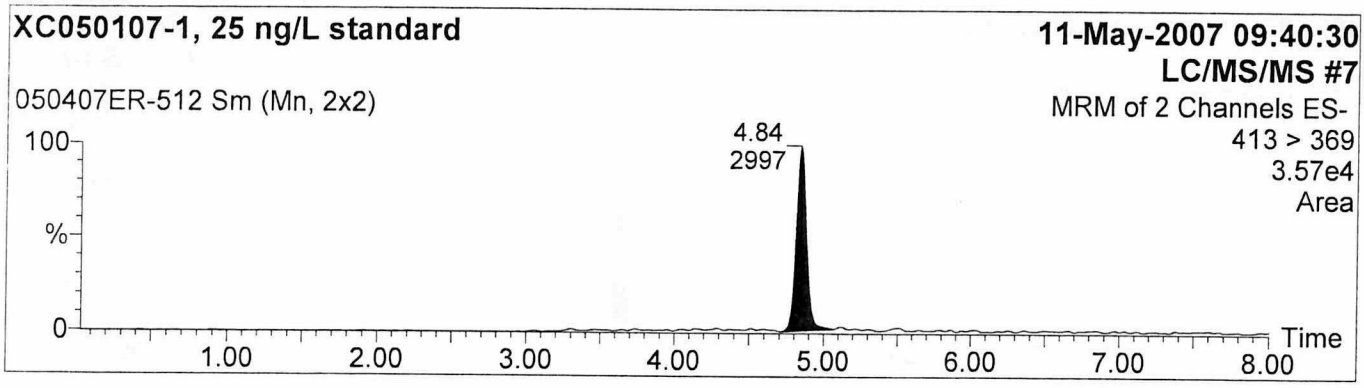
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

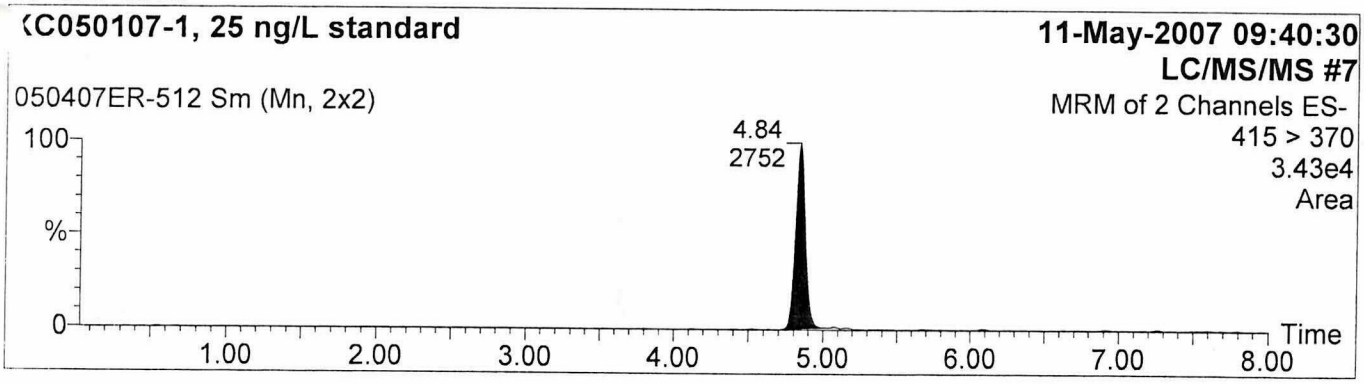
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-512
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
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Last modified: Tue May 15 08:29:30 2007
Job Code:

Printed: Tue May 15 10:33:22 2007

Name: 050407ER-513
Text:

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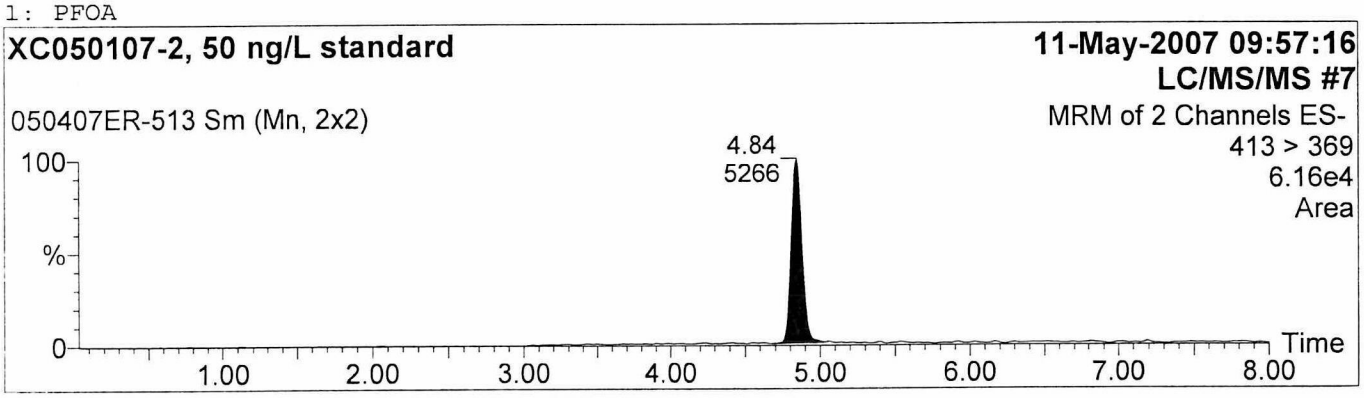
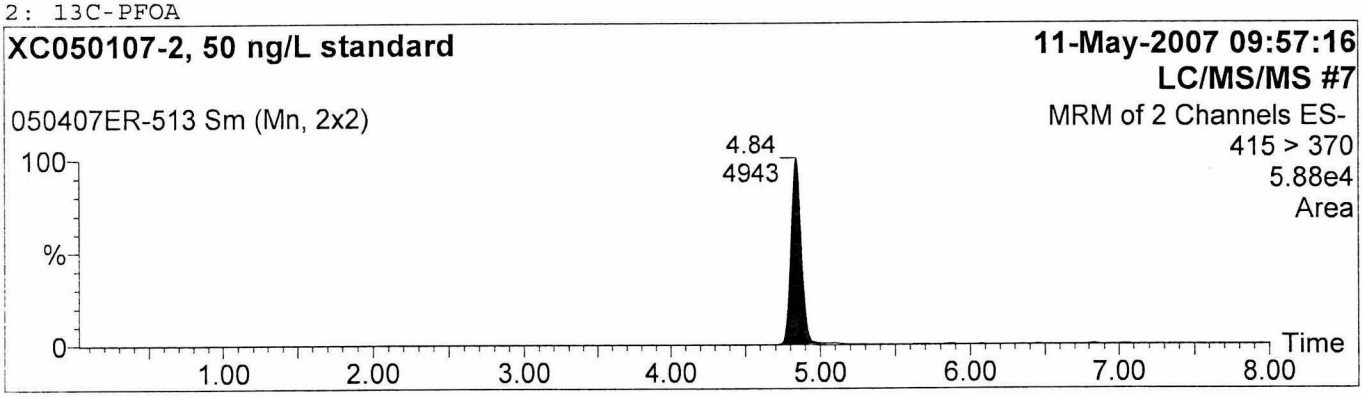


2: 13C-PFOA



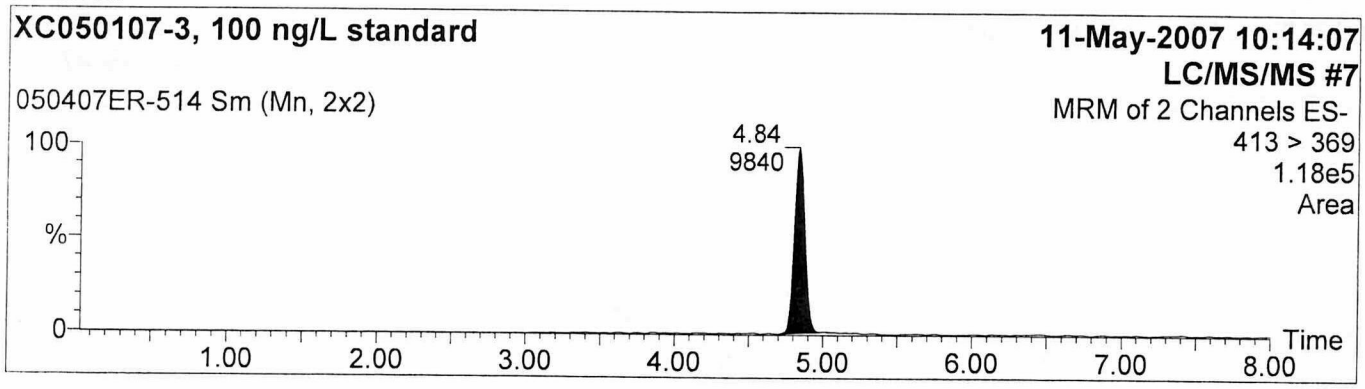
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

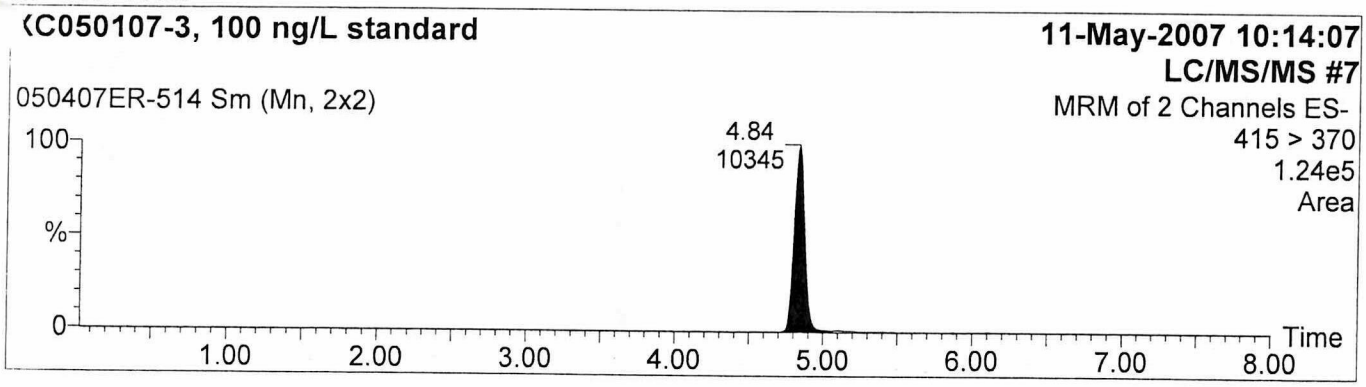
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-514
Text:

1: PFOA



2: 13C-PFOA



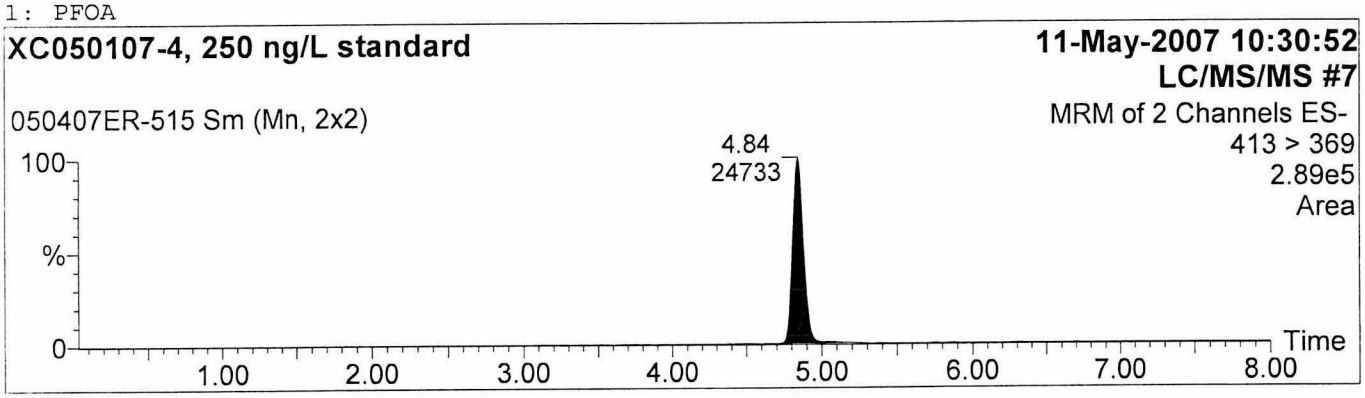
Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

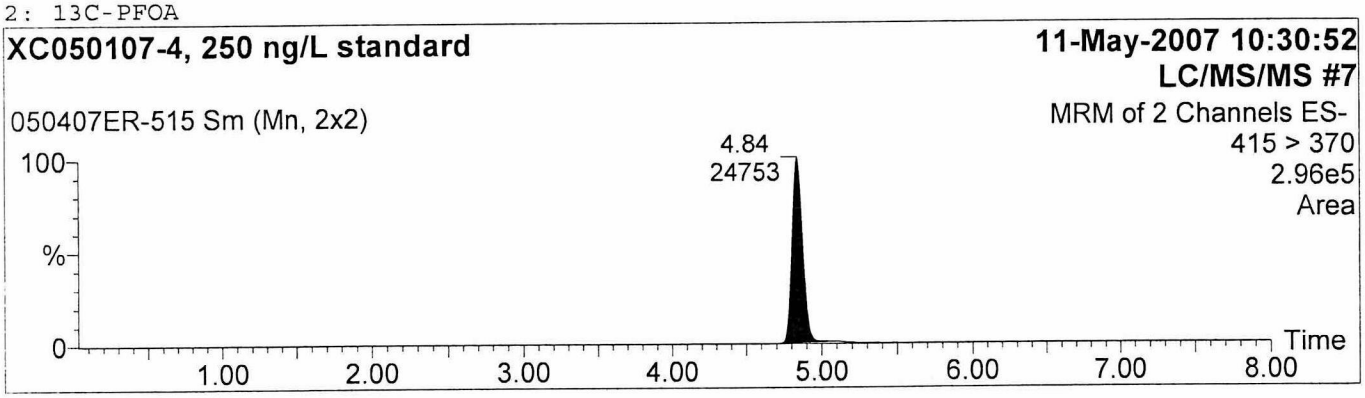
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-515
Text:

1: PFOA



2: 13C-PFOA



Quantify Sample Report
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

Printed: Tue May 15 10:33:22 2007

Name: 050407ER-516
Text:

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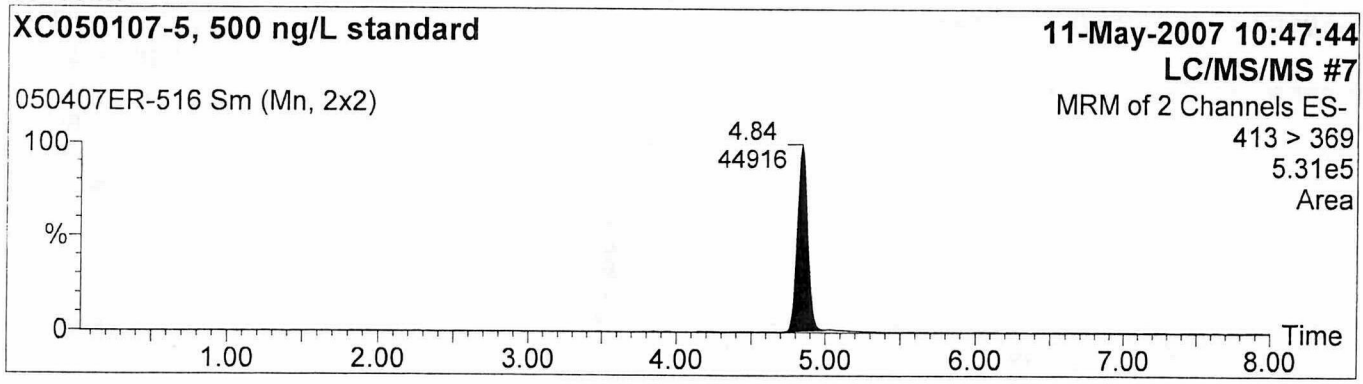
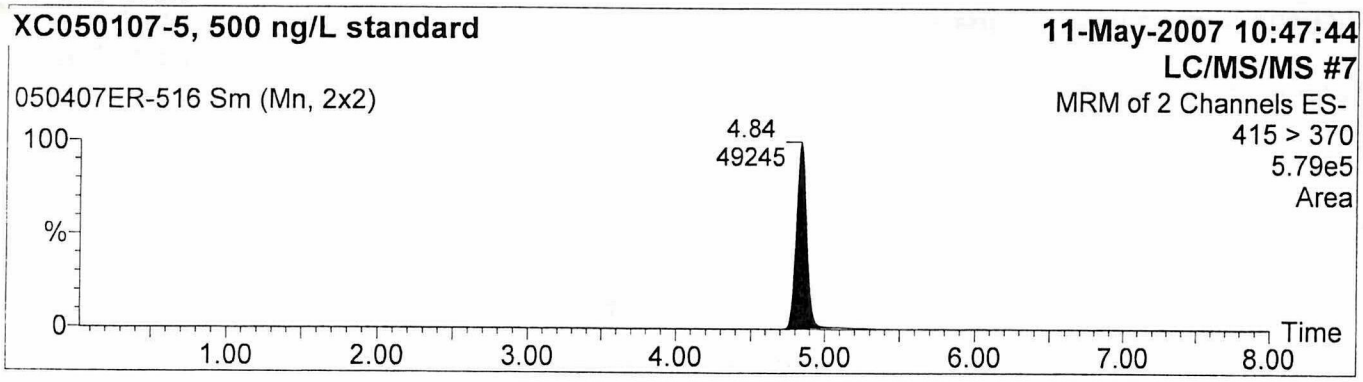


2: 13C-PFOA



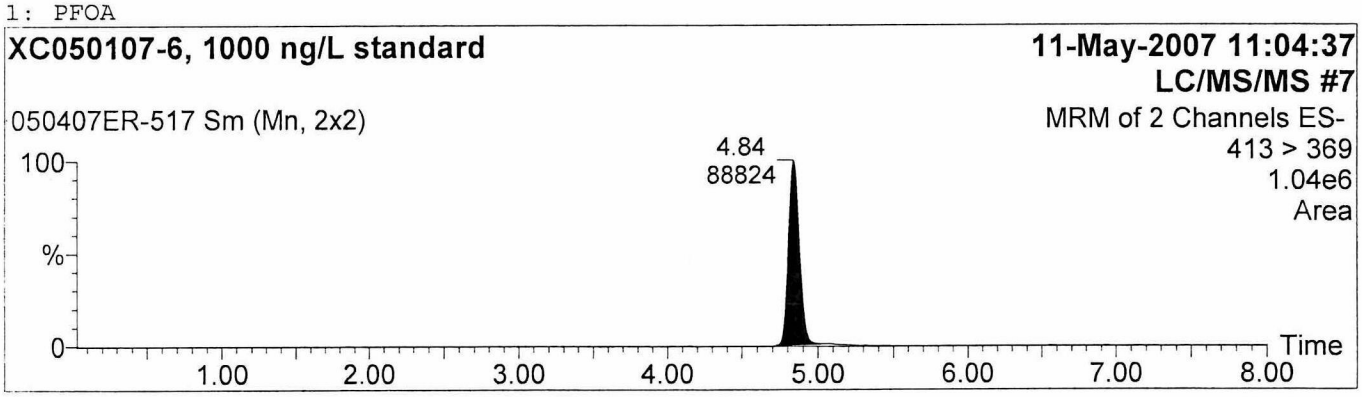
Study No: L11423, Set No: 050407ER. Ext.Date: 05/04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407ER DI Water
Last modified: Tue May 15 08:27:48 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

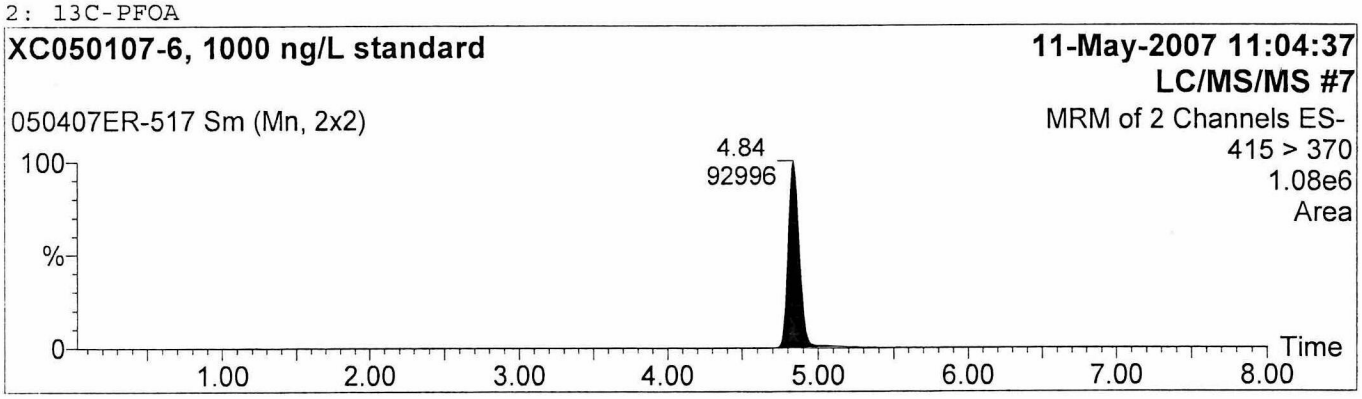
Printed: Tue May 15 10:33:22 2007

Name: 050407ER-517
Text:

1: PFOA



2: 13C-PFOA



Login

10X 1000X

Samples

Sample ID	Container	Matrix	Fraction	Sample	Date Sampled	Date Due
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L0011423-0002	C0250358	LIQUID	Impinger solution	SG-A-002 NaOH	04/26/2007	05/07/2007
L0011423-0003	C0250359	LIQUID	Impinger solution	SG-A-003 Buffer	04/26/2007	05/07/2007
L0011423-0004	C0250360	LIQUID	Impinger solution	SG-A-004 MeOH/filter	04/26/2007	05/07/2007
L0011423-0005	C0250361	LIQUID	Impinger solution	SG-A-005 DI	04/26/2007	05/07/2007
L0011423-0006	C0250362	LIQUID	Impinger solution	SG-A-006 NaOH	04/26/2007	05/07/2007
L0011423-0007	C0250363	LIQUID	Impinger solution	SG-A-007 Buffer	04/26/2007	05/07/2007
L0011423-0008	C0250364	LIQUID	Impinger solution	SG-A-008 MeOH/filter	04/26/2007	05/07/2007
L0011423-0009	C0250365	LIQUID	Impinger solution	SG-A-009 DI	04/26/2007	05/07/2007
L0011423-0010	C0250366	LIQUID	Impinger solution	SG-A-010 NaOH	04/26/2007	05/07/2007
L0011423-0011	C0250367	LIQUID	Impinger solution	SG-A-011 Buffer	04/26/2007	05/07/2007
L0011423-0012	C0250368	LIQUID	Impinger solution	SG-A-012 MeOH/filter	04/26/2007	05/07/2007
L0011423-0013	C0250369	LIQUID	Impinger solution	SG-A-013 DI	04/26/2007	05/07/2007
L0011423-0014	C0250370	LIQUID	Impinger solution	SG-A-014 NaOH	04/26/2007	05/07/2007
L0011423-0015	C0250371	LIQUID	Impinger solution	SG-A-015 Buffer	04/26/2007	05/07/2007
L0011423-0016	C0250372	LIQUID	Impinger solution	SG-A-016 MeOH/filter	04/26/2007	05/07/2007

L0011423-0017	C0250373	LIQUID	Impinger solution	SG-A-017 DI	04/26/2007	05/07/2007
L0011423-0018	C0250374	LIQUID	Impinger solution	SG-A-018 NaOH	04/26/2007	05/07/2007
L0011423-0019	C0250375	LIQUID	Impinger solution	SG-A-019 Buffer	04/26/2007	05/07/2007
L0011423-0020	C0250376	LIQUID	Impinger solution	SG-A-020 MeOH/filter	04/26/2007	05/07/2007
L0011423-0021	C0250377	LIQUID	Dispersion	SG-L-001	04/26/2007	05/07/2007
L0011423-0022	C0250378	LIQUID	Dispersion	SG-L-002	04/26/2007	05/07/2007

Login Reviewed By: _____ Date/Time: _____

Solvay

SS0019495

MPI RESEARCH

3058 Research Drive
State College, PA 16801

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

Login

Login Group: L0011423

Login #: 11534
Project: P0003133
Company Name: Barr Engineering
Submitted By: Tim Russell
Login Type: Immediate Receipt of Samples
Started: True
Date Start: 04/27/2007
Due Date: 05/07/2007
Login Initiated*: 04/27/2007

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

* Dates entered into "Login Initiated" field prior to 1/5/06 reflect dates of receipt. The field was formerly called "Received Date"

Received By: Ammerman, Mark
Spread Sample:
Label:
Exygen SD/PI: Risha, Karen
Project Title/Type: Analysis of Samples for PFOA/APFO by LCMSMS / ROUTINE
Login Notes:

Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By
PK0013603		Received Date: 4/27/07 9:00 Package & Contents Uncompromised		Wet Ice 2.0	RECEIVED Ammerman, Mark

Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0250357	139.30 g		500 ml Clear Plastic Narrow	NONE		
C0250358	151.60 g		500 ml Clear Plastic Narrow	NONE		
C0250359	158.10 g		500 ml Clear Plastic Narrow	NONE		
C0250360	121.40 g		500 ml Clear Plastic Narrow	NONE		
C0250361	155.20 g		500 ml Clear Plastic Narrow	NONE		
C0250362	156.10 g		500 ml Clear Plastic Narrow	NONE		
C0250363	156.60 g		500 ml Clear Plastic Narrow	NONE		
C0250364	129.50 g		500 ml Clear Plastic Narrow	NONE		
C0250365	158.00 g		500 ml Clear Plastic Narrow	NONE		
C0250366	158.40 g		500 ml Clear Plastic Narrow	NONE		
C0250367	156.70 g		500 ml Clear Plastic Narrow	NONE		
C0250368	305.40 g		500 ml Clear Plastic Narrow	NONE		
0369	154.80 g		500 ml Clear Plastic Narrow	NONE		

Login

Login Group: L0011423

Login #:	11534	Conform COC Sample:	True
Project:	P0003133	Conform COC:	True
Company Name:	Barr Engineering	Conform Sample:	True
Submitted By:	Tim Russell	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	04/27/2007		
Due Date:	05/07/2007		
Login Initiated*:	04/27/2007		

* Dates entered into "Login Initiated" field prior to 1/5/06 reflect dates of receipt. The field was formerly called "Received Date"

Received By: Ammerman, Mark
Spread Sample:
Label:
Exygen SD/PI: Risha, Karen
Project Title/Type: Analysis of Samples for PFOA/APFO by LCMSMS / ROUTINE
Login Notes:

Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By
PK0013603		Received Date: 4/27/07 9:00 Package & Contents Uncompromised		Wet Ice 2.0	RECEIVED Ammerman, Mark

Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0250370	160.40 g		500 ml Clear Plastic Narrow	NONE		
C0250371	158.10 g		500 ml Clear Plastic Narrow	NONE		
C0250372	341.10 g		500 ml Clear Plastic Narrow	NONE		
C0250373	156.40 g		500 ml Clear Plastic Narrow	NONE		
C0250374	160.00 g		500 ml Clear Plastic Narrow	NONE		
C0250375	155.90 g		500 ml Clear Plastic Narrow	NONE		
C0250376	368.30 g		500 ml Clear Plastic Narrow	NONE		
C0250377	380.10 g		500 ml Clear Plastic Narrow	NONE		
C0250378	387.30 g		500 ml Clear Plastic Narrow	NONE		

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050207A
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	NaOH	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	4.0 mL	Extraction Date:	05/02/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/02-03/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050207A-101	0	-	-	0	-	-	-	-
XC050107-1	-	CS	050207A-102	25	-	-	2834	-	-	-	-
XC050107-2	-	CS	050207A-103	50	-	-	5167	-	-	-	-
XC050107-3	-	CS	050207A-104	100	-	-	9481	-	-	-	-
XC050107-4	-	CS	050207A-105	250	-	-	24504	-	-	-	-
XC050107-5	-	CS	050207A-106	500	-	-	45324	-	-	-	-
XC050107-6	-	CS	050207A-107	1000	-	-	93511	-	-	-	-
Methanol Wash	-	C	050207A-108	-	-	-	130	-	-	-	-
Reagent Control	NA	C	050207A-109	-	-	1	0	ND	ND	-	-
Reagent Spk A	NA	LCS	050207A-110	-	-	1	5844	56.5	58.8	50	113
Reagent Spk B	NA	LCS	050207A-111	-	-	1	48704	518	540	500	104
L11423-14 Spk C	SG-A-014	LF	050207A-112	-	100	10000	45948	^	-	5000000	-
L11423-14 Spk C	SG-A-014	LF	050207A-113	-	100	10	14424875	*	-	5000000	-
Methanol Wash	-	C	050207A-114	-	-	-	3411	-	-	-	-
XC050107-1	-	CS	050207A-115	25	-	-	2868	-	-	-	-
XC050107-2	-	CS	050207A-116	50	-	-	5523	-	-	-	-
L11423-2	SG-A-002	S	050207A-117	-	100	10	266	ND	ND	-	-
L11423-6	SG-A-006	S	050207A-118	-	100	10	317	ND	ND	-	-
L11423-10	SG-A-010	S	050207A-119	-	100	10	144569	**	-	-	-
L11423-14	SG-A-014	S	050207A-120	-	100	10	150306	**	-	-	-
L11423-14 Rep	SG-A-014	S	050207A-121	-	100	10	150636	**	-	-	-
L11423-18	SG-A-018	S	050207A-122	-	100	10	146576	**	-	-	-
XC050107-3	-	CS	050207A-123	100	-	-	10014	-	-	-	-
XC050107-4	-	CS	050207A-124	250	-	-	24788	-	-	-	-
XC050107-5	-	CS	050207A-125	500	-	-	48104	-	-	-	-
XC050107-6	-	CS	050207A-126	1000	-	-	92318	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 607.210
Slope = 92.7662
Coef. Of Det. = 0.999427

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

Spreadsheet prepared by: MLC / 5/3/07

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.
**Sample requires dilution. See data set 050407C.
^Sample requires a lower spiking level. For re-extraction and analysis see data set 050407C.

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050207A
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	^{13}C-PFOA	Matrix:	NaOH	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	415 -> 370	Sample Volume Extracted:	4.0 mL	Extraction Date:	05/02/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/02-03/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	13C-PFOA Found (ng/L)	Amount 13C-PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050207A-101	0	-	-	0	-	-	-
XC050107-1	-	CS	050207A-102	25	-	-	2547	-	-	-
XC050107-2	-	CS	050207A-103	50	-	-	4921	-	-	-
XC050107-3	-	CS	050207A-104	100	-	-	9089	-	-	-
XC050107-4	-	CS	050207A-105	250	-	-	23083	-	-	-
XC050107-5	-	CS	050207A-106	500	-	-	45758	-	-	-
XC050107-6	-	CS	050207A-107	1000	-	-	89508	-	-	-
Methanol Wash	-	C	050207A-108	-	-	-	0	-	-	-
Reagent Control	NA	C	050207A-109	-	-	1	0	ND	-	-
Reagent Spk A	NA	LCS	050207A-110	-	-	1	5024	52.1	50	104
Reagent Spk B	NA	LCS	050207A-111	-	-	1	47046	512	500	102
L11423-14 Spk C	SG-A-014	LF	050207A-112	-	100	10000	98	*	5000	-
L11423-14 Spk C	SG-A-014	LF	050207A-113	-	100	10	27745	3010	5000	60
Methanol Wash	-	C	050207A-114	-	-	-	0	-	-	-
XC050107-1	-	CS	050207A-115	25	-	-	2345	-	-	-
XC050107-2	-	CS	050207A-116	50	-	-	4832	-	-	-
L11423-2	SG-A-002	S	050207A-117	-	100	10	43155	4690	5000	94
L11423-6	SG-A-006	S	050207A-118	-	100	10	43571	4740	5000	95
L11423-10	SG-A-010	S	050207A-119	-	100	10	42413	4610	5000	92
L11423-14	SG-A-014	S	050207A-120	-	100	10	38178	4150	5000	83
L11423-14 Rep	SG-A-014	S	050207A-121	-	100	10	37514	4070	5000	81
L11423-18	SG-A-018	S	050207A-122	-	100	10	41475	4510	5000	90
XC050107-3	-	CS	050207A-123	100	-	-	9908	-	-	-
XC050107-4	-	CS	050207A-124	250	-	-	24267	-	-	-
XC050107-5	-	CS	050207A-125	500	-	-	47373	-	-	
XC050107-6	-	CS	050207A-126	1000	-	-	91602	-	-	

13C-PFOA Found (ng/L) = (peak area - intercept) / slope x DF

Recovery (%) = $\frac{\text{13C-PFOA found (ng/L)}}{\text{amount 13C-PFOA added (ng/L)}} \times 100$

Standard Curve : Linear (1/x weighted)
Intercept = 259.396
Slope = 91.4599
Coef. Of Det. = 0.999260

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

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.

Spreadsheet prepared by: MLC / 5/3/07

Exygen RESEARCH
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State College, PA 16801
Phone: 814-272-1039
Fax: 814-231-1580

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423 Matrix: NaOH Impinger Sample
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 34
Time 8:25 AM Date 5/02/2007 Initials EDD

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-A-014	L11423-14* Spk C	100 mL	40.0	20000.0
SG-A-002	L11423-2*	100 mL	40.0	-
SG-A-006	L11423-6*	100 mL	40.0	-
SG-A-010	L11423-10*	100 mL	40.0	-
SG-A-014	L11423-14 Rep*	100 mL	40.0	-
SG-A-014	L11423-14*	100 mL	40.0	-
SG-A-018	L11423-18*	100 mL	(RE kg 05/24/07 / 40.0	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
L11423-14* Spk C	SS20794 (100000 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-

All samples were measured:
Time 10:25 AM Date 5/02/2007 Initials EDD

After measuring samples were returned to refrigerator No. 34
Time 3:30 PM Date 5/02/2007 Initials EDD

Comments: *Sample is pre-diluted ten times by taking 4 mL of sample and bringing the volume up to 40 mL with HPLC water

Analysis Summary:
Data Set: 050207A Initials/Date: MLC / 5/2/07
Data Set: 050407C Initials/Date: MLC / 5/4/07
Data Set: - Initials/Date: - / -

Set extraction/analysis data verified by: CEE Date: 05/24/07

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Fax: 814-231-1580

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007C[$$] PROTOCOL NUMBER: NA

MATRIX: NaOH Impinger Sample ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	Reagent Control			-		-	-	Methanol	46303
na	Reagent Spk A			-		-	-	C18 SPE	005387081B
na	Reagent Spk B			-		-	-	HPLC Water	46335
SG-A-014	L11423-14* Spk C			0.1/10, 0.1/1.0		-	-	-	-
SG-A-002	L11423-2*			-		-	-	-	-
SG-A-006	L11423-6*			-		-	-	-	-
SG-A-010	L11423-10*			-		0.1/1.0		-	-
SG-A-014	L11423-14 Rep*			-		0.1/1.0		-	-
SG-A-014	L11423-14*			-		0.1/1.0		-	-
SG-A-018	L11423-18*	↓	↓	-	↓	0.1/1.0	↓	*Initials/Date*	EDD 5/2/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C44404
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date	-	EDD 5/2/07	EDD 5/2/07	MLC 5/2/07	MLC 5/2/07	MLC 5/4/07	MLC 5/4/07	*Initials/Date*	MLC 5/2/07

①DF=1000 MLC 5/2/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

*Sample was diluted ten times prior to extraction.
**Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:

Final extracts stored in refrigerator 32 Initials: EDD Date: 5/2/2007

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Fax: 814-231-1580

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Exygen STUDY NO. L11423

Protocol No.: None
Method No.: $$

Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA

Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC050107-0	Methanol	46303
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC050107-1	C18 SPE	0053370818
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC050107-2	Water	46335
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC050107-3	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC050107-4	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC050107-5	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC050107-6	-	-
-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/01/07

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) freezer # 34 Time: 9:10 AM — Initials/Date: EDD 5/01/07

40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD 5/01/07

After measuring, samples returned to (refrigerator) freezer # 34 Time: 11:10 AM — Initials/Date: EDD 5/01/07

Samples fortified: — Initials/Date: AMB 05/01/07

SPE clean-up: — Initials/Date: EDD 5/01/07

Final volume adjusted to 5 mL: — Initials/Date: EDD 5/01/07

Extracts placed in refrigerator # 32 — Initials/Date: EDD 5/01/07

Comments:

STANDARD EXPIRATION DATE: 5/15/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/2/07

Masslynx - Sample List

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050207A NaOH.SPL
Printed: Wed May 02 15:52:22 2007 MLC 5/2/07

Exygen STUDY NO. L11423

Page Position: (1, 1)

L (RE) MLC 5/2/07

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/~~mL~~)	Conc B	Conc C	Test ID	DF
1	71	050207A-101	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---	0	1
2	72	050207A-102	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
3	73	050207A-103	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
4	74	050207A-104	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
5	75	050207A-105	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
6	76	050207A-106	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
7	77	050207A-107	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1
8	92	050207A-108	---	---	Methanol Wash	---	Blank	---	---	---	0	1
9	21	050207A-109	---	---	Reagent Control	---	Blank	---	---	---	0	1
10	22	050207A-110	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---	0	1
11	23	050207A-111	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500	---	0	1
12	24	050207A-112	---	---	L11423-14 Spk C, 5,000,000 ng/L, DF=10,000	---	QC	5000000	5000	---	0	10000
13	25	050207A-113	---	---	L11423-14 Spk C, 5,000,000 ng/L, DF=10	---	QC	5000000	5000	---	0	10
14	92	050207A-114	---	---	Methanol Wash	---	Blank	---	---	---	0	1
15	72	050207A-115	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
16	73	050207A-116	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
17	26	050207A-117	---	---	L11423-2, DF=10	---	Analyte	---	5000	---	0	10
18	27	050207A-118	---	---	L11423-6, DF=10	---	Analyte	---	5000	---	0	10
19	28	050207A-119	---	---	L11423-10, DF=10	---	Analyte	---	5000	---	0	10
20	29	050207A-120	---	---	L11423-14, DF=10	---	Analyte	---	5000	---	0	10
21	30	050207A-121	---	---	L11423-14 Rep, DF=10	---	Analyte	---	5000	---	0	10
22	31	050207A-122	---	---	L11423-18, DF=10	---	Analyte	---	5000	---	0	10
23	74	050207A-123	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
24	75	050207A-124	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
25	76	050207A-125	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
26	77	050207A-126	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1

Exygen STUDY NO. 411423

	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
20	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
21	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
22	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
23	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
24	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
25	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
26	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA | Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/2/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/3/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Wed May 02 15:52:35 2007 MLC 5/2/07

Solvent Delay (mins) : 0.00

Function : 1 MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Wednesday, May 02, 2007 15:50:12

Printed: Wednesday, May 02, 2007 15:52:47 MLC 5/2/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

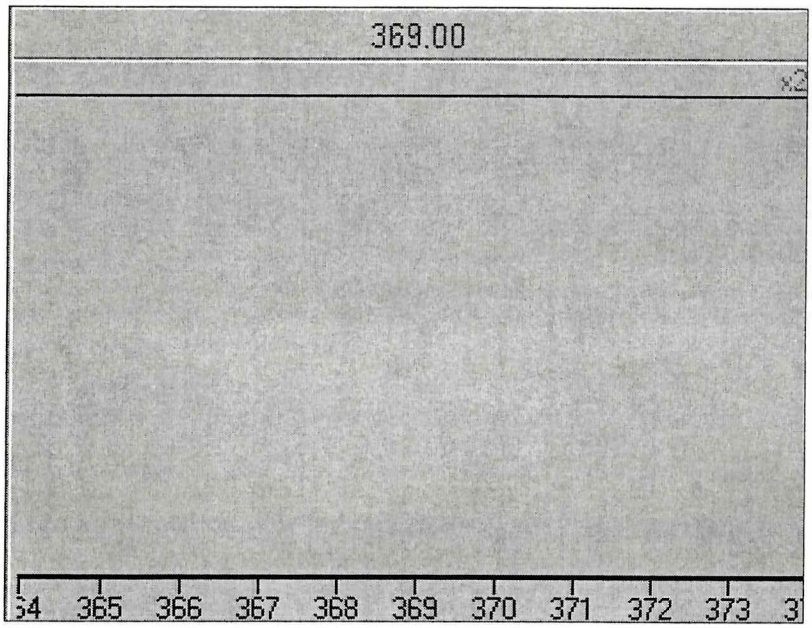
HP1100 Autosampler Initial Conditions

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	92

Tuning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Wed May 02 16:02:21 2007 MLC 5/2/07



Dau 413.00

SOURCE (ESP-)	Set	Rdbk
Capillary	3.00	-2.94
Cone	10	-10
Hexapole 1	0.0	
Aperture 1	0.0	
Hexapole 2	0.0	
Source Block Temp.	100	102
Desolvation Temp.	300	300

Analyser	Set	Rdbk
LM Res 1	14.0	
HM Res 1	14.0	
IEnergy 1	1.0	
Entrance	-2	8
Collision	10	9
Exit	2	11
LM Res 2	14.0	
HM Res 2	14.0	
IEnergy 2	2.0	
Multiplier	700	-695

Pressures	Rdbk
Analyser Vacuum	OFF
Gas Cell	2.2e-3

Gas Flows	Rdbk
Cone Gas	89.7
Desolvation	682.4

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050207A
Extraction Date: 05/02/07
Analyzed on: 05/02-03/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2834
XC050107-2	50	5167
XC050107-3	100	9481
XC050107-4	250	24504
XC050107-5	500	45324
XC050107-6	1000	93511

Coefficient of Determination: 0.999485536

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999742735
R Square	0.999485536
Adjusted R Square	0.99935692
Standard Error	883.8822453
Observations	6

MLC
5/3/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	6071139588	6071139588	7771.080321	9.92695E-08
Residual	4	3124991.294	781247.8235		
Total	5	6074264579			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	427.5911432	493.7488641	0.866009371	0.435338768	-943.2754737	1798.45776	-943.2754737	1798.45776
X Variable 1	92.6002354	1.05044034	88.15373118	9.92695E-08	89.68374546	95.51672534	89.68374546	95.51672534

Initial Calibration Curve for ^{13}C-PFOA

Exygen Study No.: L11423
Set No: 050207A
Extraction Date: 05/02/07
Analyzed on: 05/02-03/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2547
XC050107-2	50	4921
XC050107-3	100	9089
XC050107-4	250	23083
XC050107-5	500	45758
XC050107-6	1000	89508

Coefficient of Determination: 0.999881045

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999940521
R Square	0.999881045
Adjusted R Square	0.999851307
Standard Error	410.0128036
Observations	6

MLC
5/3/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	5652257640	5652257640	33622.27624	5.30653E-09
Residual	4	672441.9965	168110.4991		
Total	5	5652930082			

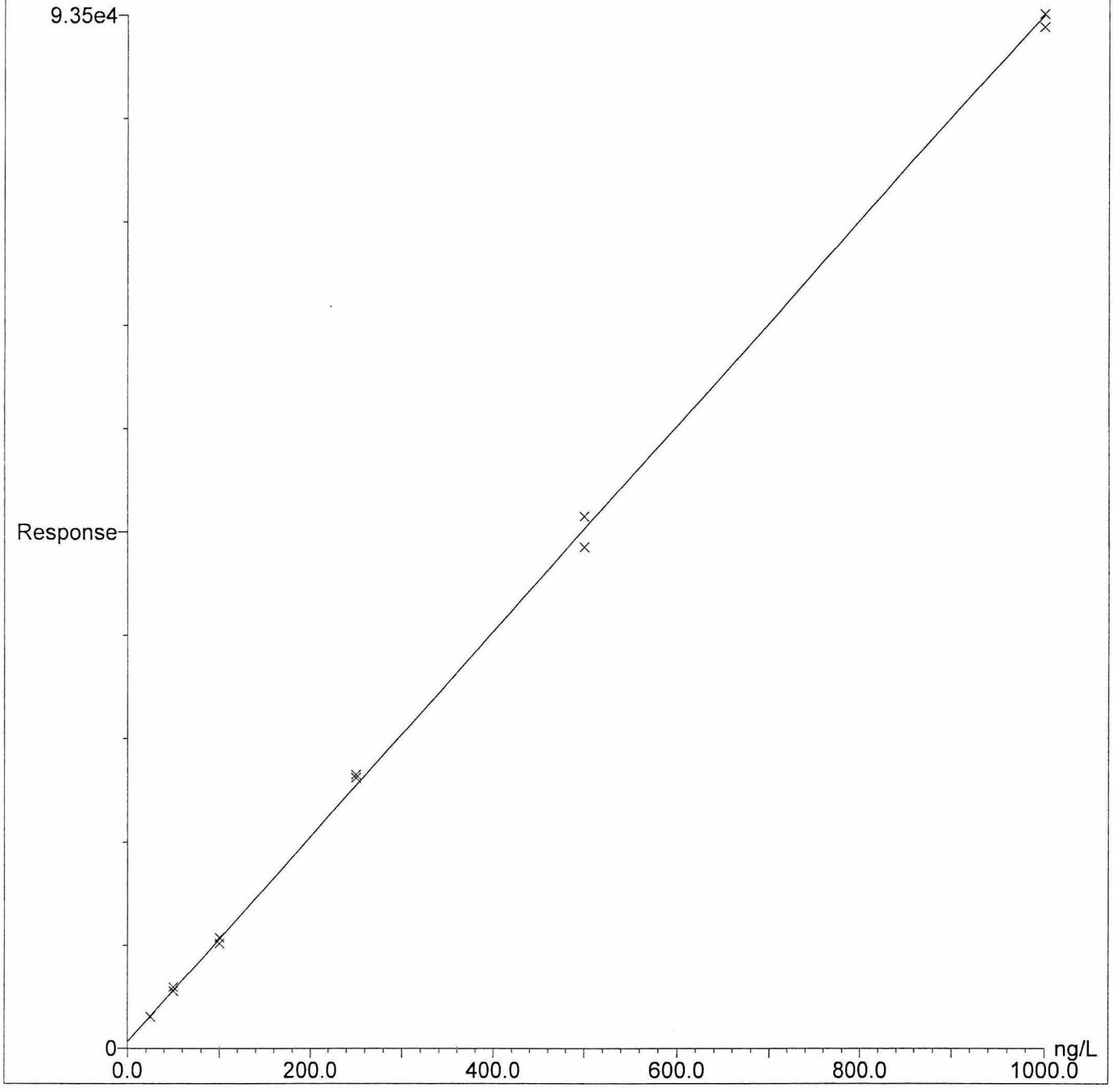
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	484.9773429	229.0388308	2.117445942	0.101634012	-150.9363978	1120.891084	-150.9363978	1120.891084
X Variable 1	89.34864205	0.487275303	183.3637812	5.30653E-09	87.99574892	90.70153518	87.99574892	90.70153518

Quantify Calibration Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050207A NaOH Impinger
Last modified: Thu May 03 11:08:33 2007
Printed: Thu May 03 11:09:14 2007

MLC 5/3/07

Compound 1 name: PFOA
Coefficient of Determination: 0.999427
Calibration curve: 92.7662 * x + 607.210
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

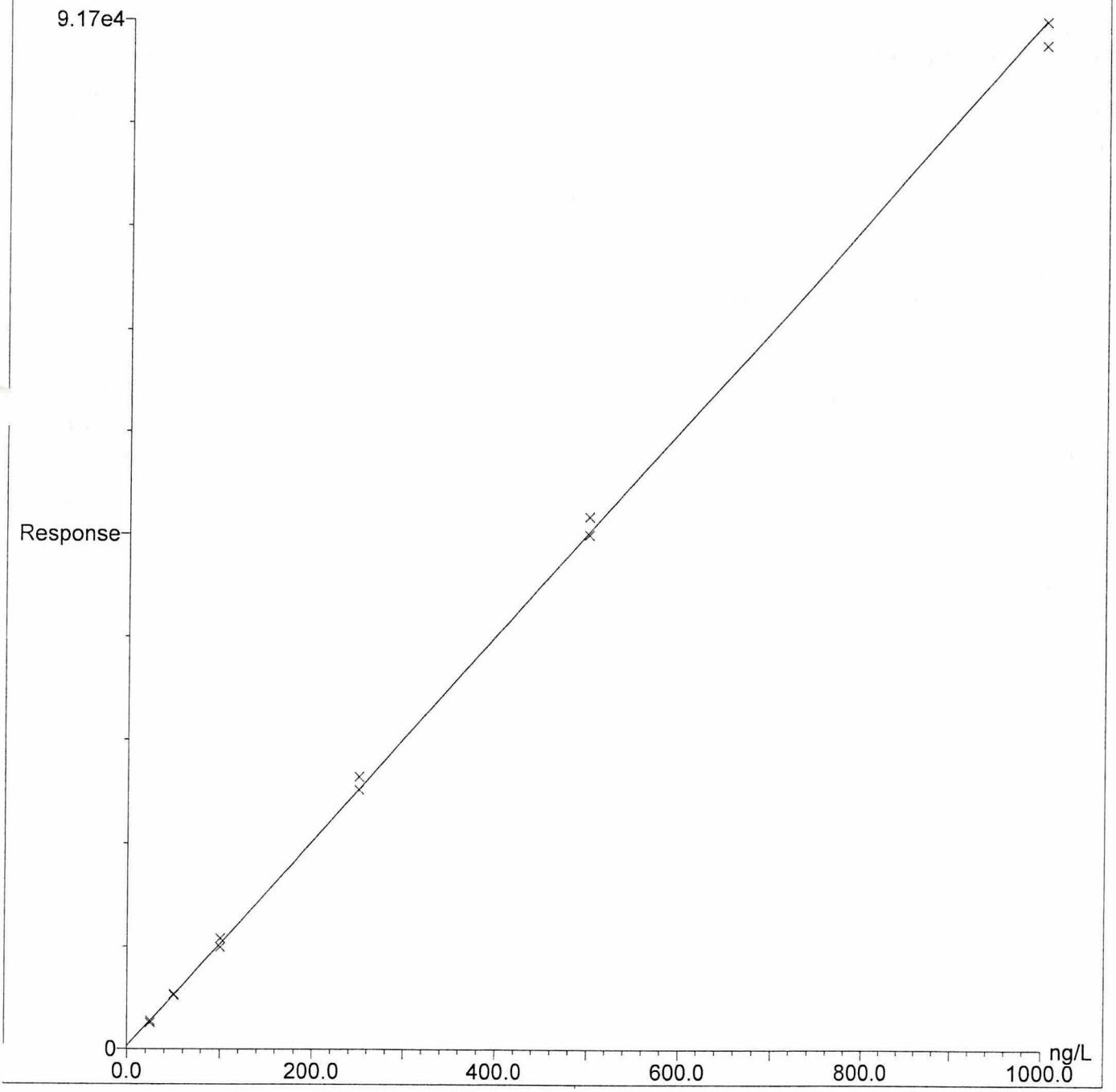


Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050207A NaOH Impinger
Last modified: Thu May 03 11:08:33 2007
Printed: Thu May 03 11:09:14 2007

MLC 5/3/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.999260
Calibration curve: 91.4599 * x + 259.396
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

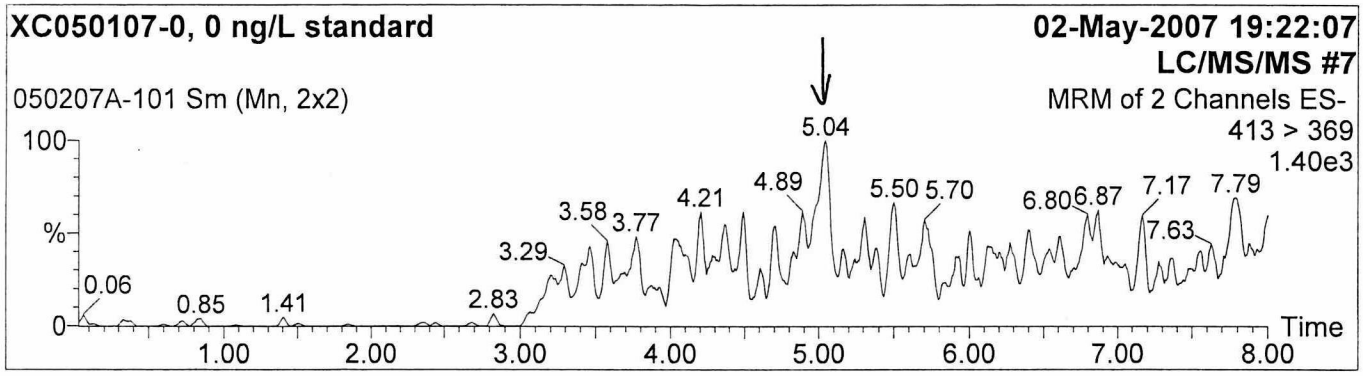
Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

Printed: Thu May 03 11:09:15 2007

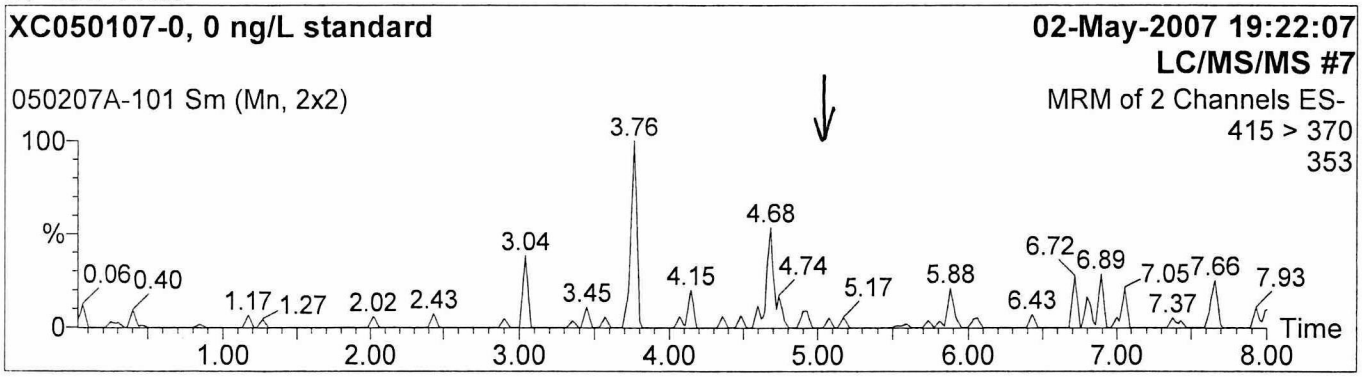
Initials MLC
Date 5/3/07
Run# 050207A-101 To 050207A-126

Name: 050207A-101
Text:

1: PFOA



2: 13C-PFOA



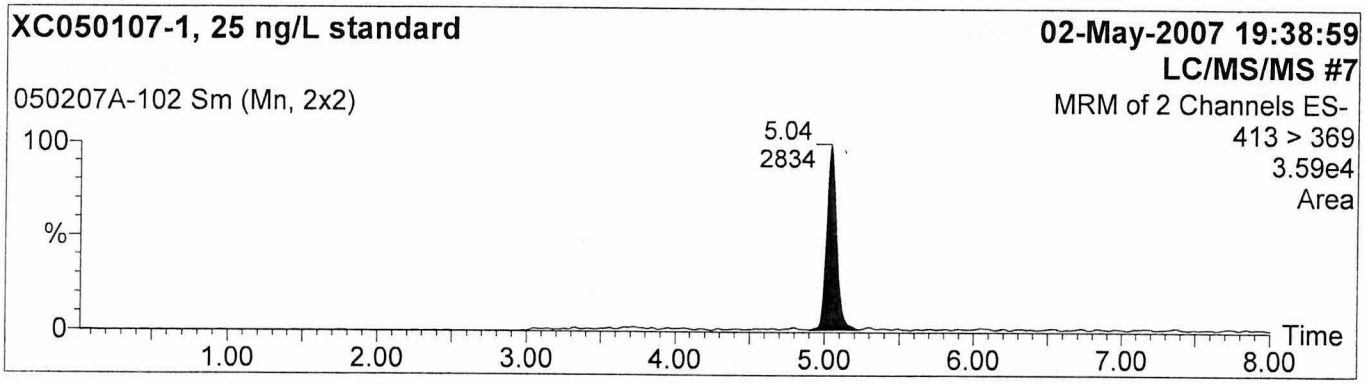
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

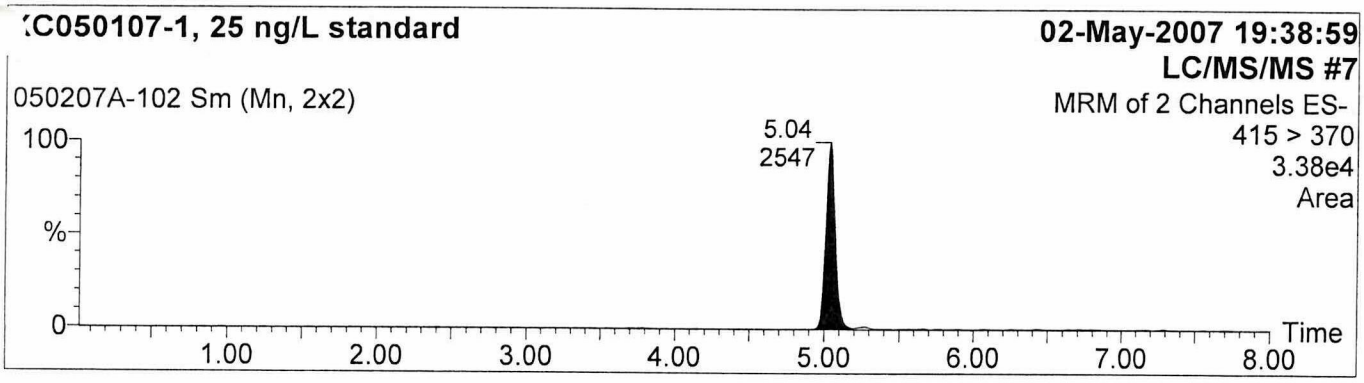
Printed: Thu May 03 11:09:15 2007

Name: 050207A-102
Text:

1: PFOA



2: 13C-PFOA



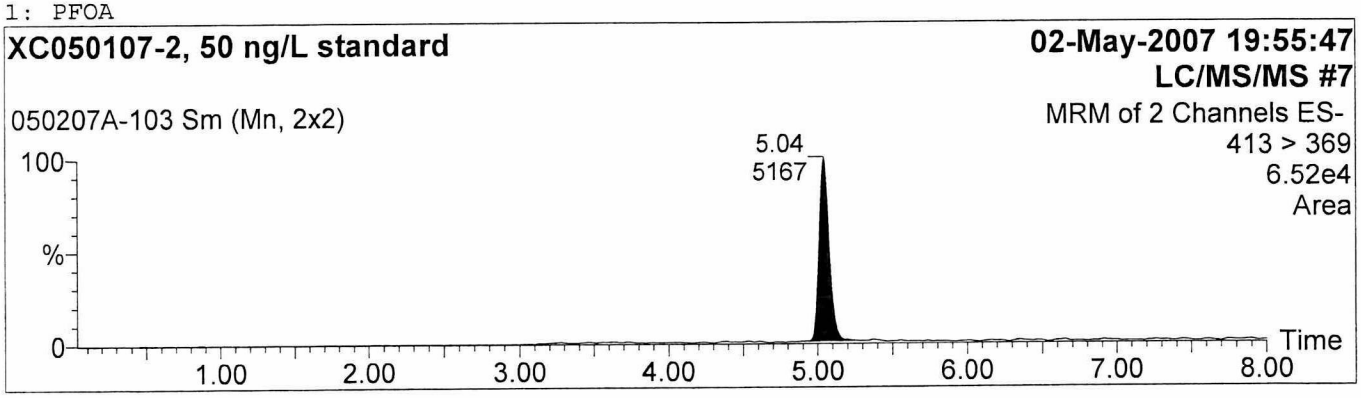
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

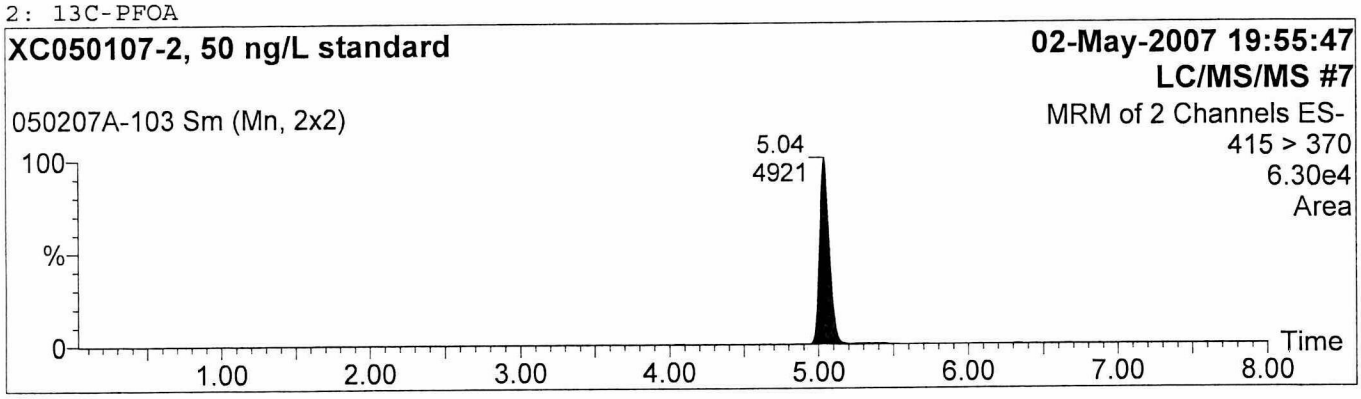
Printed: Thu May 03 11:09:15 2007

Name: 050207A-103
Text:

1: PFOA



2: 13C-PFOA



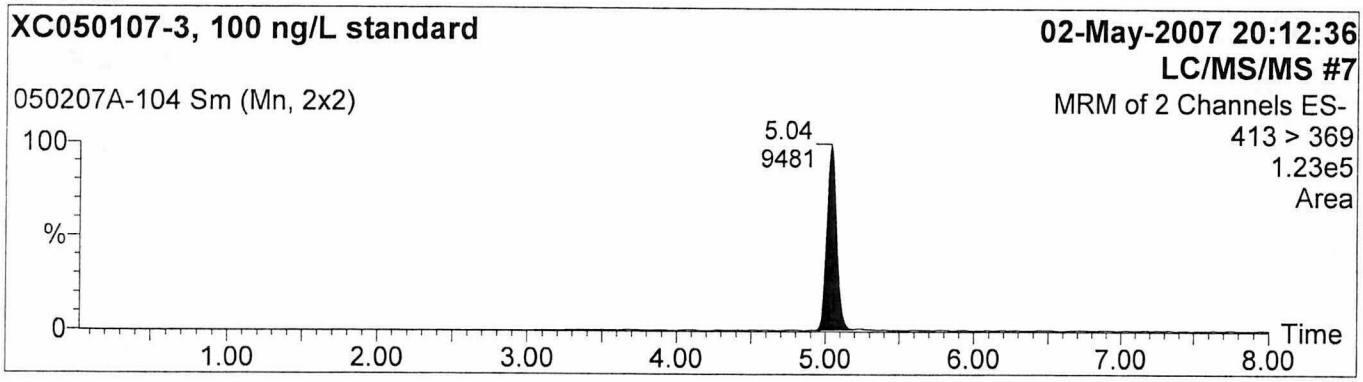
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

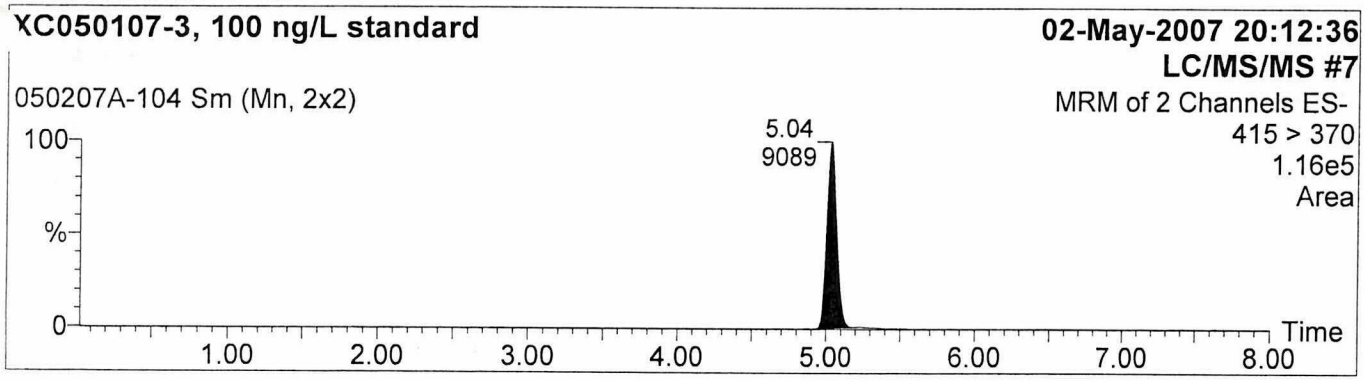
Printed: Thu May 03 11:09:15 2007

Name: 050207A-104
Text:

1: PFOA



2: 13C-PFOA



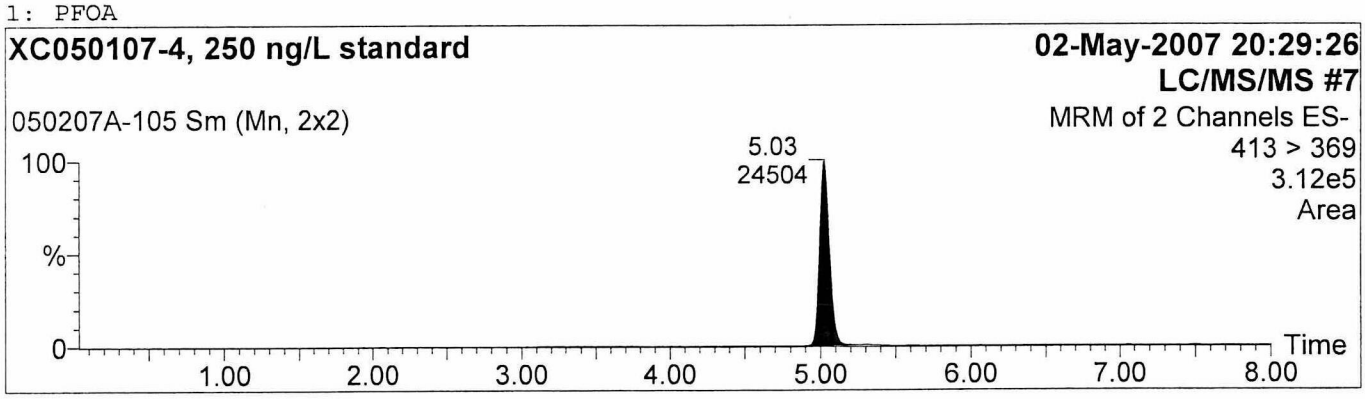
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

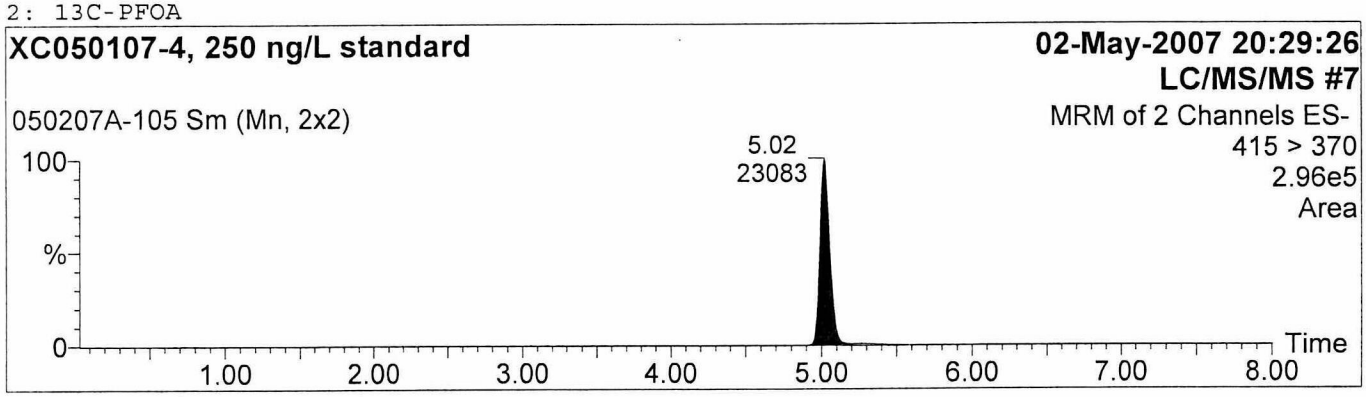
Printed: Thu May 03 11:09:15 2007

Name: 050207A-105
Text:

1: PFOA



2: 13C-PFOA



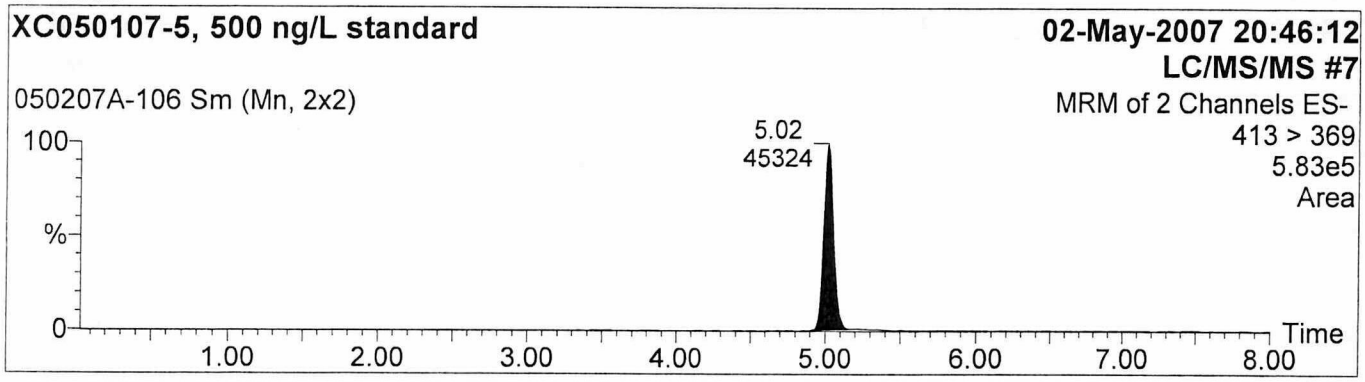
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

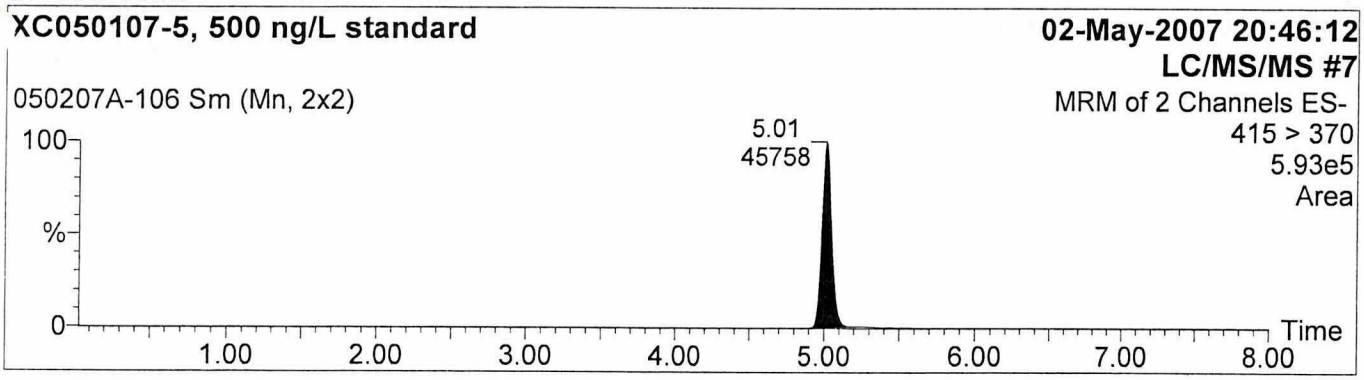
Printed: Thu May 03 11:09:15 2007

Name: 050207A-106
Text:

1: PFOA



2: 13C-PFOA



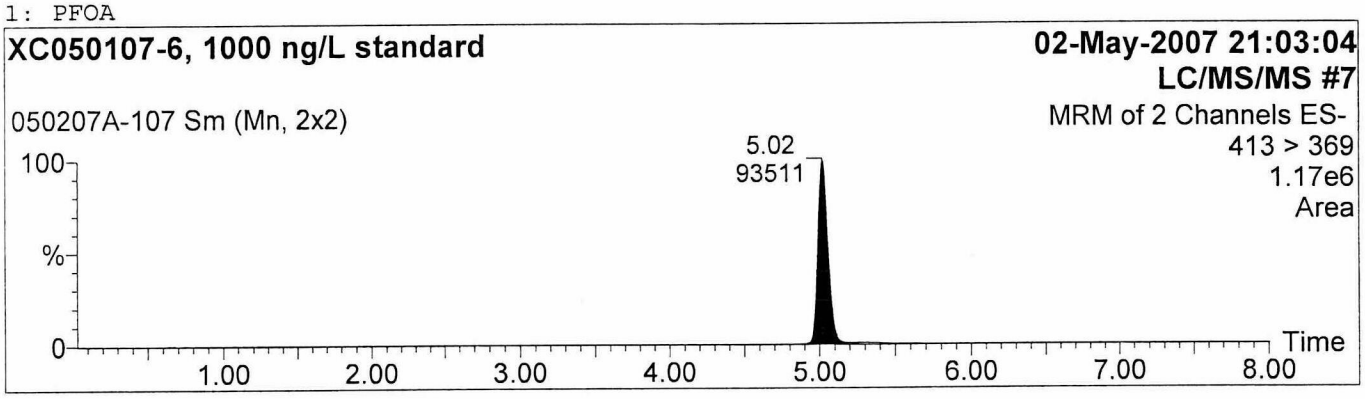
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

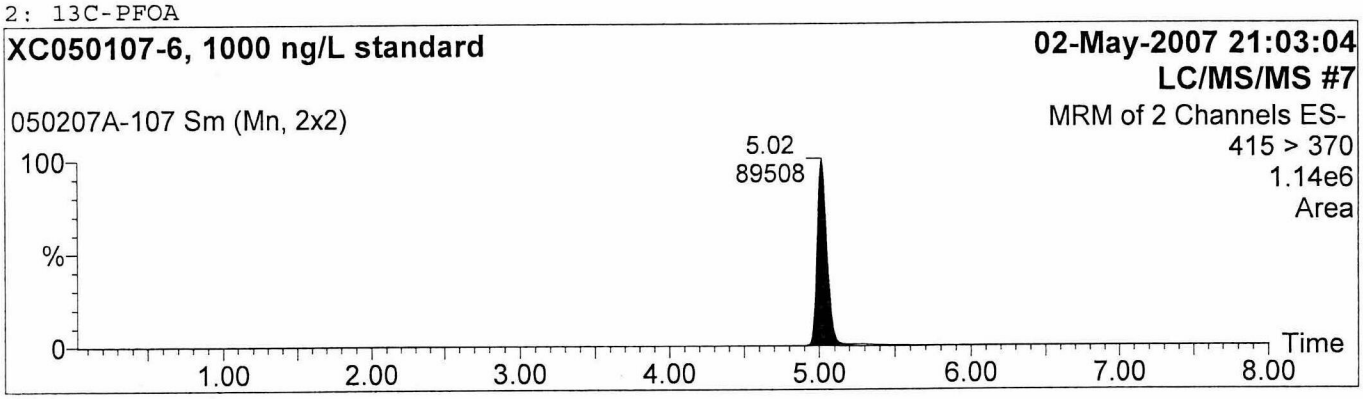
Printed: Thu May 03 11:09:15 2007

Name: 050207A-107
Text:

1: PFOA



2: 13C-PFOA



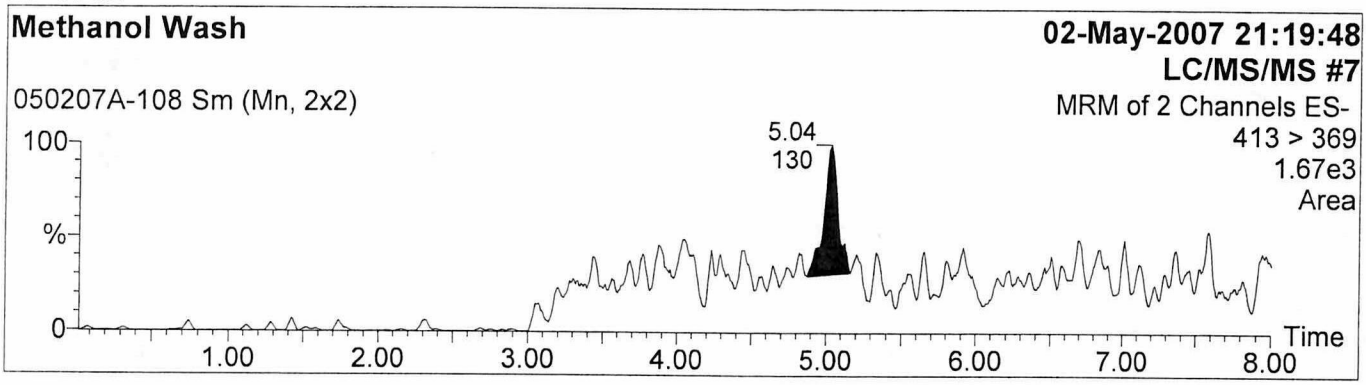
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

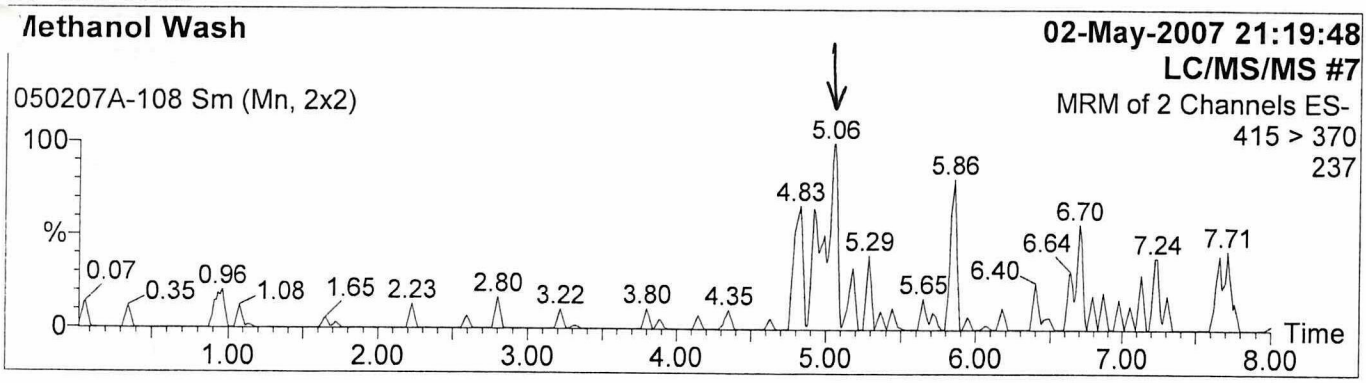
Printed: Thu May 03 11:09:15 2007

Name: 050207A-108
Text:

1: PFOA



2: 13C-PFOA



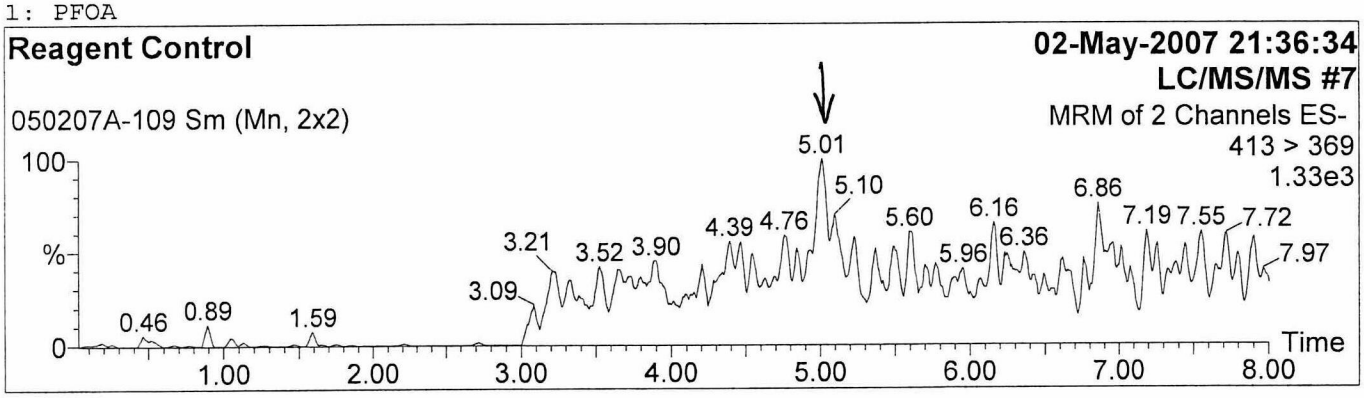
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

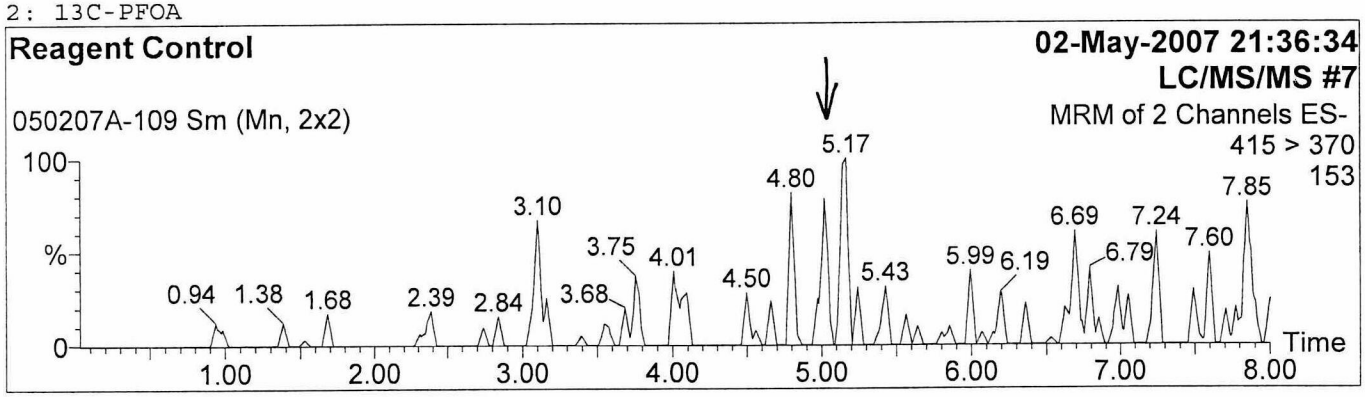
Printed: Thu May 03 11:09:15 2007

Name: 050207A-109
Text:

1: PFOA



2: 13C-PFOA



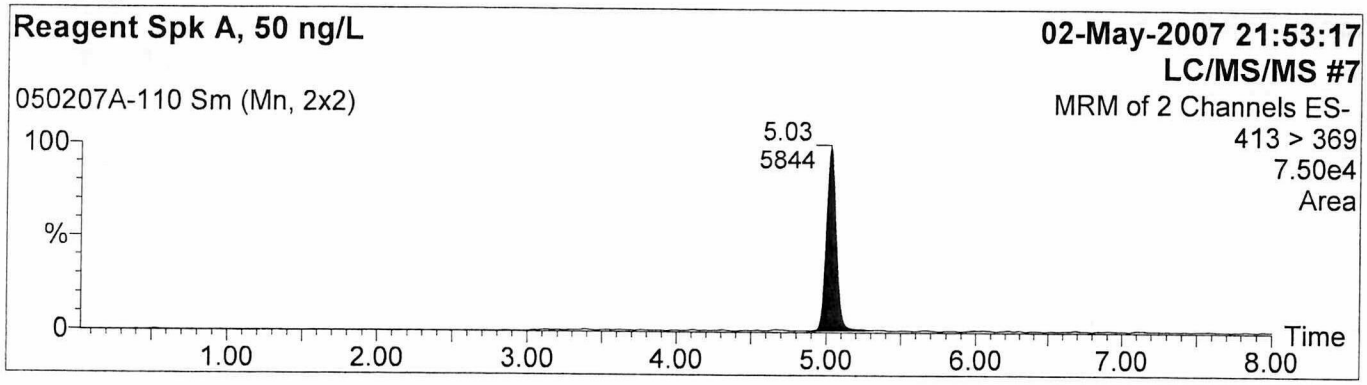
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

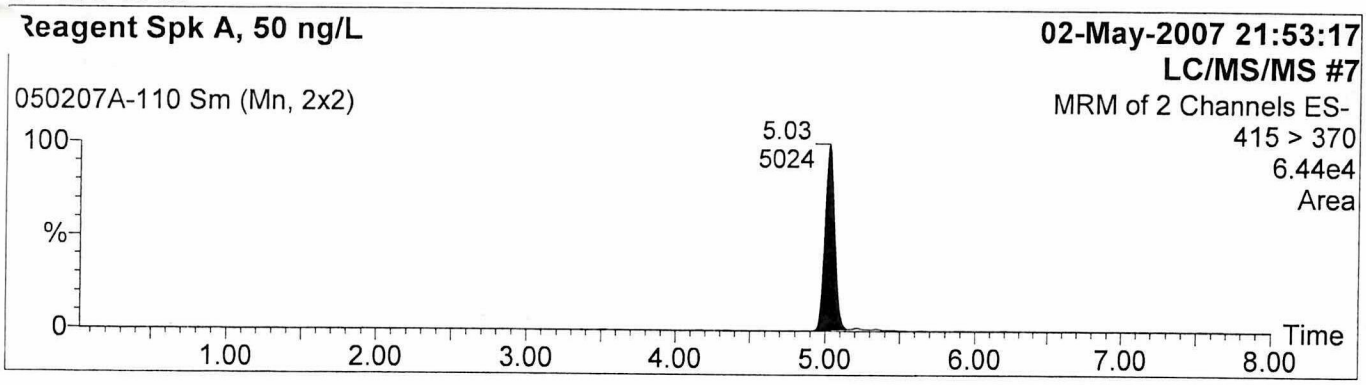
Printed: Thu May 03 11:09:15 2007

Name: 050207A-110
Text:

1: PFOA



2: 13C-PFOA



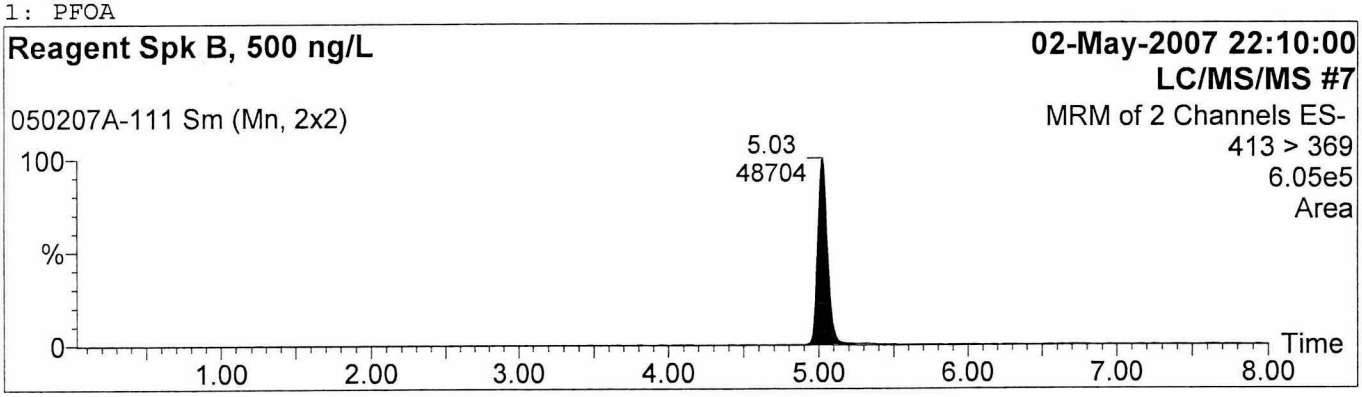
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

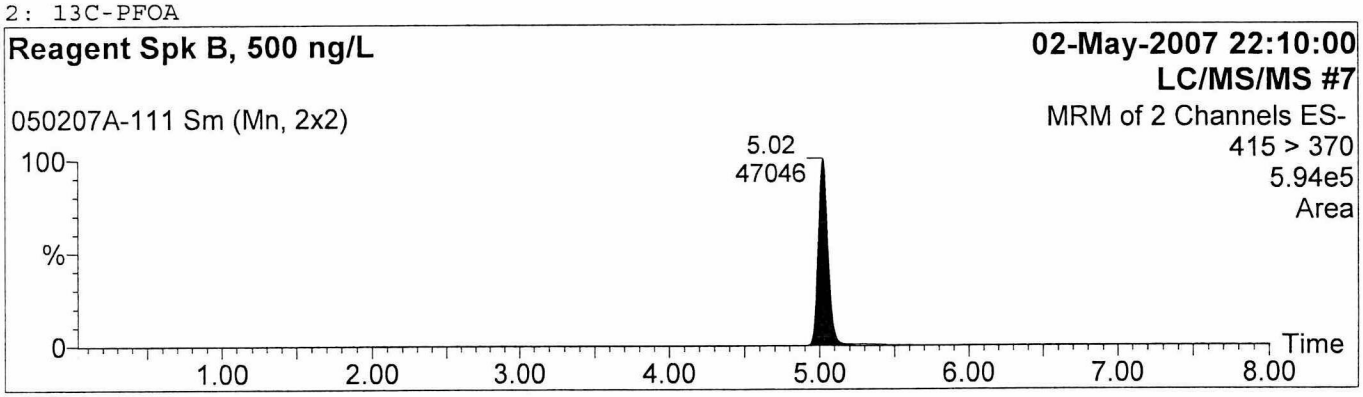
Printed: Thu May 03 11:09:15 2007

Name: 050207A-111
Text:

1: PFOA



2: 13C-PFOA



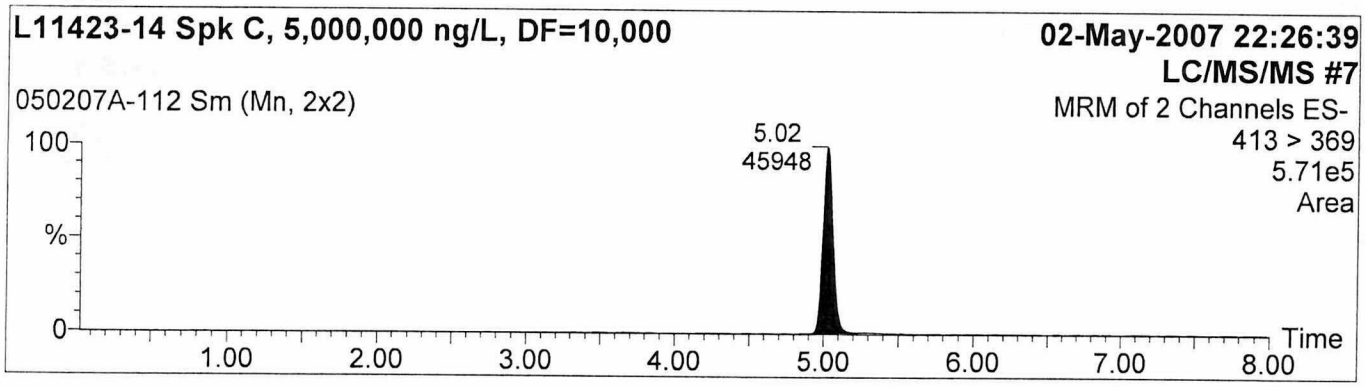
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

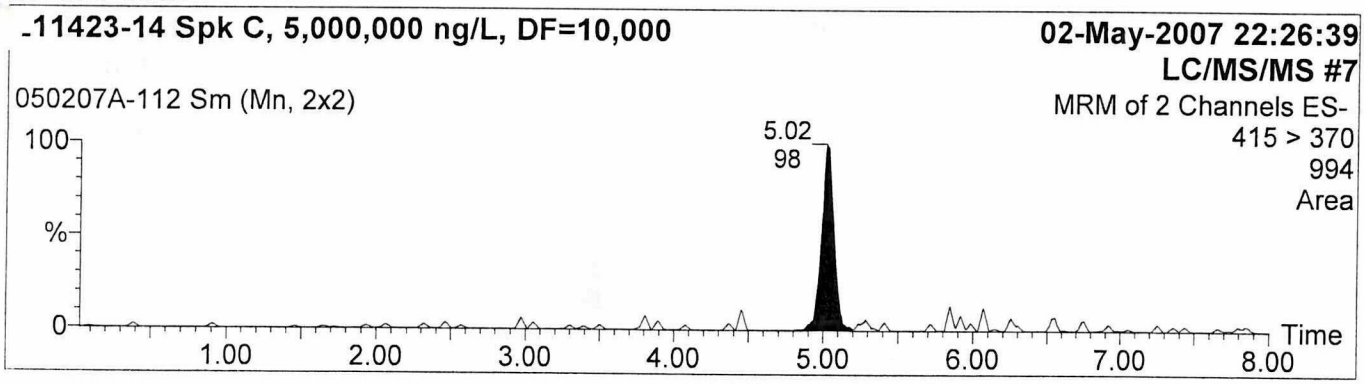
Printed: Thu May 03 11:09:15 2007

Name: 050207A-112
Text:

1: PFOA



2: 13C-PFOA



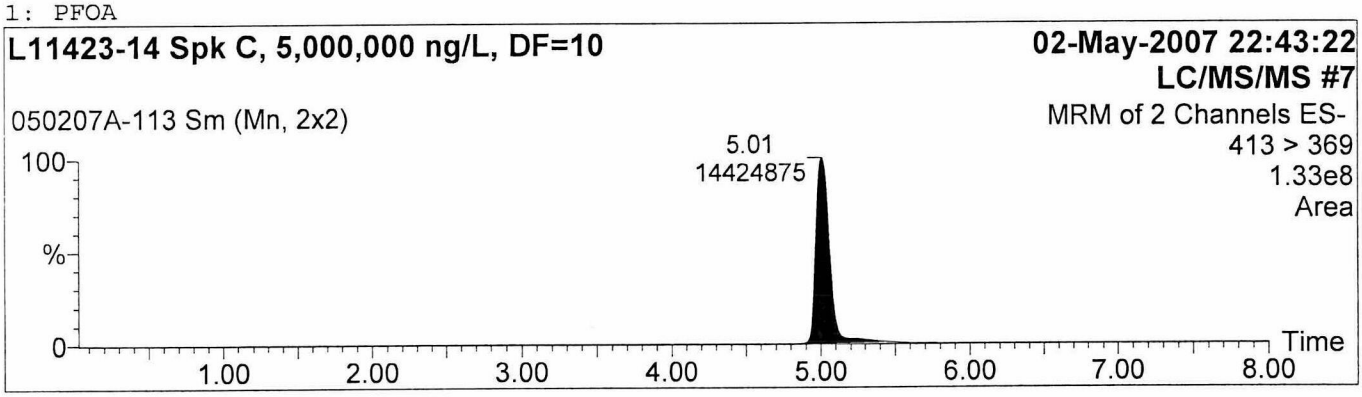
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

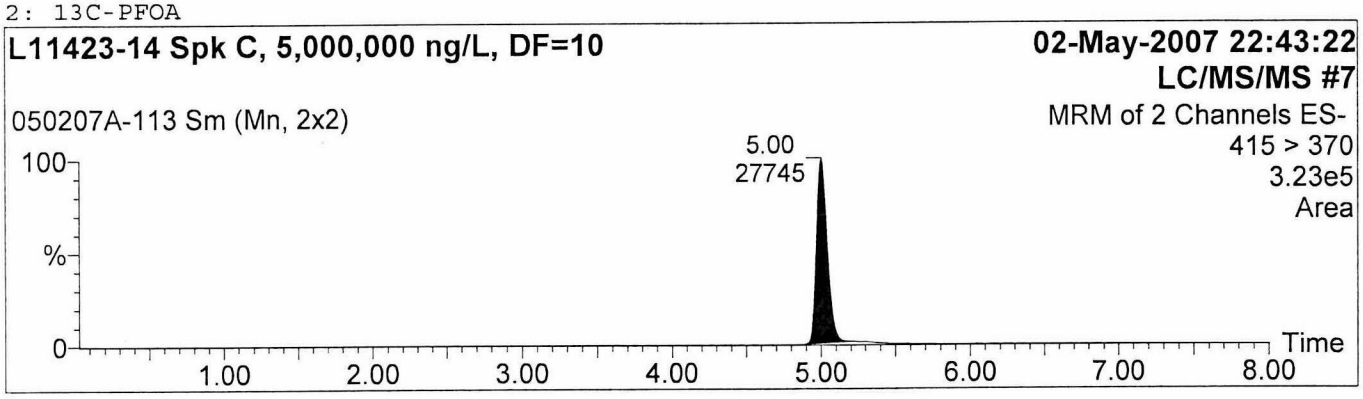
Printed: Thu May 03 11:09:15 2007

Name: 050207A-113
Text:

1: PFOA



2: 13C-PFOA



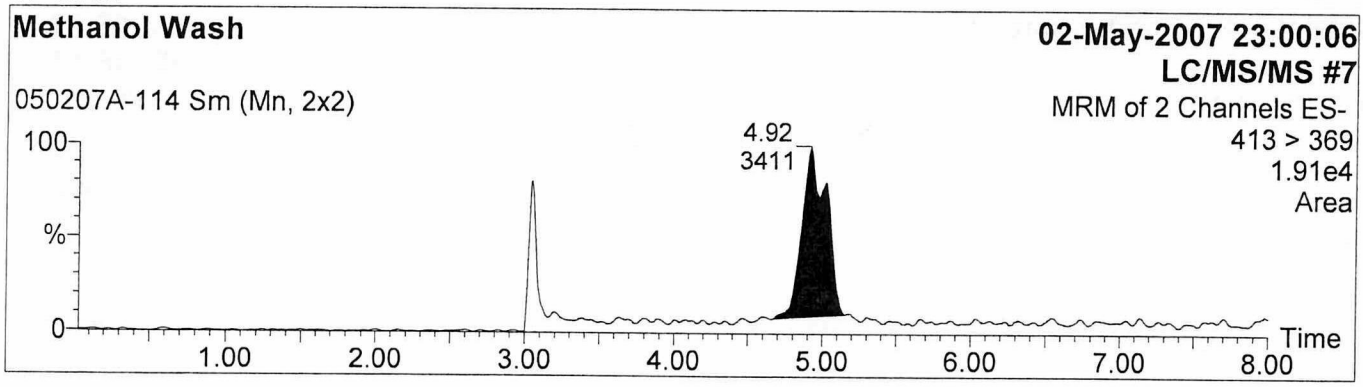
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

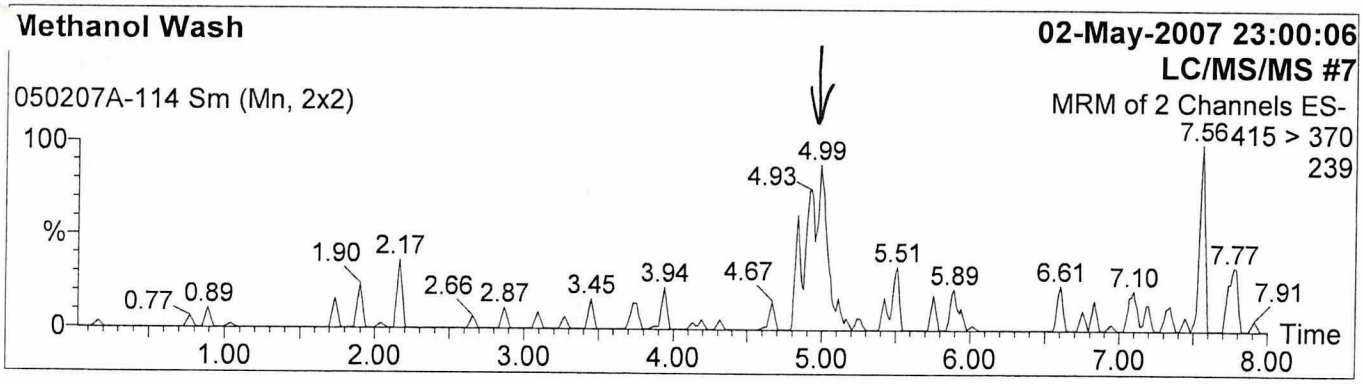
Printed: Thu May 03 11:09:15 2007

Name: 050207A-114
Text:

1: PFOA



2: 13C-PFOA

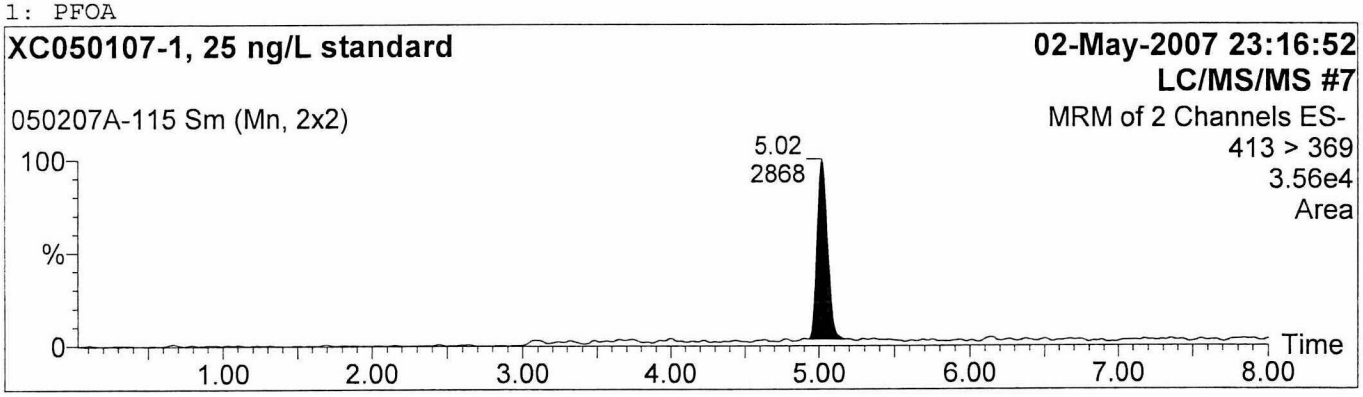


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

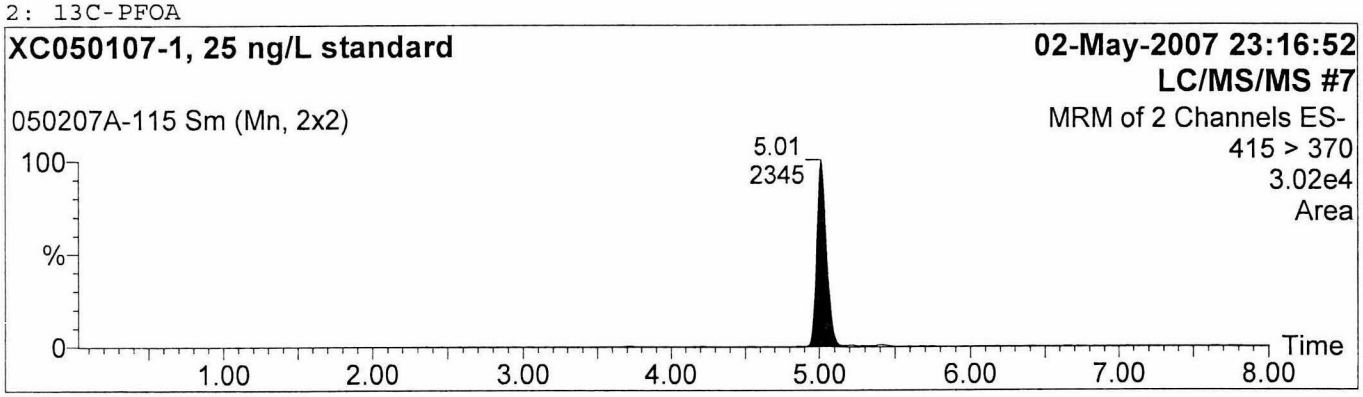
Printed: Thu May 03 11:09:15 2007

Name: 050207A-115
Text:

1: PFOA



2: 13C-PFOA



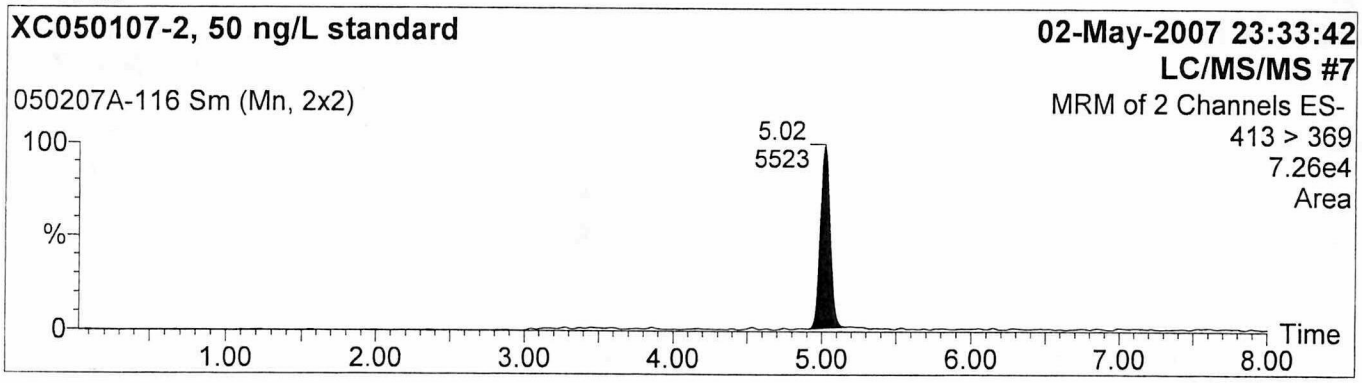
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

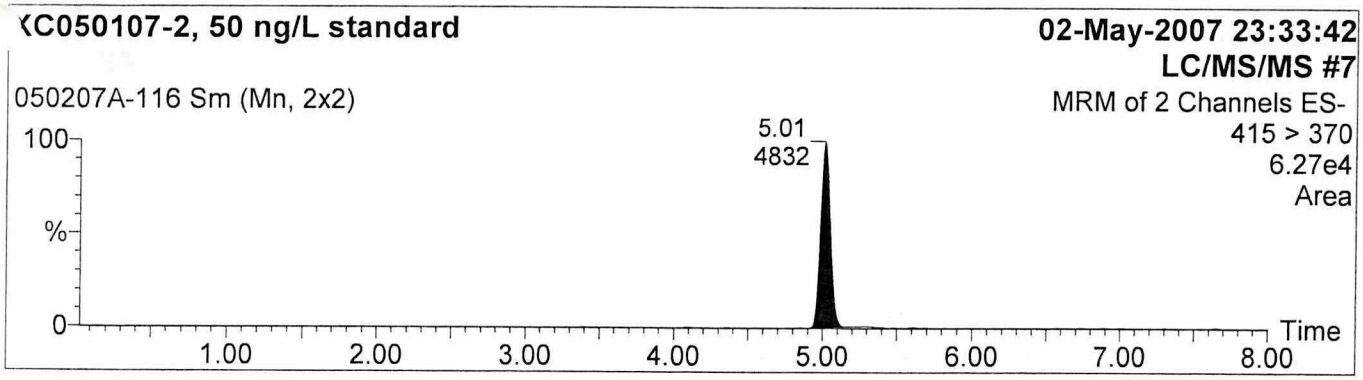
Printed: Thu May 03 11:09:15 2007

Name: 050207A-116
Text:

1: PFOA



2: 13C-PFOA



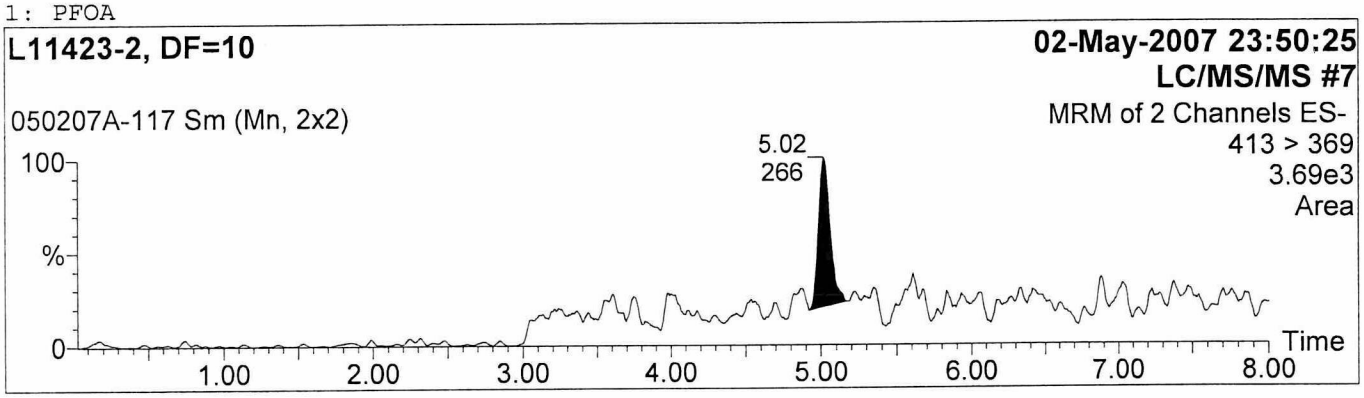
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

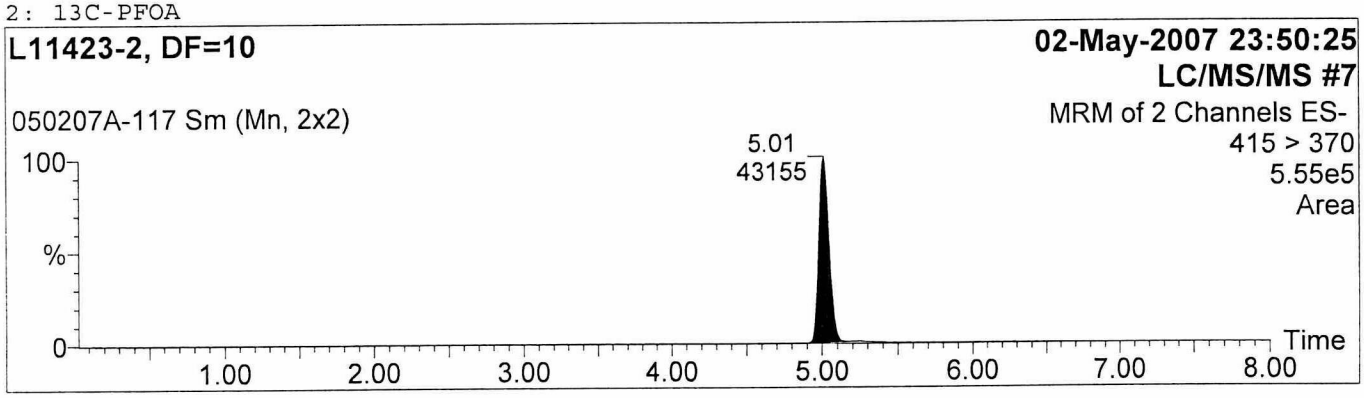
Printed: Thu May 03 11:09:15 2007

Name: 050207A-117
Text:

1: PFOA



2: 13C-PFOA



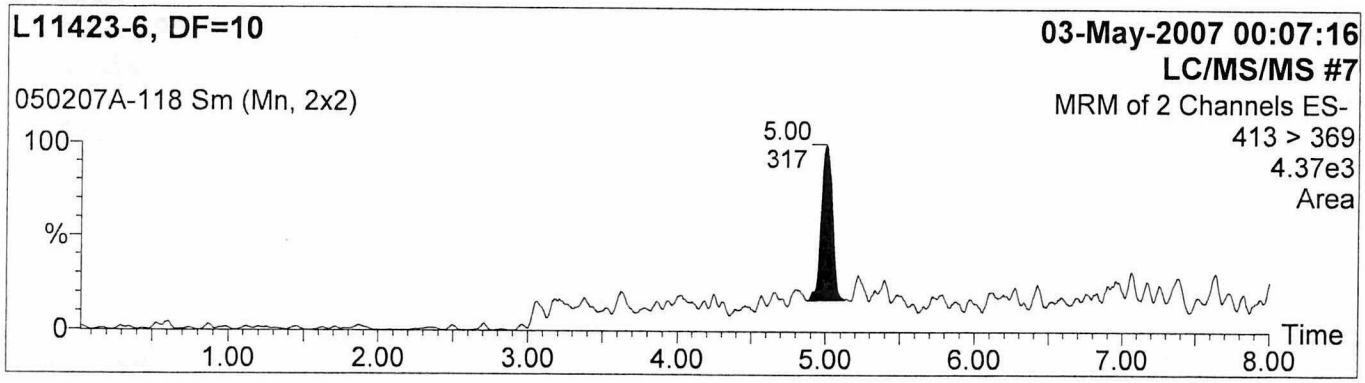
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

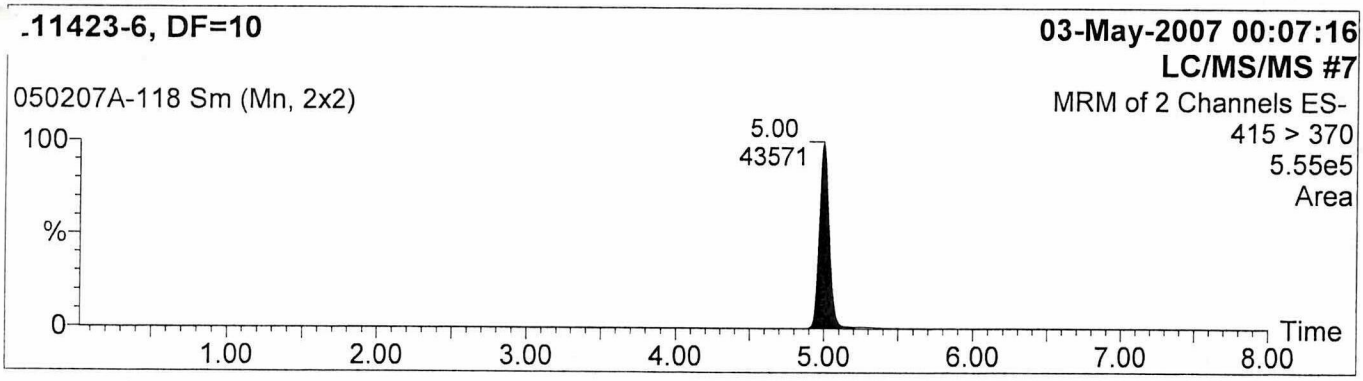
Printed: Thu May 03 11:09:15 2007

Name: 050207A-118
Text:

1: PFOA



2: 13C-PFOA



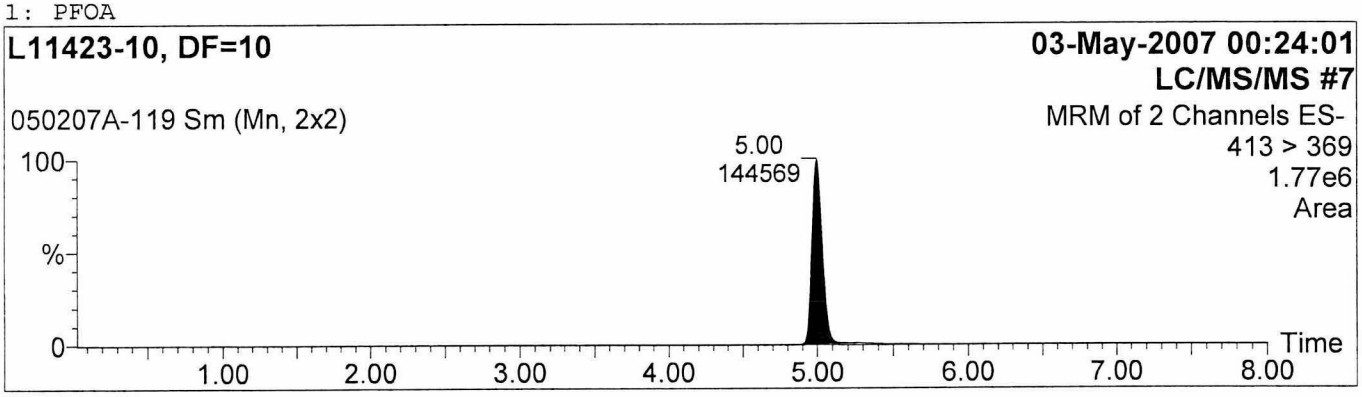
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

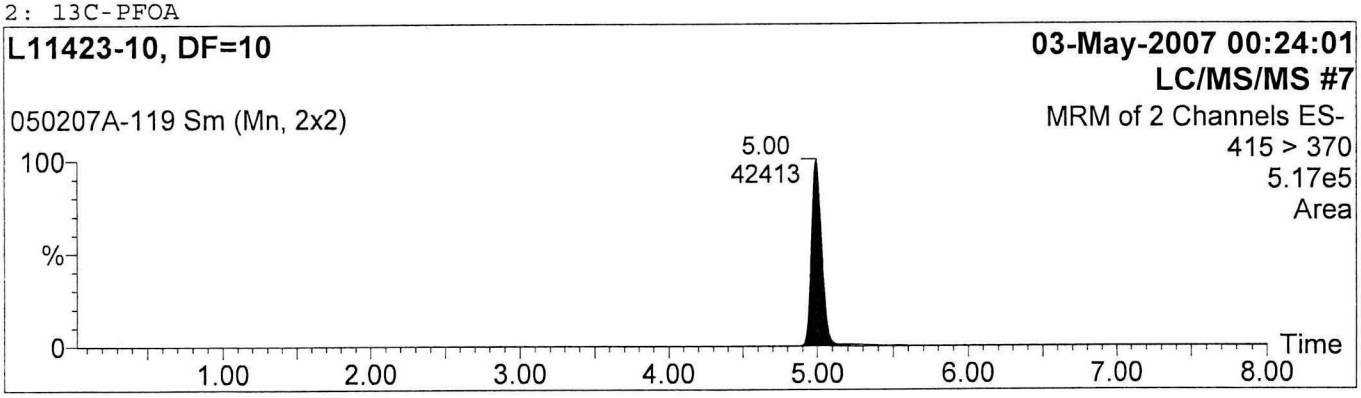
Printed: Thu May 03 11:09:15 2007

Name: 050207A-119
Text:

1: PFOA



2: 13C-PFOA



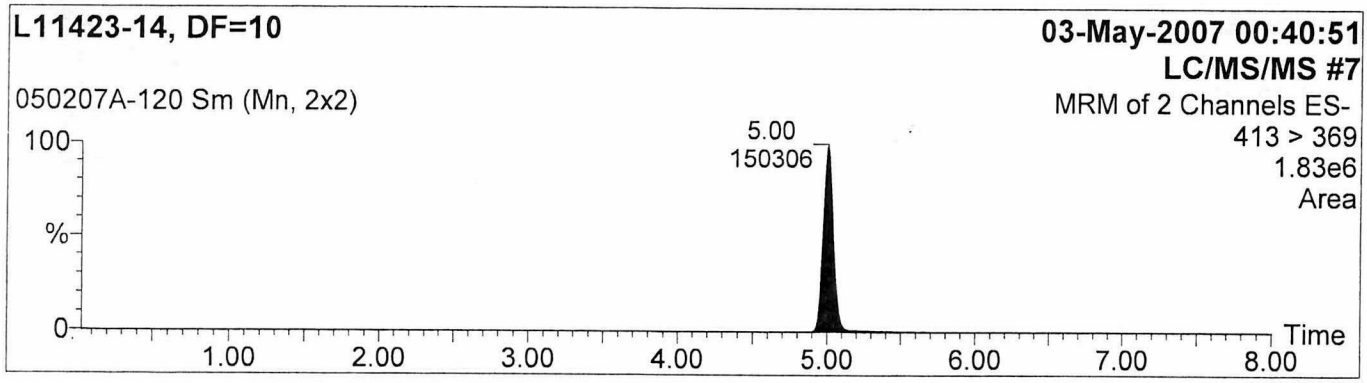
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

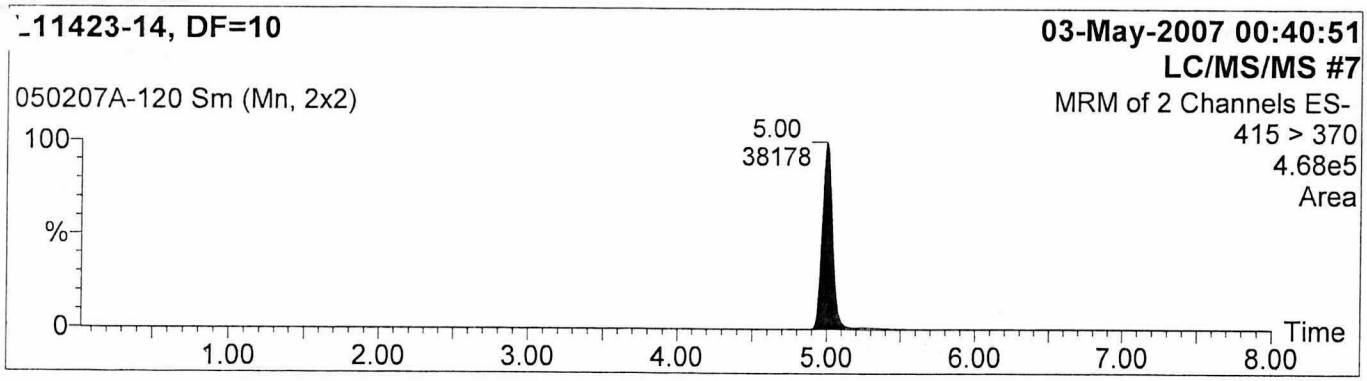
Printed: Thu May 03 11:09:15 2007

Name: 050207A-120
Text:

1: PFOA



2: 13C-PFOA



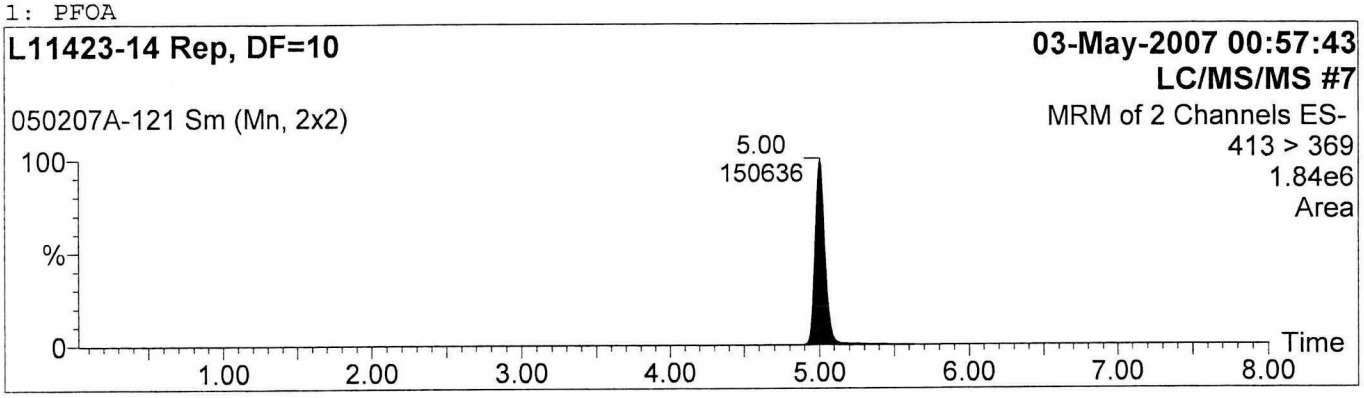
Quantify Sample Report
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

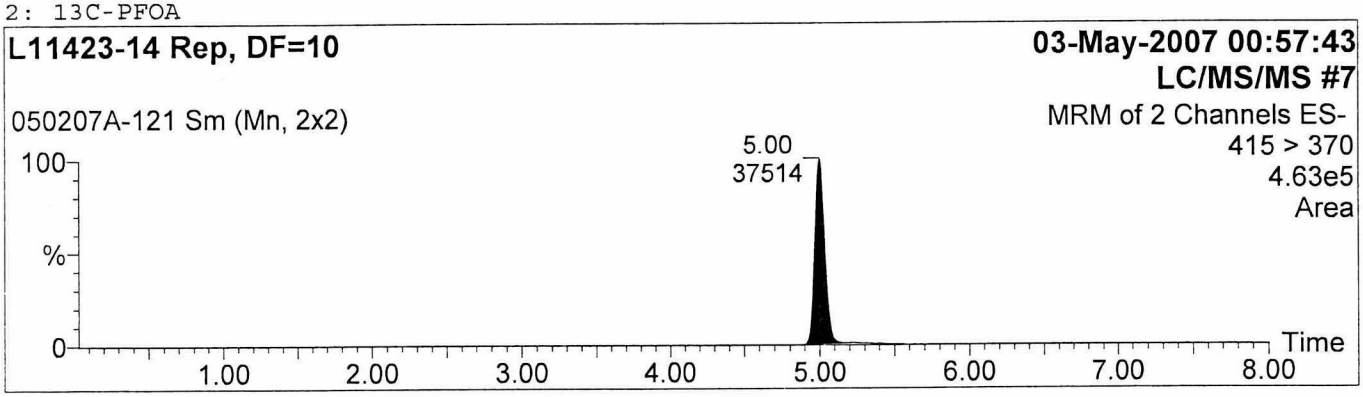
Printed: Thu May 03 11:09:15 2007

Name: 050207A-121
Text:

1: PFOA



2: 13C-PFOA



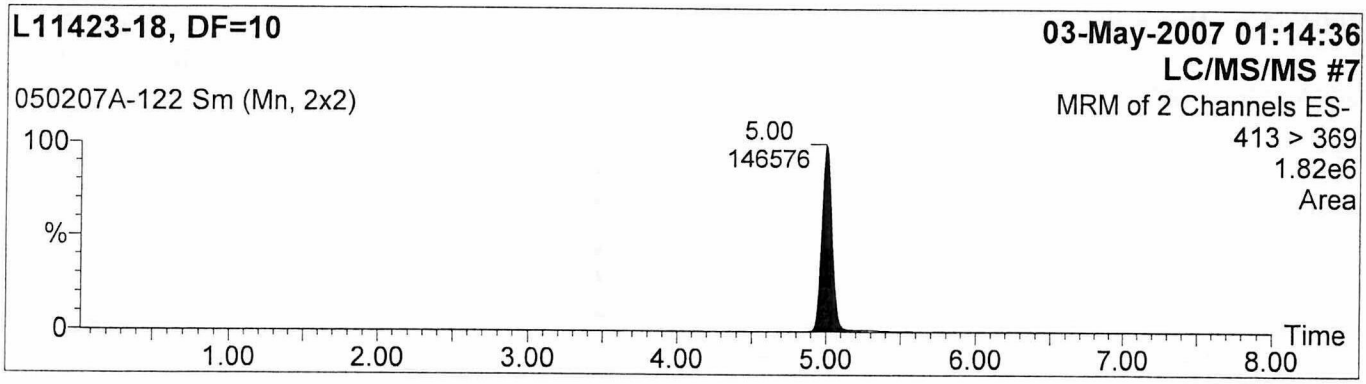
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

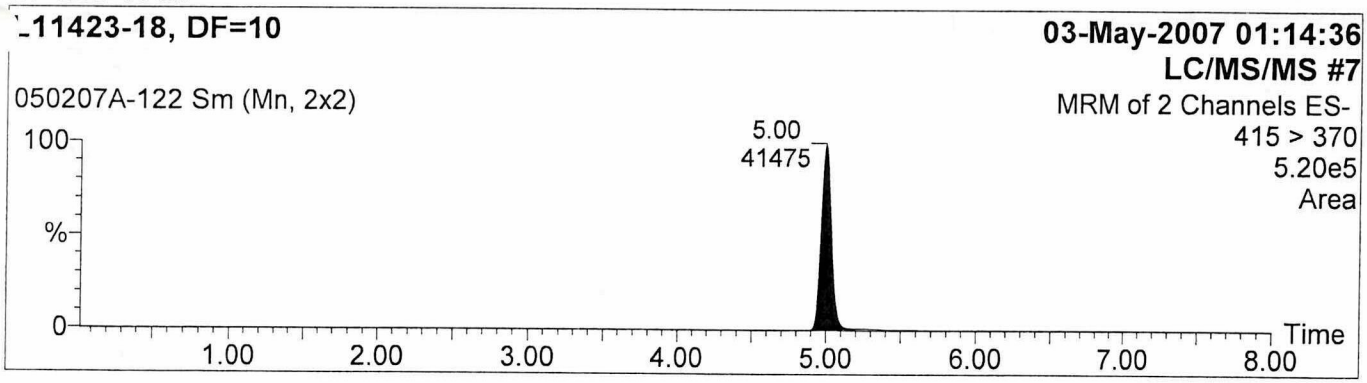
Printed: Thu May 03 11:09:15 2007

Name: 050207A-122
Text:

1: PFOA



2: 13C-PFOA

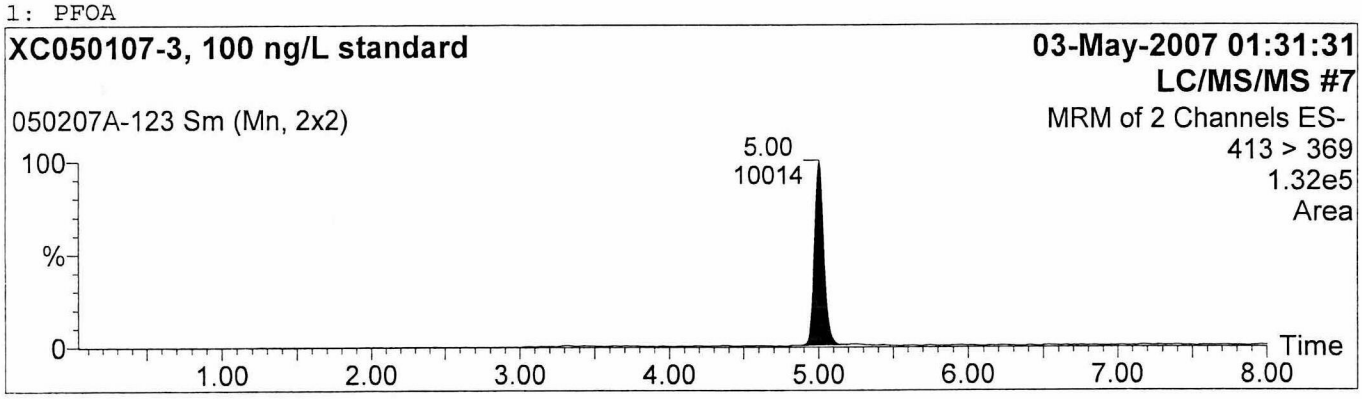


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

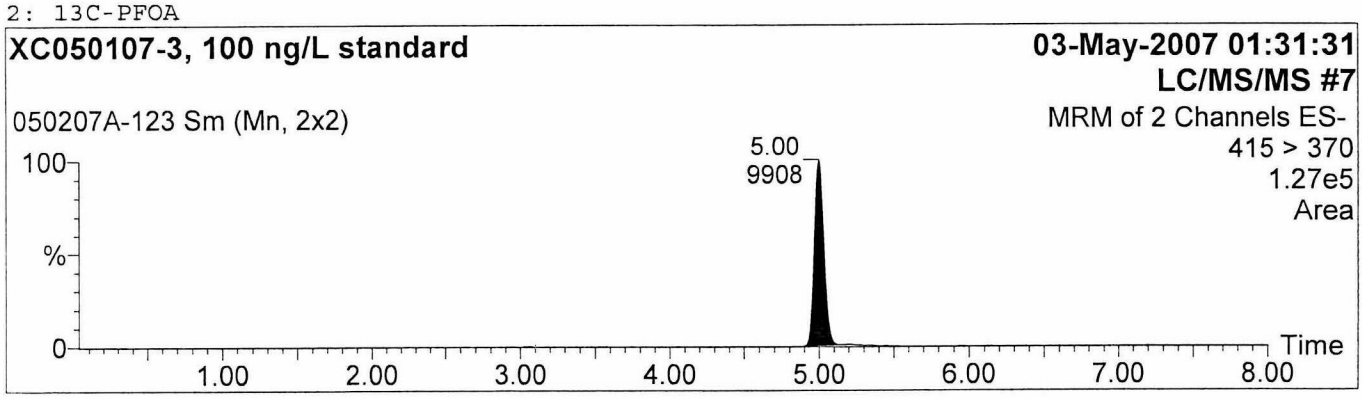
Printed: Thu May 03 11:09:15 2007

Name: 050207A-123
Text:

1: PFOA



2: 13C-PFOA



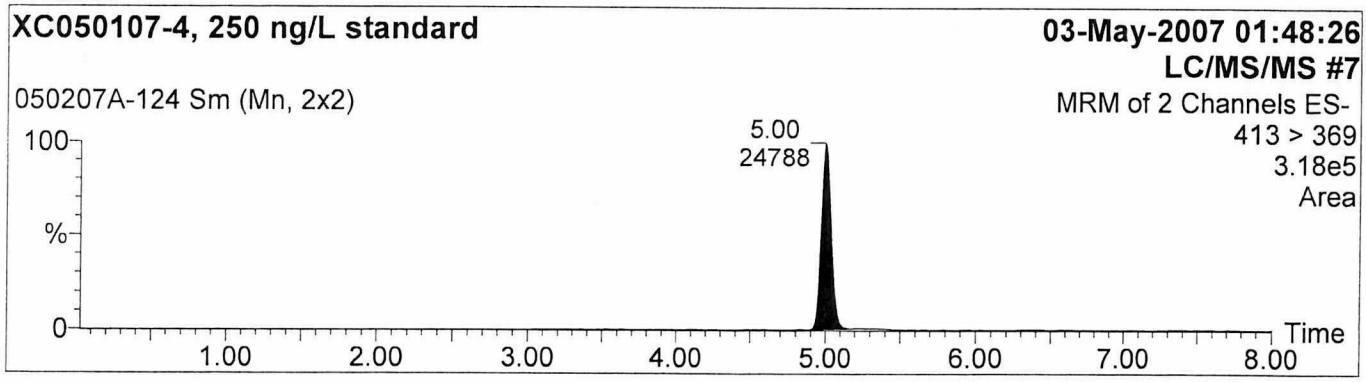
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

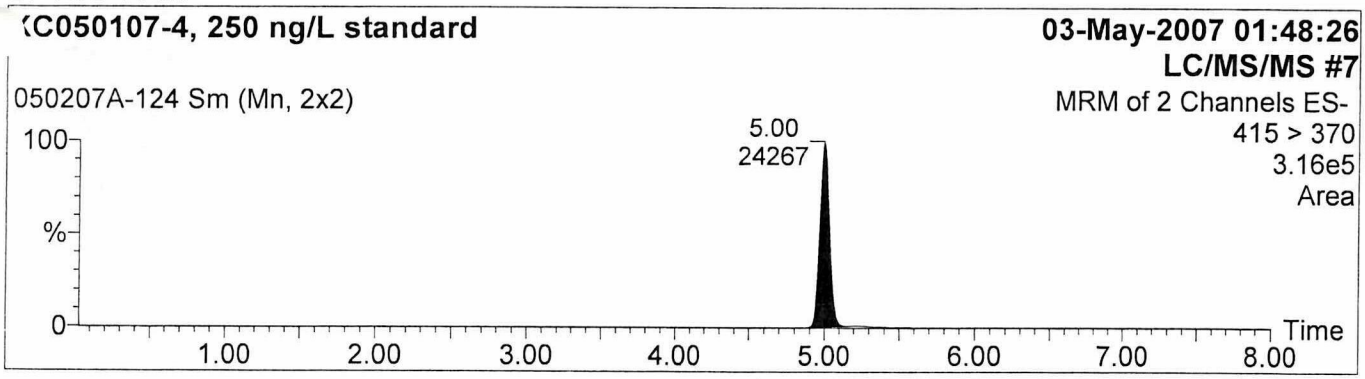
Printed: Thu May 03 11:09:15 2007

Name: 050207A-124
Text:

1: PFOA



2: 13C-PFOA

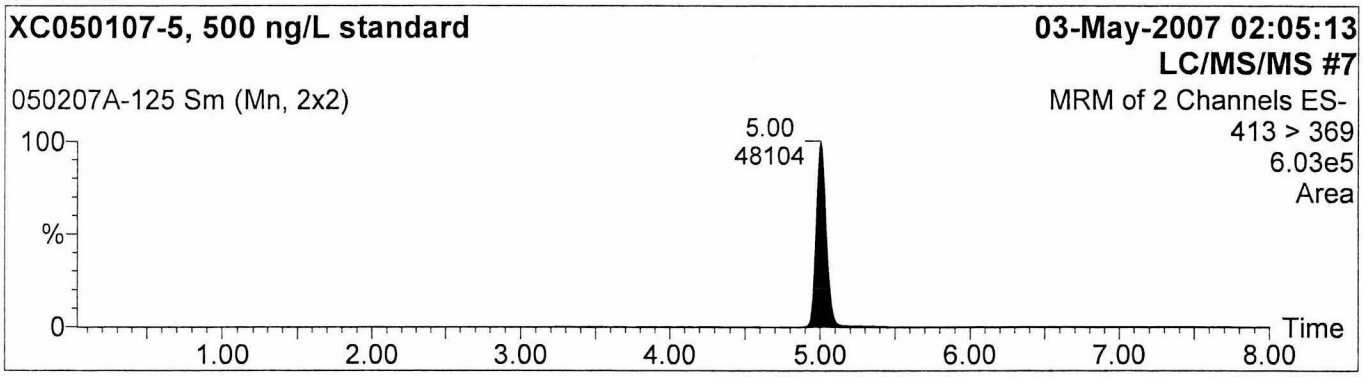


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

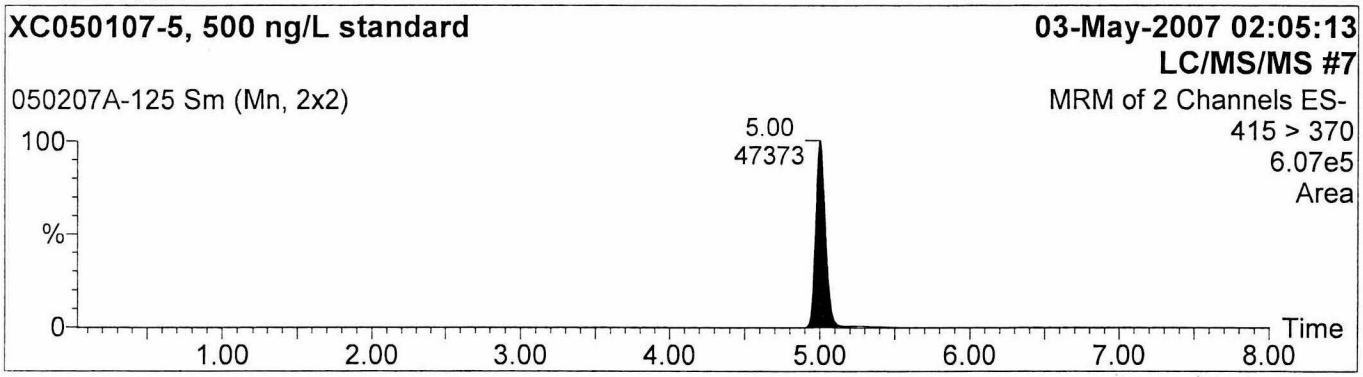
Printed: Thu May 03 11:09:15 2007

Name: 050207A-125
Text:

1: PFOA



2: 13C-PFOA



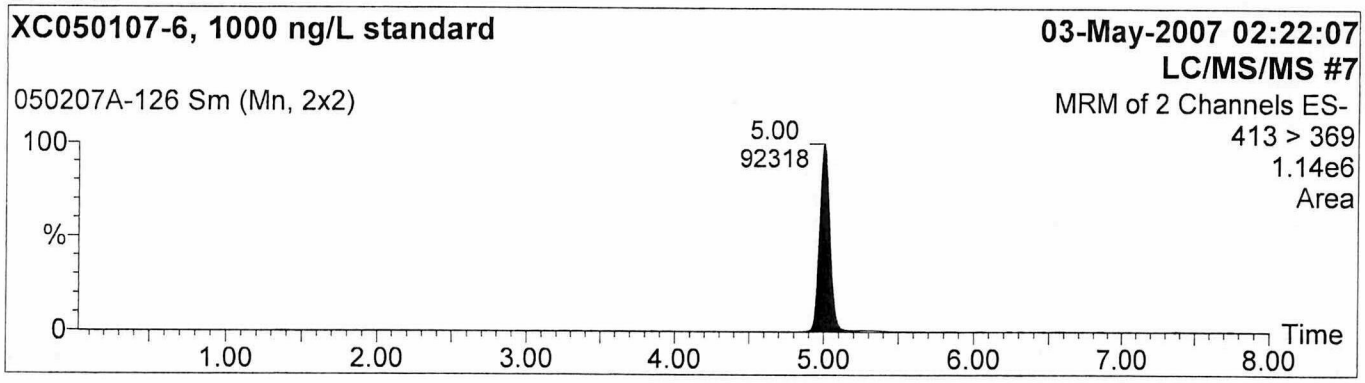
Study No: L11423, Set No: 050207A. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207A NaOH
Last modified: Thu May 03 11:05:13 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

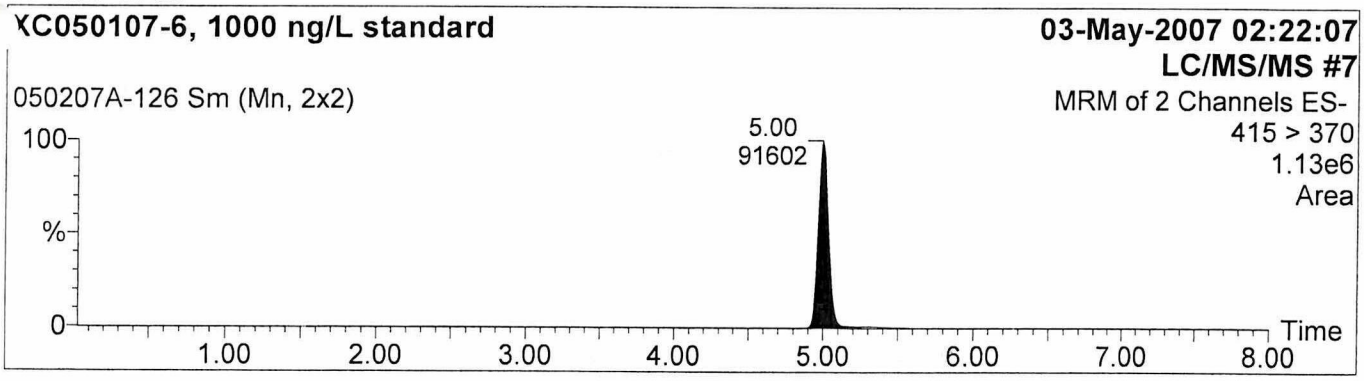
Printed: Thu May 03 11:09:15 2007

Name: 050207A-126
Text:

1: PFOA



2: 13C-PFOA



RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050407C
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	NaOH	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	4.0 mL	Extraction Date:	5/02 & 04/2007
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/05-06/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050407C-301	0	-	-	0	-	-	-	-
XC050107-1	-	CS	050407C-302	25	-	-	3041	-	-	-	-
XC050107-2	-	CS	050407C-303	50	-	-	5575	-	-	-	-
XC050107-3	-	CS	050407C-304	100	-	-	11098	-	-	-	-
XC050107-4	-	CS	050407C-305	250	-	-	25072	-	-	-	-
XC050107-5	-	CS	050407C-306	500	-	-	51124	-	-	-	-
XC050107-6	-	CS	050407C-307	1000	-	-	98106	-	-	-	-
Methanol Wash	-	C	050407C-308	-	-	-	1566	-	-	-	-
Reagent Control	NA	C	050407C-309	-	-	1	0	ND	ND	-	-
Reagent Spk A	NA	LCS	050407C-310	-	-	1	6774	63.7	66.3	50	127
Reagent Spk B	NA	LCS	050407C-311	-	-	1	49592	500	521	500	100
L11423-14 Spk C	SG-A-014	LF	050407C-312	-	100	1000	6378	59700	62200	50000	89
L11423-14 Spk C	SG-A-014	LF	050407C-313	-	100	10	592517	*	-	50000	-
Methanol Wash	-	C	050407C-314	-	-	-	1329	-	-	-	-
XC050107-1	-	CS	050407C-315	25	-	-	2660	-	-	-	-
XC050107-2	-	CS	050407C-316	50	-	-	5472	-	-	-	-
L11423-10	SG-A-010	S	050407C-317	-	100	100	14578	14300	14900	-	-
L11423-14	SG-A-014	S	050407C-318	-	100	100	15381	15100	15700	-	-
L11423-14 Rep	SG-A-014	S	050407C-319	-	100	100	15724	15500	16100	-	-
L11423-18	SG-A-018	S	050407C-320	-	100	100	15827	15600	16200	-	-
XC050107-3	-	CS	050407C-321	100	-	-	10550	-	-	-	-
XC050107-4	-	CS	050407C-322	250	-	-	24134	-	-	-	-
XC050107-5	-	CS	050407C-323	500	-	-	48109	-	-	-	-
XC050107-6	-	CS	050407C-324	1000	-	-	98895	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 526.129
Slope = 98.0574
Coef. Of Det. = 0.999524

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.

Spreadsheet prepared by: MLC / 5/10/07

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

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423 Matrix: NaOH Impinger Sample
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 34
Time 8:10 AM Date 5/4/2007 Initials EDN

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-A-014	L11423-14* Spk C	100 mL	40.0	200.0
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
L11423-14* Spk C	SS20796 (1000 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-

All samples were measured:
Time 10:10 AM Date 5/4/2007 Initials EDN

After measuring samples were returned to refrigerator No. 34
Time 3:06 PM Date 5/4/2007 Initials EDN

Comments: *Sample is pre-diluted ten times by taking 4 mL of sample and bringing the volume up to 40 mL with HPLC water

Analysis Summary:
Data Set: 050407C Initials/Date: MLC / 5/4/07
Data Set: - Initials/Date: - / -
Data Set: _____ Initials/Date: _____ / _____

Set extraction/analysis data verified by: CEE Date: 05/24/07

Exygen™ RESEARCH

3058 Research Drive
State College, PA 16801

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

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007C[$$] PROTOCOL NUMBER: NA

MATRIX: NaOH Impinger Sample ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	Reagent Control	↓	↓	-	↓	-	-	Methanol	46314
na	Reagent Spk A	↓	↓	-	↓	-	-	C18 SPE	005337081B
na	Reagent Spk B	↓	↓	-	↓	-	-	HPLC Water	46335
SG-A-014	L11423-14* Spk C	↓	↓	0.4/10	↓	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	*Initials/Date*	EDD 5/4/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C44H04
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date	-	EDD 5/4/07	EDD 5/4/07	MLC 5/4/07	MLC 5/4/07	-	-	*Initials/Date*	MLC 5/4/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

*Sample was diluted ten times prior to extraction.
**Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:

Final extracts stored in refrigerator 32 Initials: EDD Date: 5/4/2007

Exygen™ RESEARCH

3058 Research Drive
State College, PA 16801

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

Exygen STUDY NO. L11423

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Protocol No.: None
Method No.: $$

Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA

Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC050107-0	Methanol	46303
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC050107-1	C18 SPE	0053370818
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC050107-2	Water	46335
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC050107-3	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC050107-4	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC050107-5	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC050107-6	-	-
-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/01/07

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) freezer # 34 Time: 9:10 AM — Initials/Date: EDD 1 5/01/07

40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD 1 5/01/07

After measuring, samples returned to (refrigerator) freezer # 34 Time: 11:10 AM — Initials/Date: EDD 1 5/01/07

Samples fortified: — Initials/Date: AMB 1 05/01/07

SPE clean-up: — Initials/Date: EDD 1 5/01/07

Final volume adjusted to 5 mL: — Initials/Date: EDD 1 5/01/07

Extracts placed in refrigerator # 32 — Initials/Date: EDD 1 5/01/07

Comments:

STANDARD EXPIRATION DATE: 5/15/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/10/07

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050407C NaOH.SPL
Printed: Fri May 04 15:06:45 2007 MLC 5/4/07

Page Position: (1, 1)

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/mL)	Conc B	Conc C	Test ID	DF
1	94	050407C-301	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---	0	1
2	95	050407C-302	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
3	96	050407C-303	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
4	97	050407C-304	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
5	98	050407C-305	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
6	99	050407C-306	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
7	100	050407C-307	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1
8	92	050407C-308	---	---	Methanol Wash	---	Blank	---	---	---	0	1
9	55	050407C-309	---	---	Reagent Control	---	Blank	---	---	---	0	1
10	56	050407C-310	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---	0	1
11	57	050407C-311	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500	---	0	1
12	58	050407C-312	---	---	L11423-14 Spk C, 50,000 ng/L, DF=1,000	---	QC	50000	5000	---	0	1000
13	59	050407C-313	---	---	L11423-14 Spk C, 50,000 ng/L, DF=10	---	QC	50000	5000	---	0	10
14	92	050407C-314	---	---	Methanol Wash	---	Blank	---	---	---	0	1
15	95	050407C-315	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
16	96	050407C-316	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
17	60	050407C-317	---	---	L11423-10, DF=100	---	Analyte	---	5000	---	0	100
18	61	050407C-318	---	---	L11423-14, DF=100	---	Analyte	---	5000	---	0	100
19	62	050407C-319	---	---	L11423-14 Rep, DF=100	---	Analyte	---	5000	---	0	100
20	63	050407C-320	---	---	L11423-18, DF=100	---	Analyte	---	5000	---	0	100
21	97	050407C-321	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
22	98	050407C-322	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
23	99	050407C-323	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
24	100	050407C-324	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050407C NaOH.SPL
Printed: Fri May 04 15:06:45 2007 MLC 5/4/07

Exygen STUDY NO. L11423

	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
20	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
21	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
22	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
23	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
24	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/4/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/10/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Fri May 04 15:06:54 2007 MLC 5/4/07

Solvent Delay (mins) : 0.00

Function : 1 MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method Report

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Friday, May 04, 2007 14:57:57

Printed: Friday, May 04, 2007 15:07:04 MLC 5/4/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

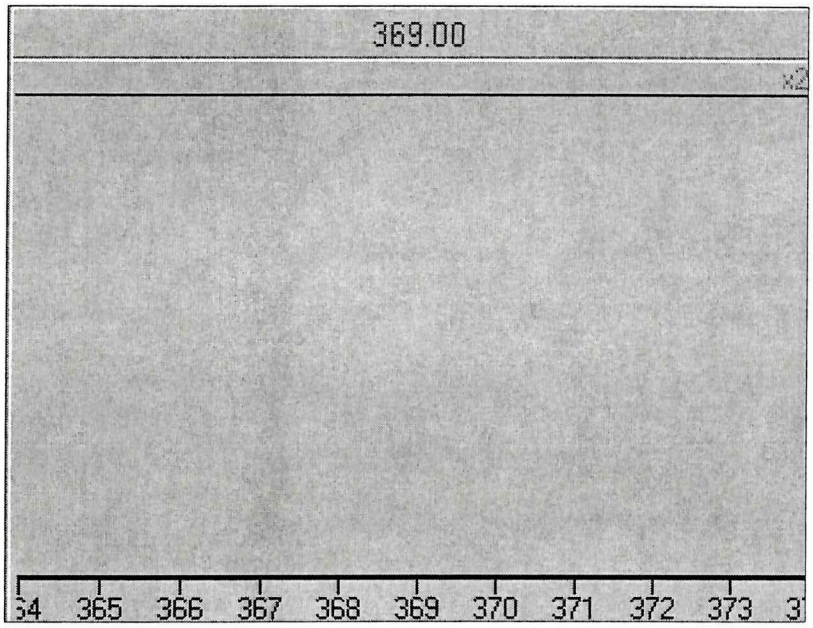
HP1100 Autosampler Initial Conditions

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	77

Tuning Method Report **Page 1**

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Fri May 04 15:07:14 2007 MLC 5/4/07



Dau 413.00

SOURCE (ESP-)	Set	Rdbk	Analyser	Set	Rdbk
Capillary	3.00	-2.94	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	90.0
Gas Cell	2.3e-3	Desolvation	682.4

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050407C
Extraction Date: 5/02 & 04/2007
Analyzed on: 05/05-06/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	3041
XC050107-2	50	5575
XC050107-3	100	11098
XC050107-4	250	25072
XC050107-5	500	51124
XC050107-6	1000	98106

Coefficient of Determination: 0.999635286

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999817626
R Square	0.999635286
Adjusted R Square	0.999544107
Standard Error	784.9073371
Observations	6

MLC
5/10/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	6754384012	6754384012	10963.49368	4.98872E-08
Residual	4	2464318.112	616079.5279		
Total	5	6756848330			

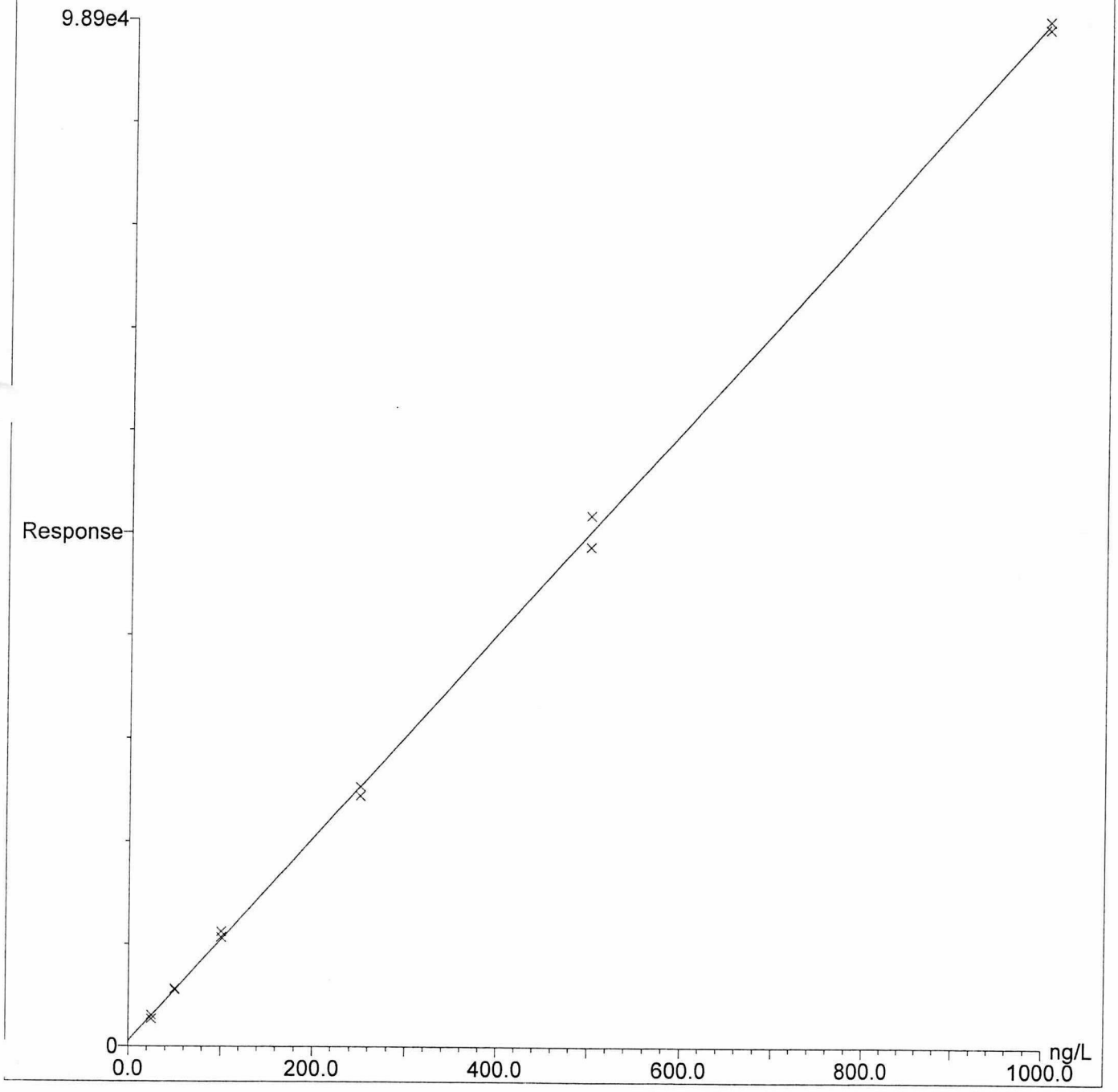
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	999.5829042	438.4601096	2.279757913	0.084809211	-217.7775208	2216.943329	-217.7775208	2216.943329
X Variable 1	97.67194939	0.932814676	104.7067031	4.98872E-08	95.08204065	100.2618581	95.08204065	100.2618581

Quantify Calibration Report
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050407C NaOH
Last modified: Thu May 10 09:42:47 2007
Printed: Thu May 10 09:46:09 2007

MLC 5/10/07

Compound 1 name: PFOA
Coefficient of Determination: 0.999524
Calibration curve: 98.0574 * x + 526.129
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

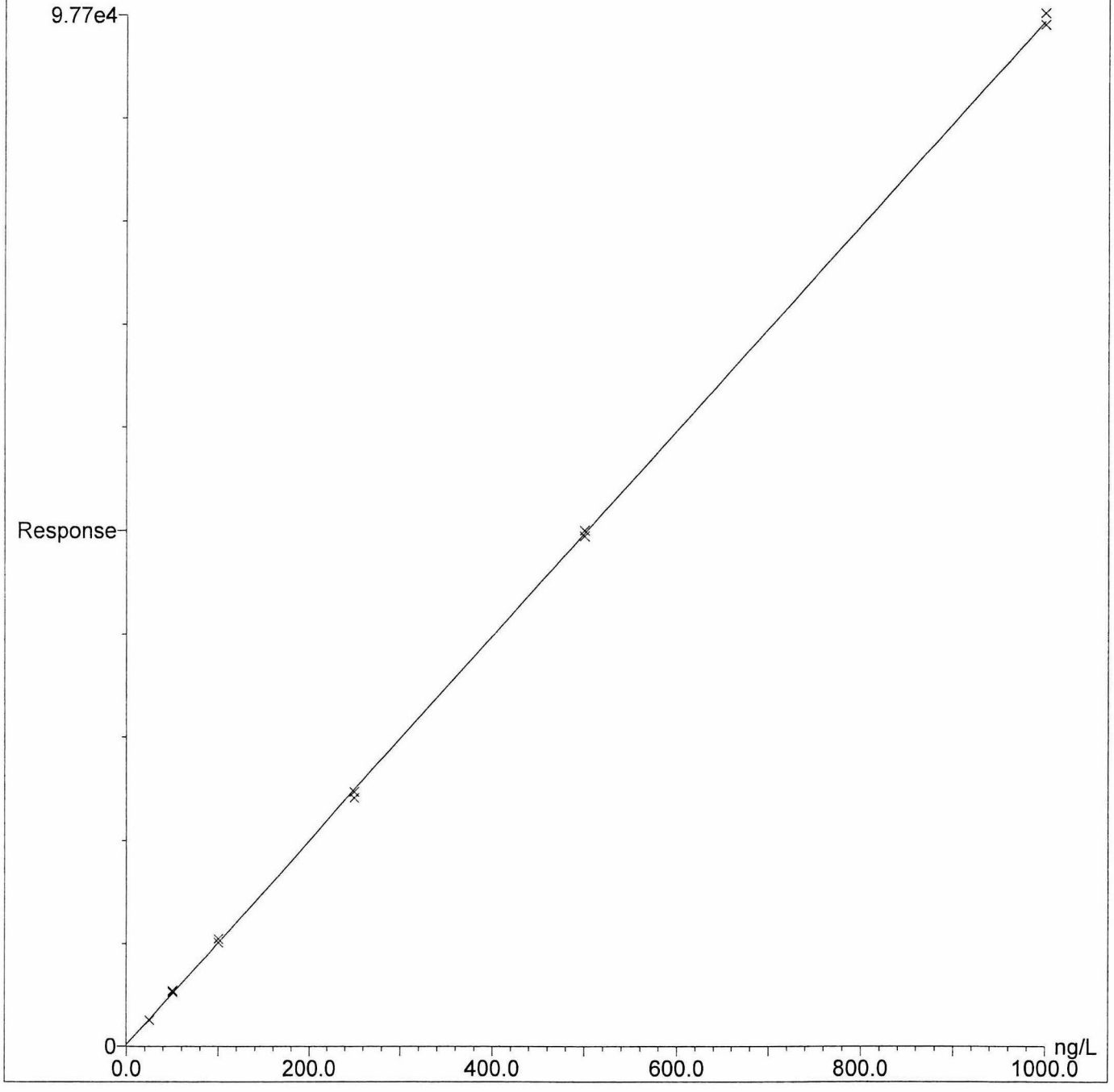


Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050407C NaOH
Last modified: Thu May 10 09:42:47 2007
Printed: Thu May 10 09:46:09 2007

MLC 5/10/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.999864
Calibration curve: 96.7446 * x + 167.613
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

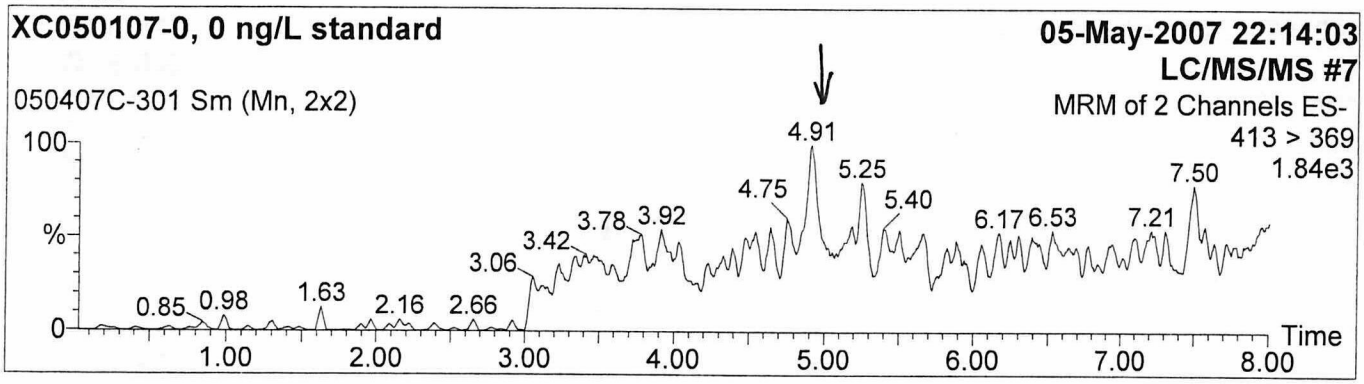
Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

Initials MLC
Date 5/10/07
Run# 050407C-301 To 050407C-324

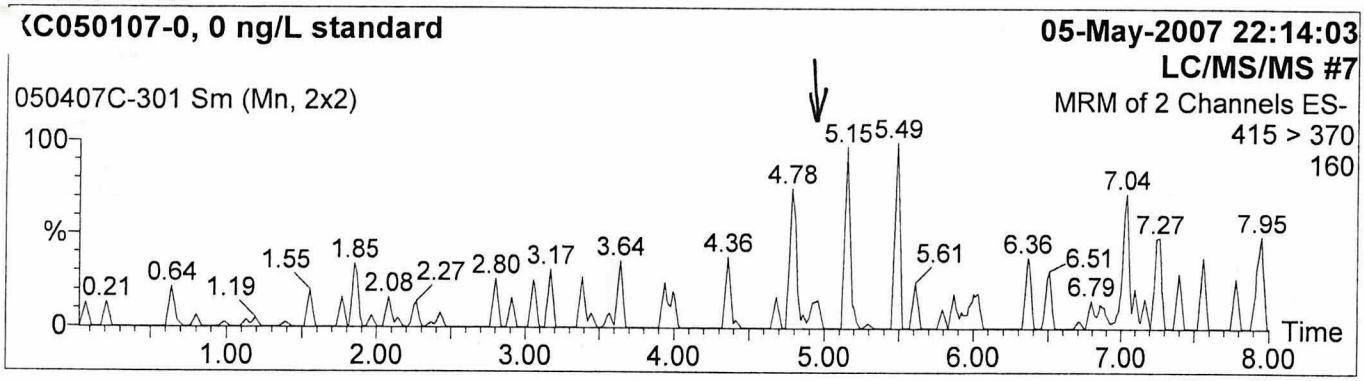
Printed: Thu May 10 09:46:10 2007

Name: 050407C-301
Text:

1: PFOA



2: 13C-PFOA



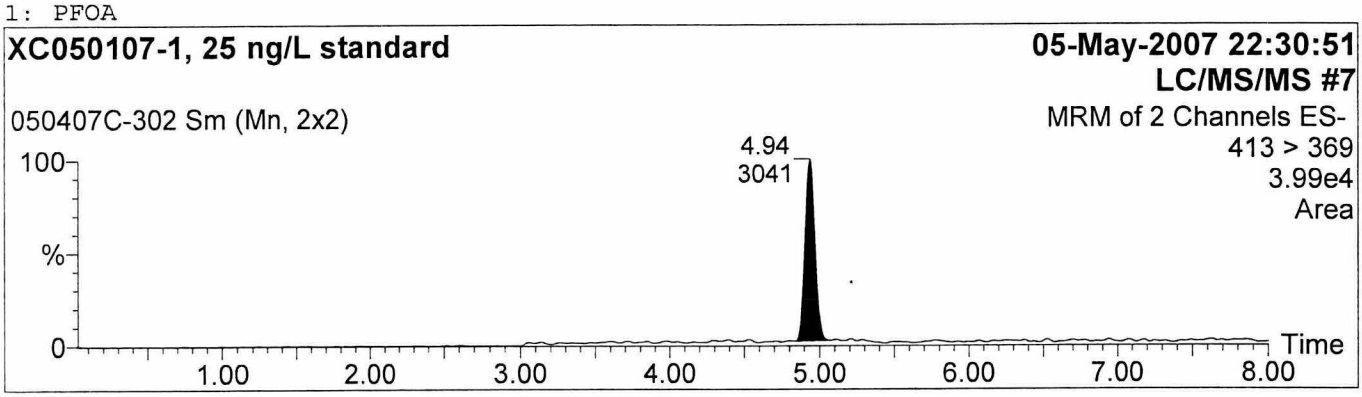
Quantify Sample Report
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

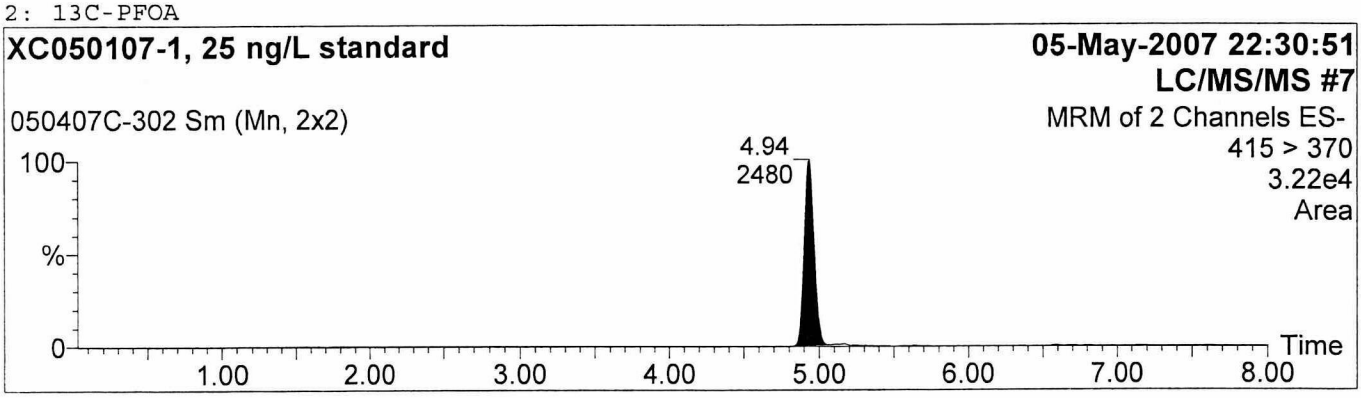
Printed: Thu May 10 09:46:10 2007

Name: 050407C-302
Text:

1: PFOA



2: 13C-PFOA



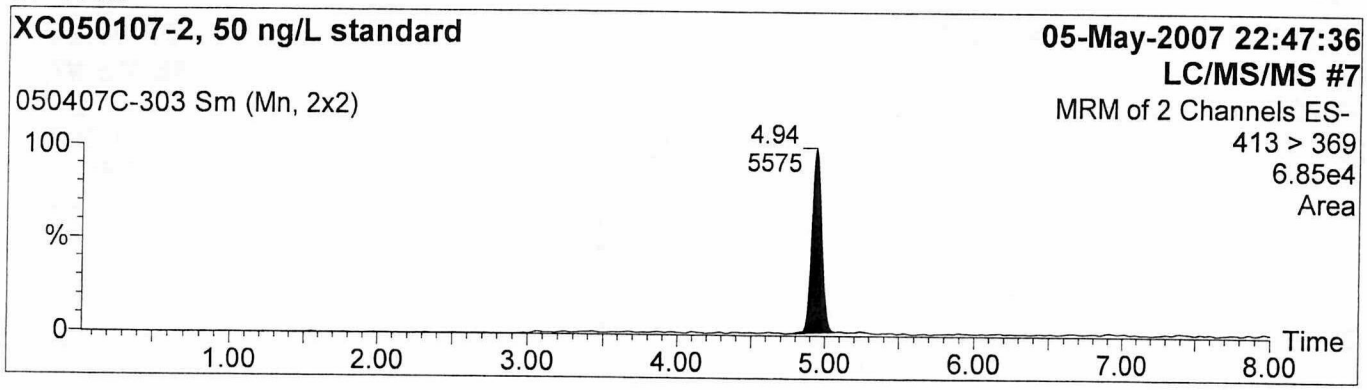
Quantify Sample Report
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

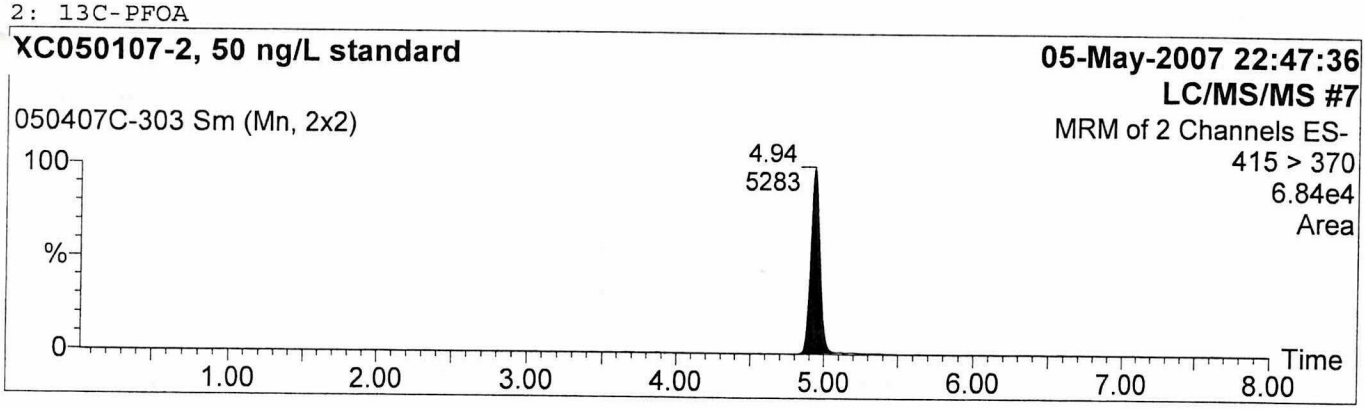
Printed: Thu May 10 09:46:10 2007

Name: 050407C-303
Text:

1: PFOA



2: 13C-PFOA



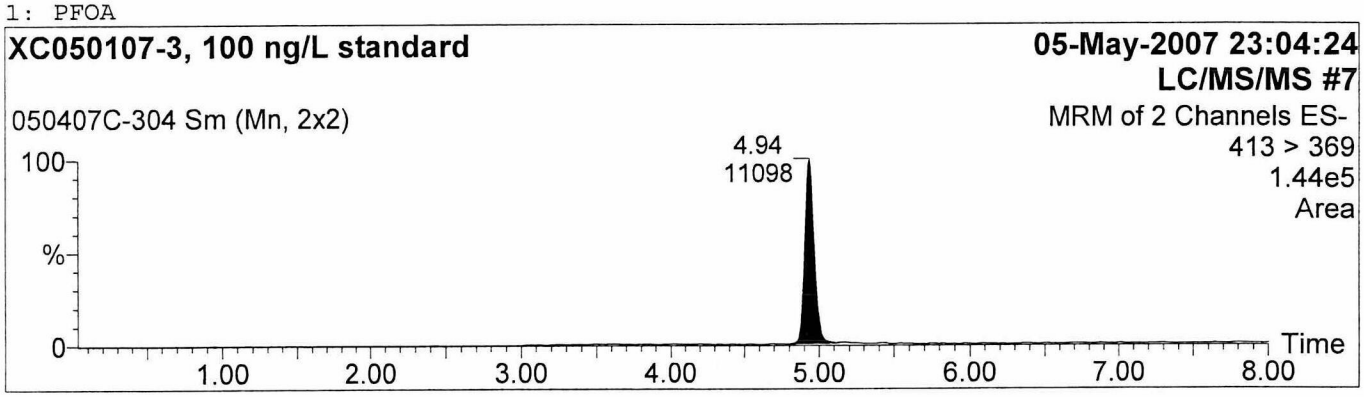
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

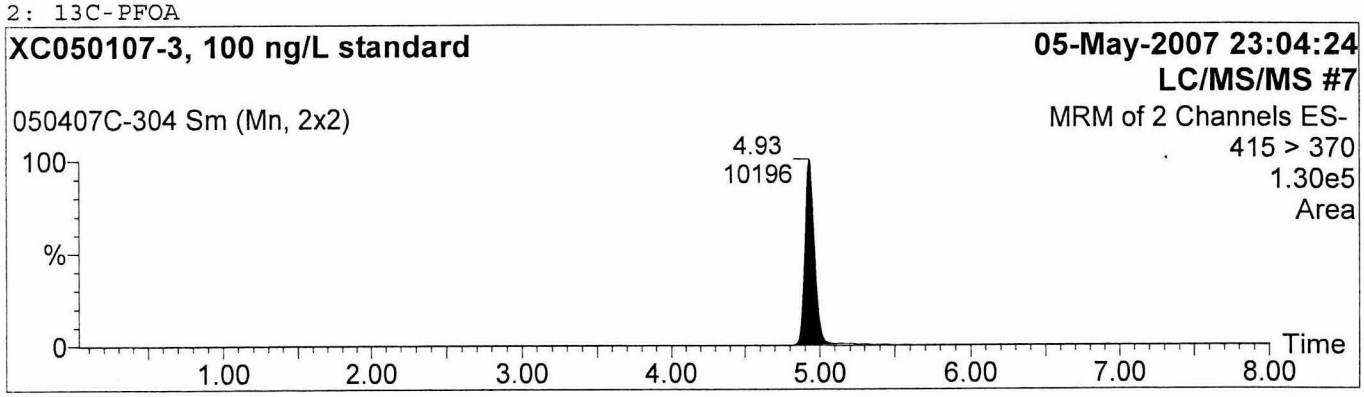
Printed: Thu May 10 09:46:10 2007

Name: 050407C-304
Text:

1: PFOA



2: 13C-PFOA



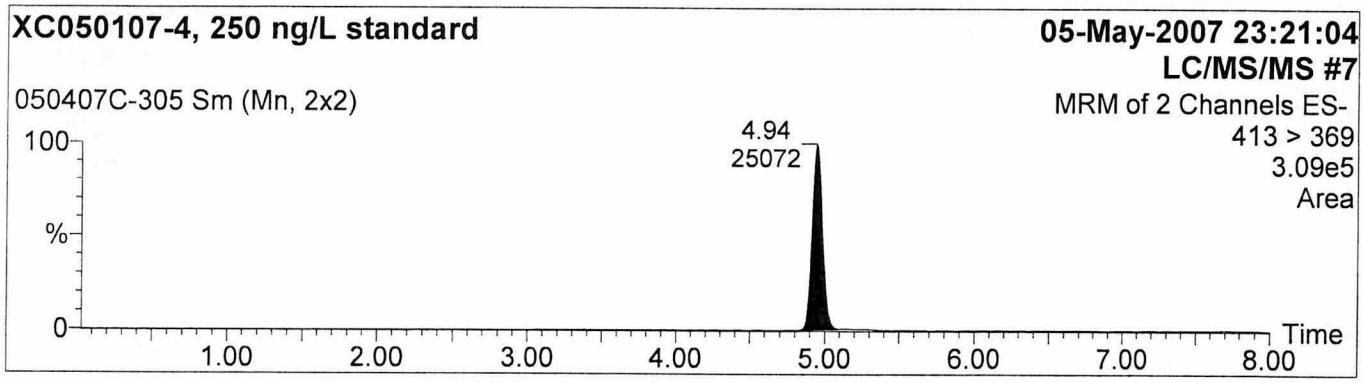
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

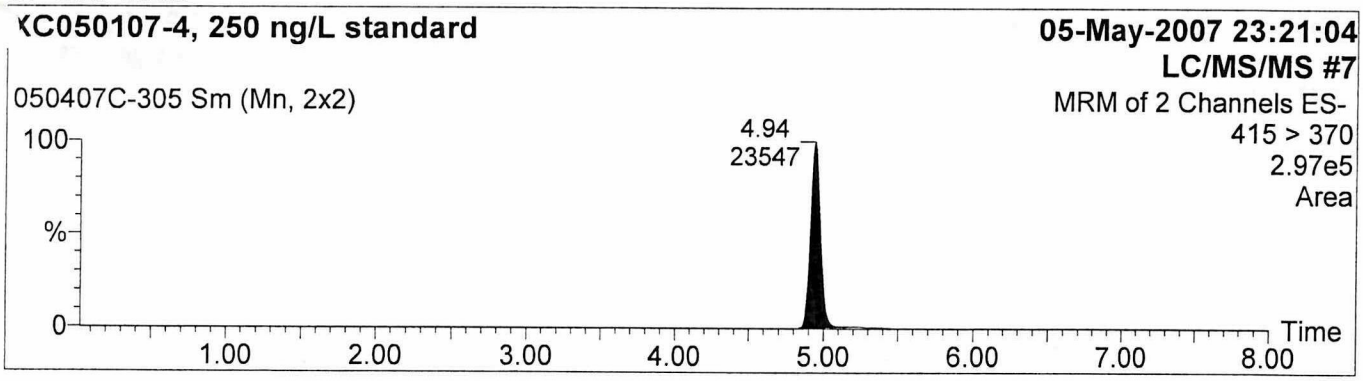
Printed: Thu May 10 09:46:10 2007

Name: 050407C-305
Text:

1: PFOA



2: 13C-PFOA



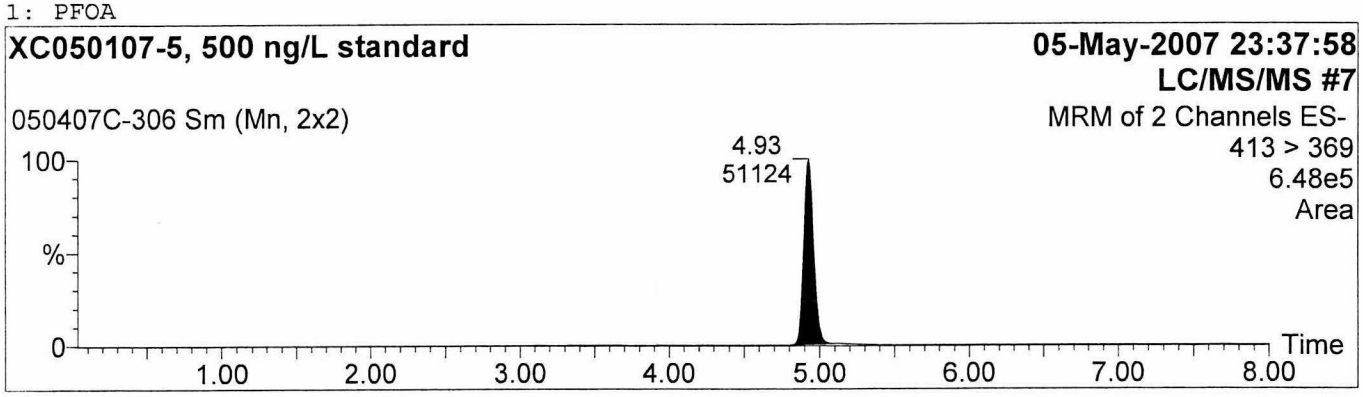
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

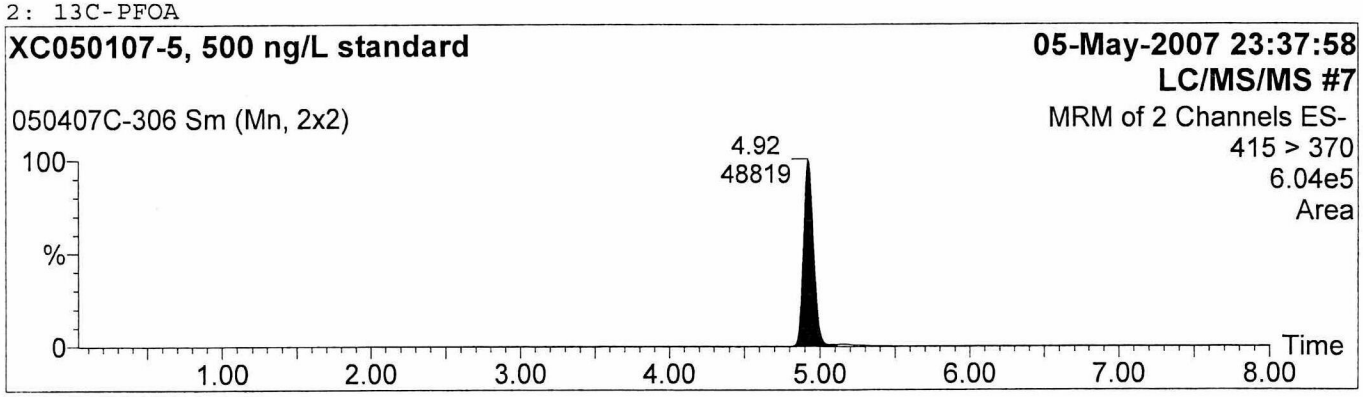
Printed: Thu May 10 09:46:10 2007

Name: 050407C-306
Text:

1: PFOA



2: 13C-PFOA



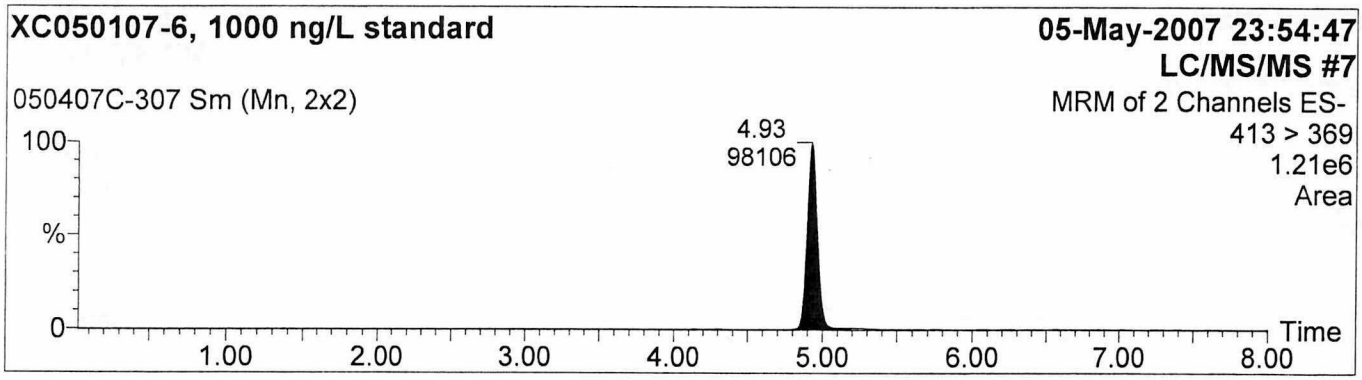
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

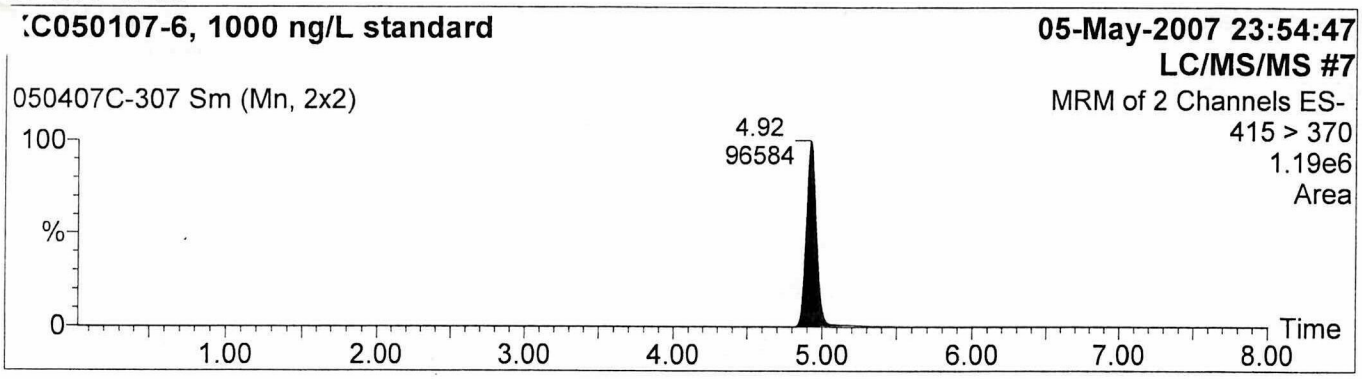
Printed: Thu May 10 09:46:10 2007

Name: 050407C-307
Text:

1: PFOA



2: 13C-PFOA



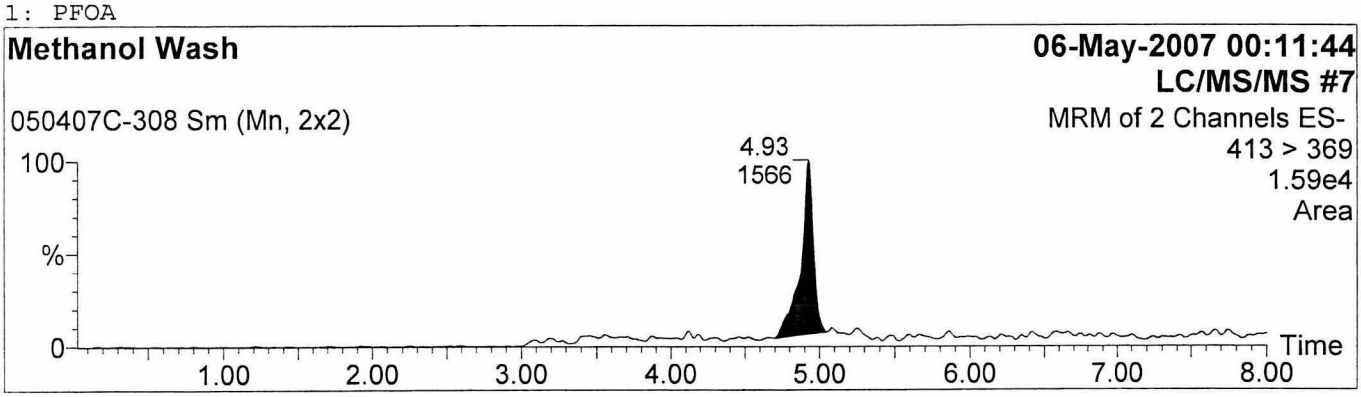
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

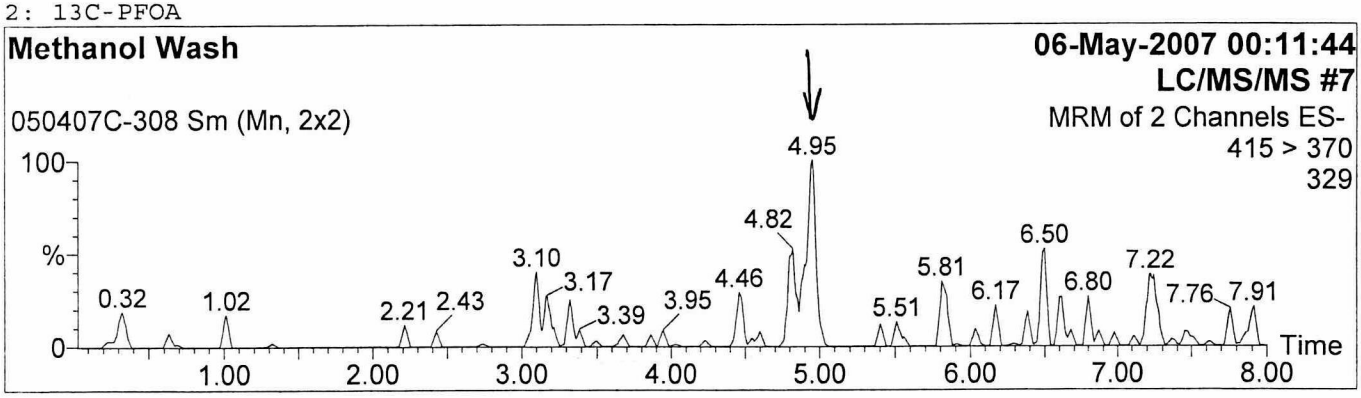
Printed: Thu May 10 09:46:10 2007

Name: 050407C-308
Text:

1: PFOA



2: 13C-PFOA



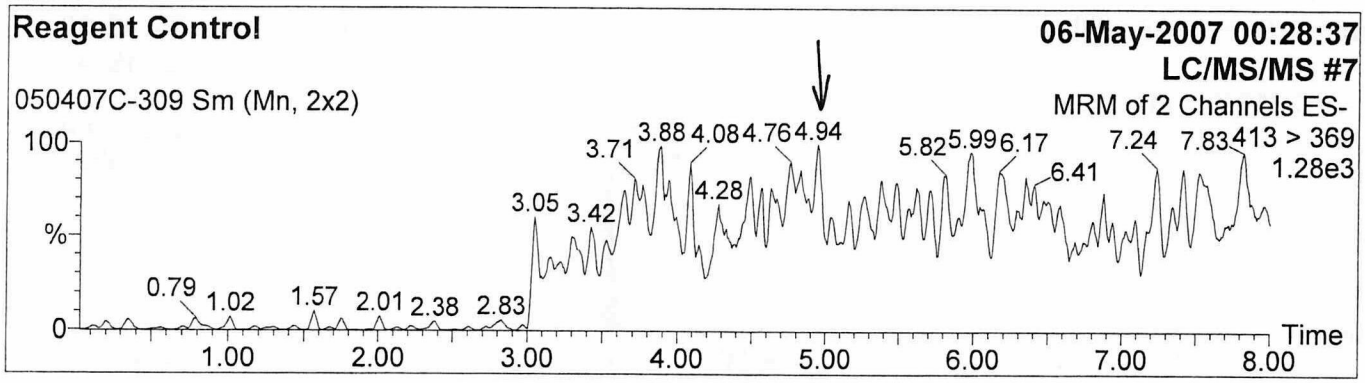
Quantify Sample Report
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

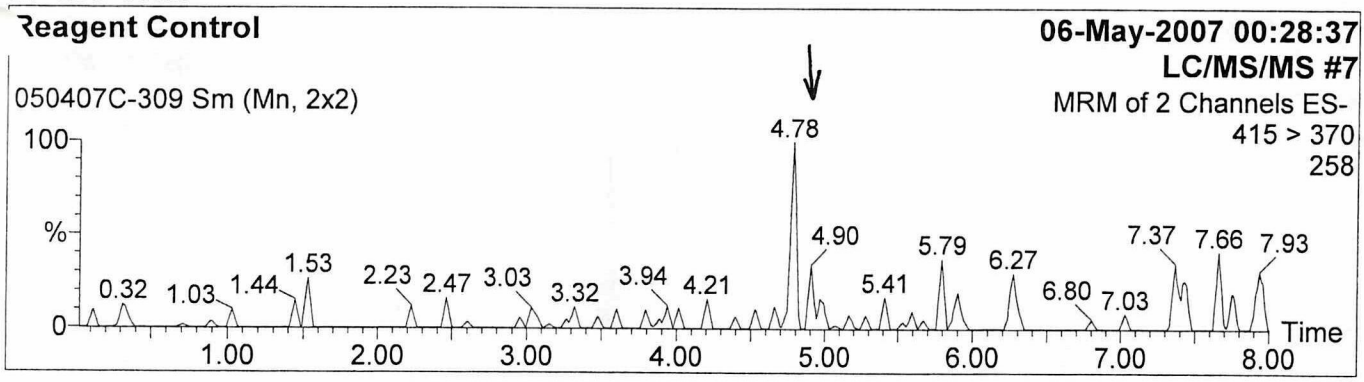
Printed: Thu May 10 09:46:10 2007

Name: 050407C-309
Text:

1: PFOA



2: 13C-PFOA



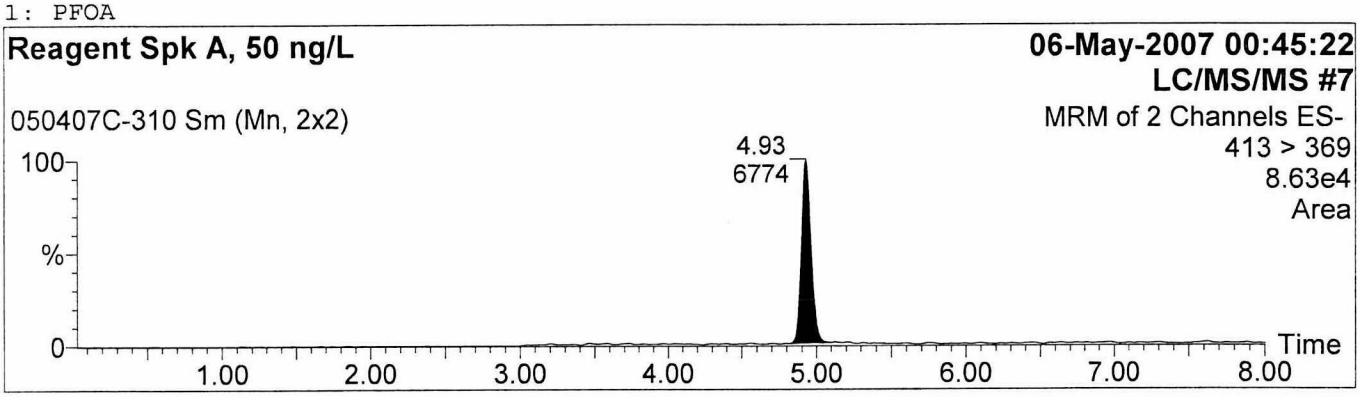
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

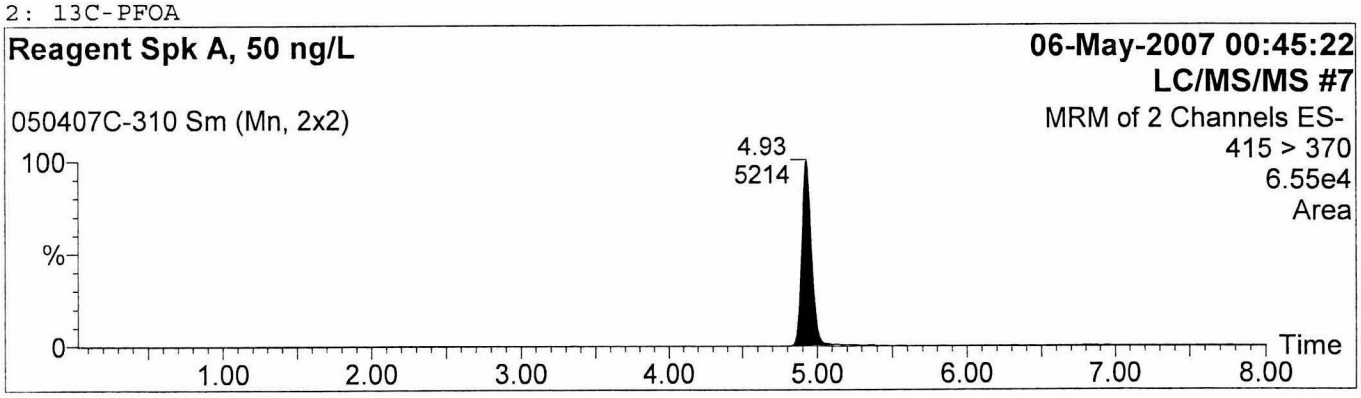
Printed: Thu May 10 09:46:10 2007

Name: 050407C-310
Text:

1: PFOA



2: 13C-PFOA



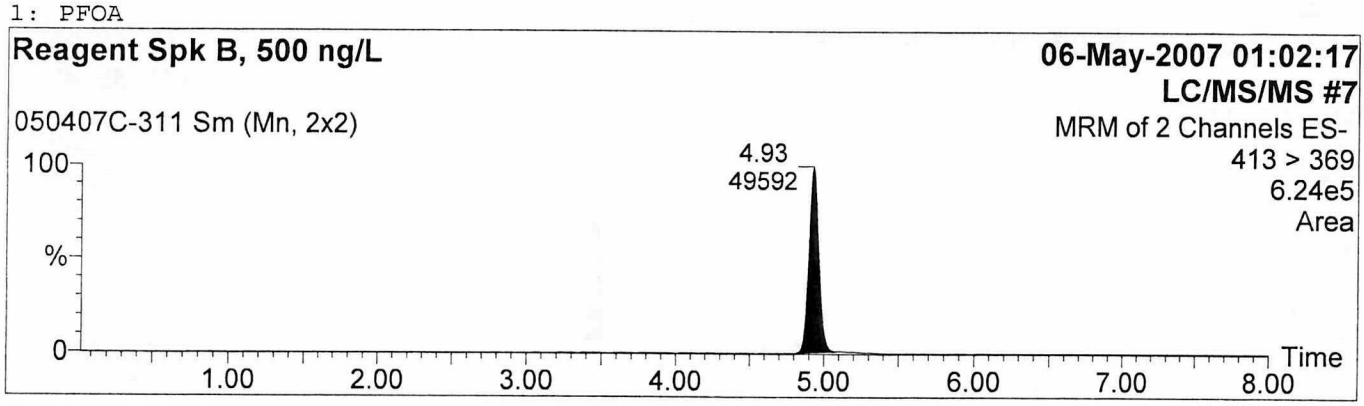
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

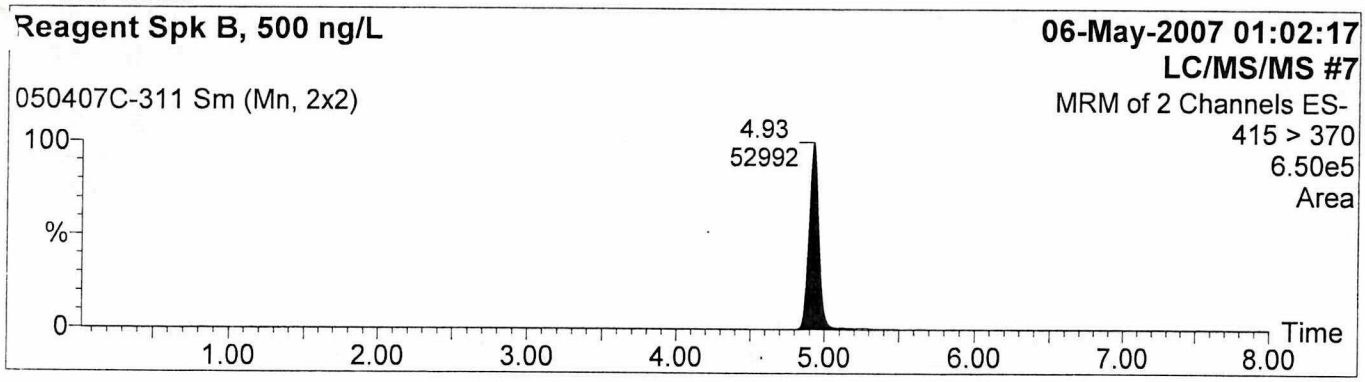
Printed: Thu May 10 09:46:10 2007

Name: 050407C-311
Text:

1: PFOA



2: 13C-PFOA



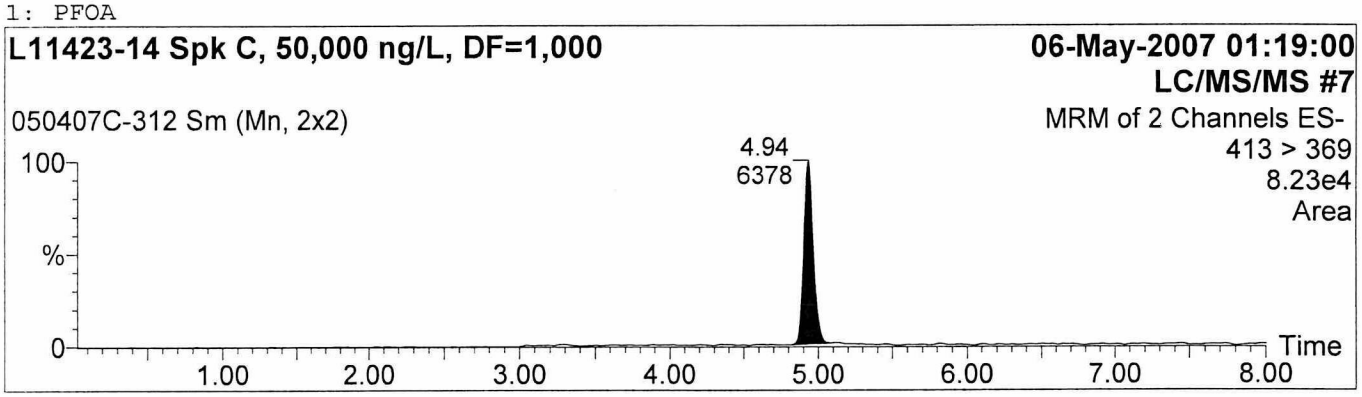
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

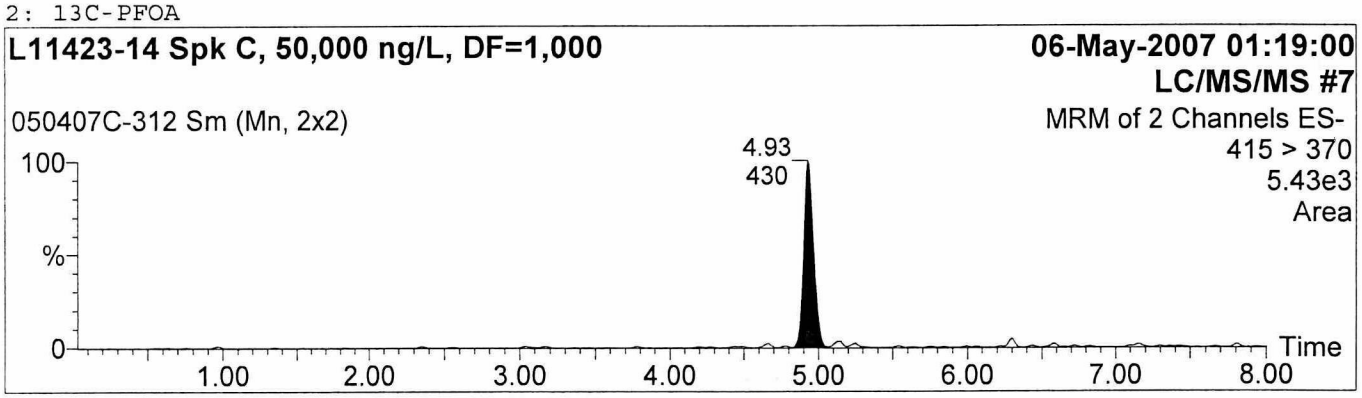
Printed: Thu May 10 09:46:10 2007

Name: 050407C-312
Text:

1: PFOA



2: 13C-PFOA



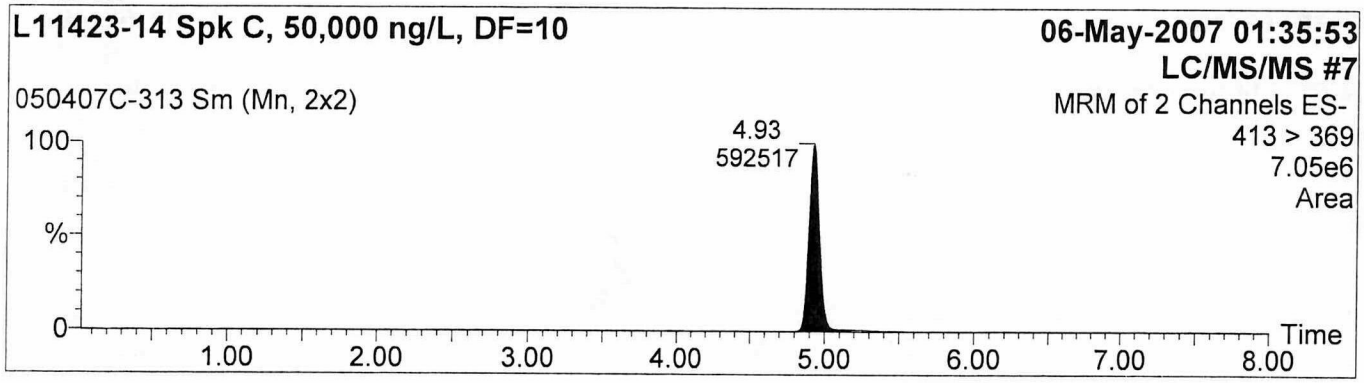
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

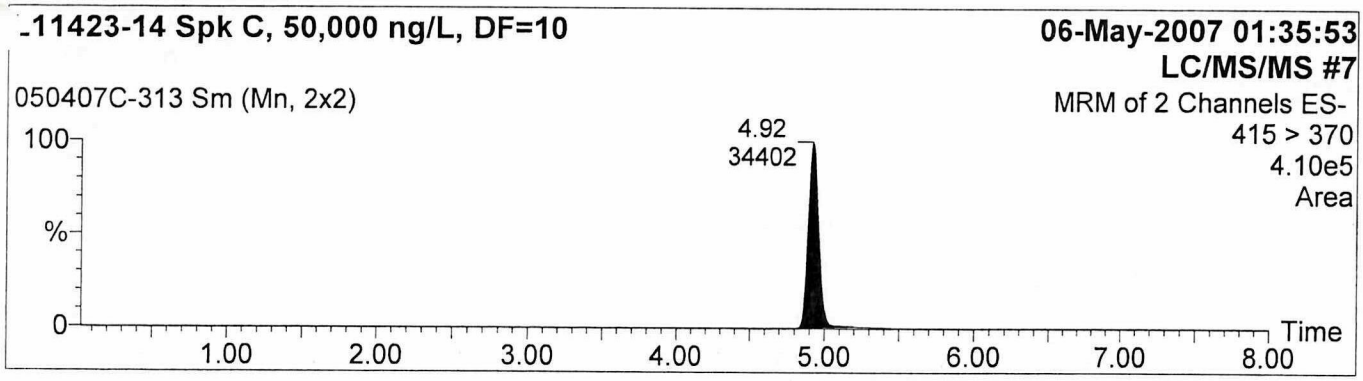
Printed: Thu May 10 09:46:10 2007

Name: 050407C-313
Text:

1: PFOA



2: 13C-PFOA



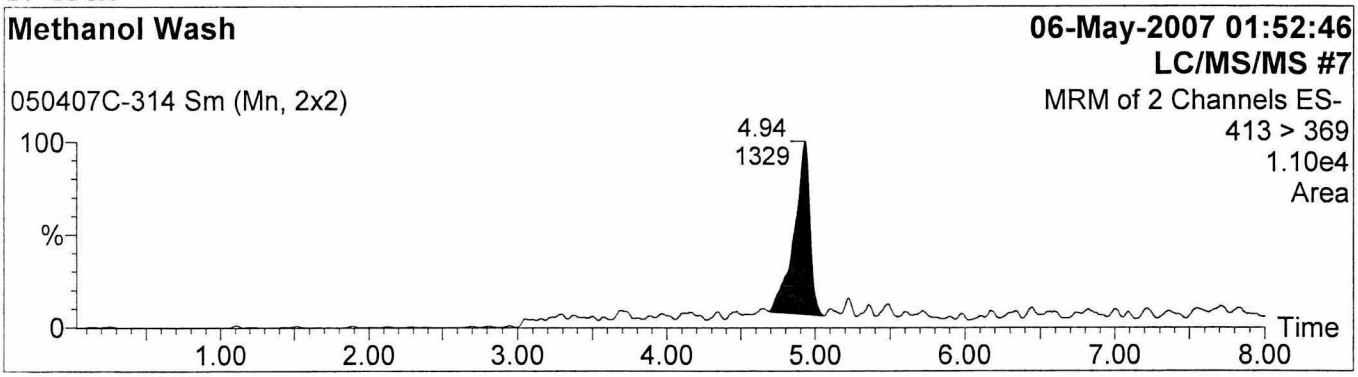
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

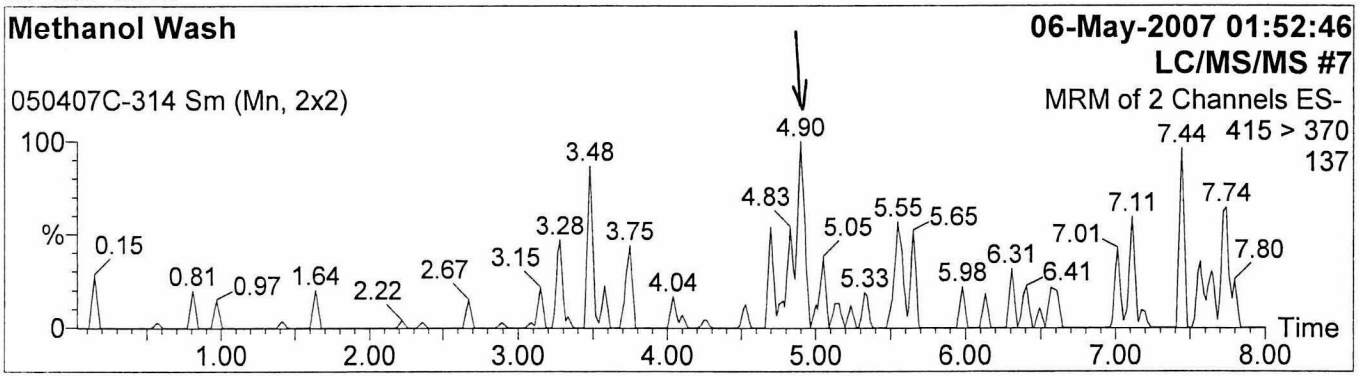
Printed: Thu May 10 09:46:10 2007

Name: 050407C-314
Text:

1: PFOA



2: 13C-PFOA



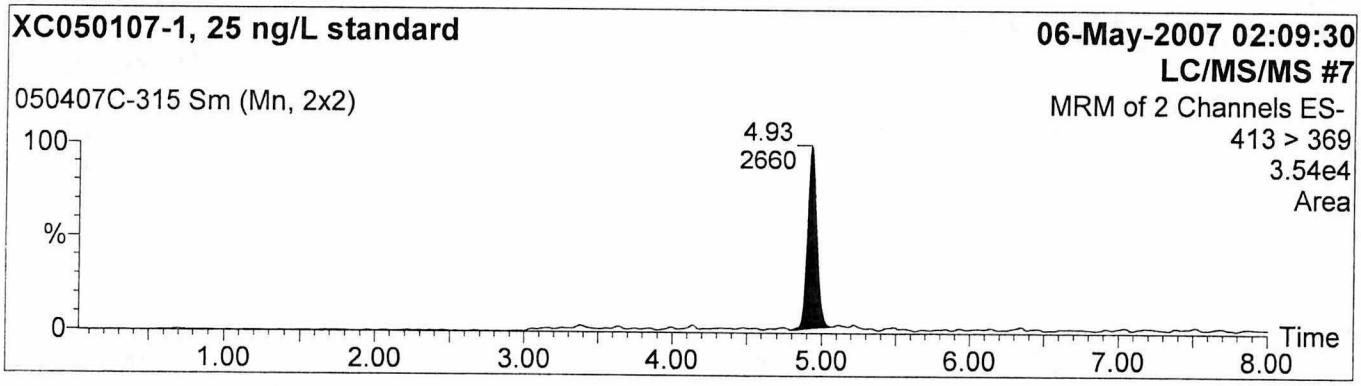
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

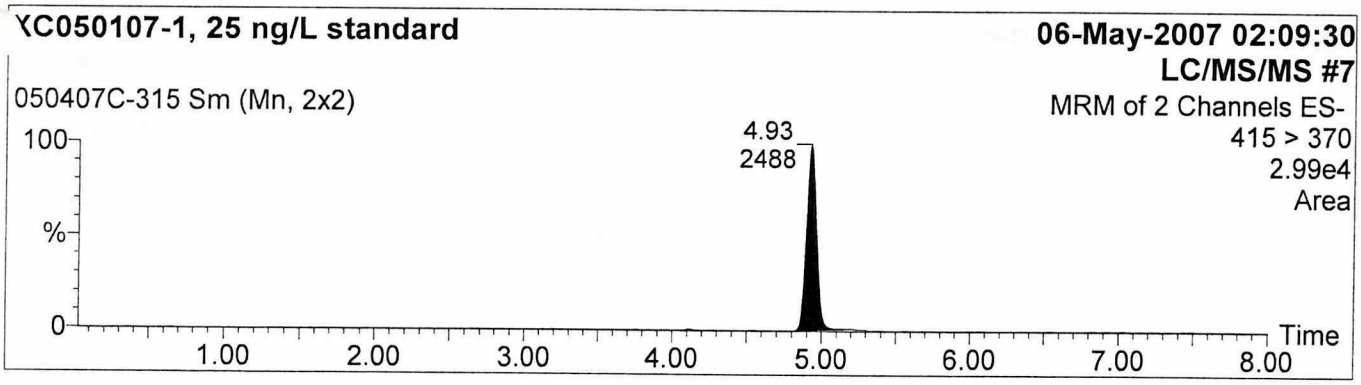
Printed: Thu May 10 09:46:10 2007

Name: 050407C-315
Text:

1: PFOA



2: 13C-PFOA



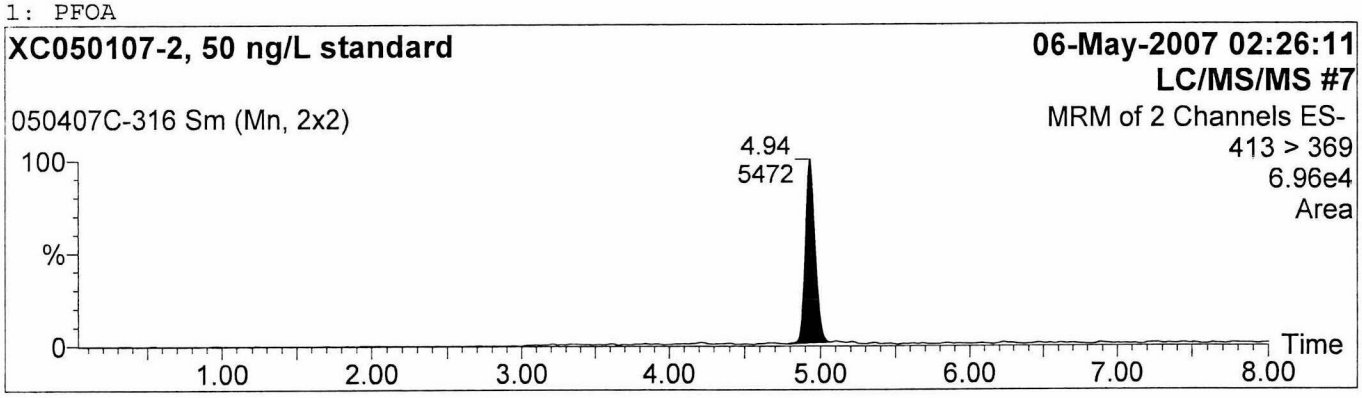
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

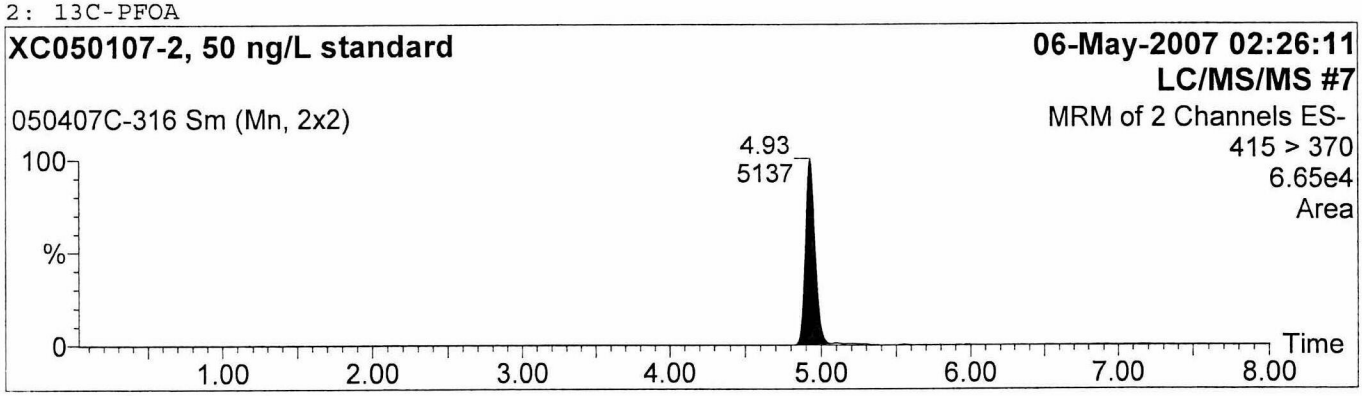
Printed: Thu May 10 09:46:10 2007

Name: 050407C-316
Text:

1: PFOA



2: 13C-PFOA



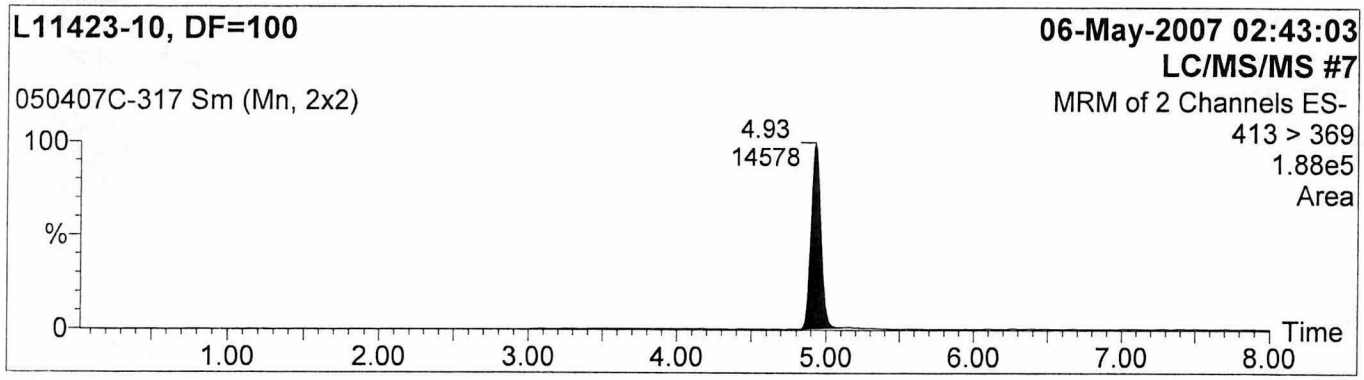
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

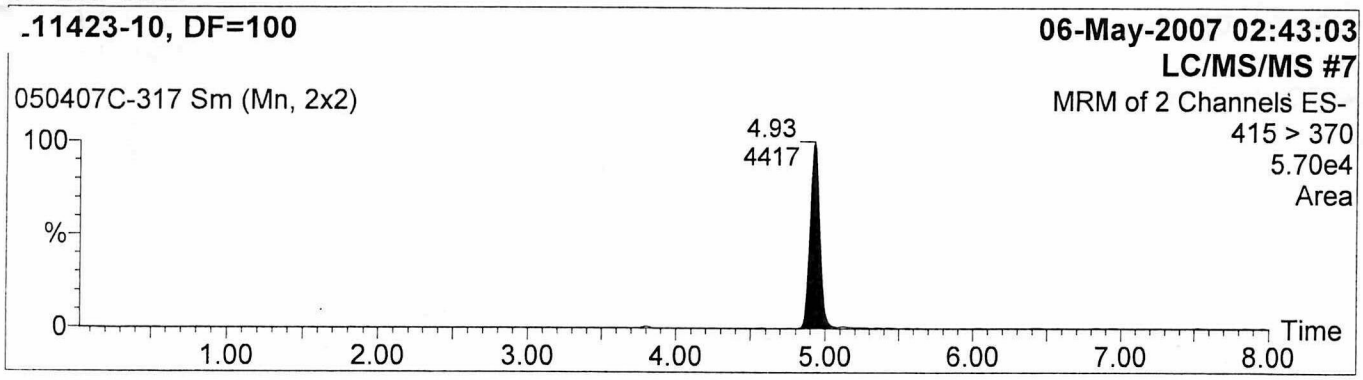
Printed: Thu May 10 09:46:10 2007

Name: 050407C-317
Text:

1: PFOA



2: 13C-PFOA



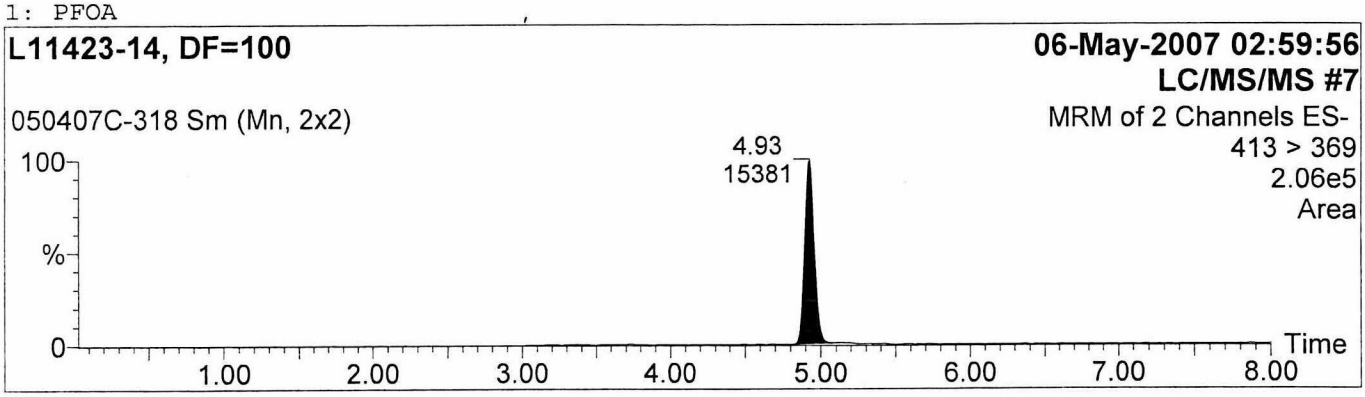
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

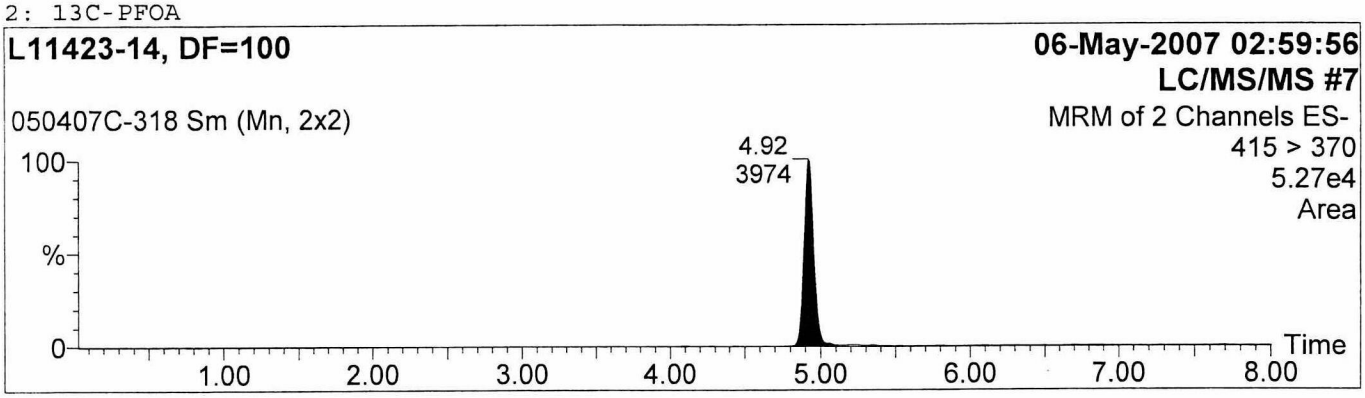
Printed: Thu May 10 09:46:10 2007

Name: 050407C-318
Text:

1: PFOA



2: 13C-PFOA



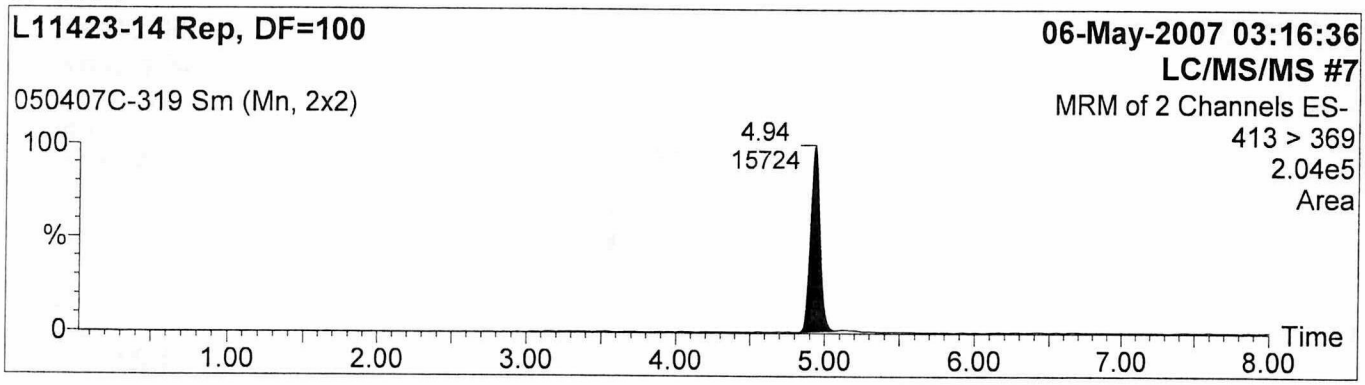
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

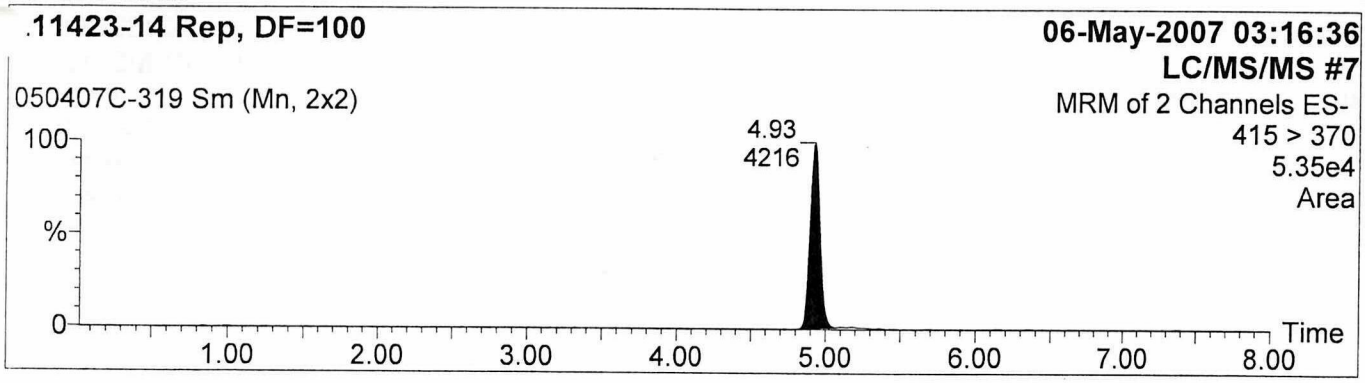
Printed: Thu May 10 09:46:10 2007

Name: 050407C-319
Text:

1: PFOA



2: 13C-PFOA

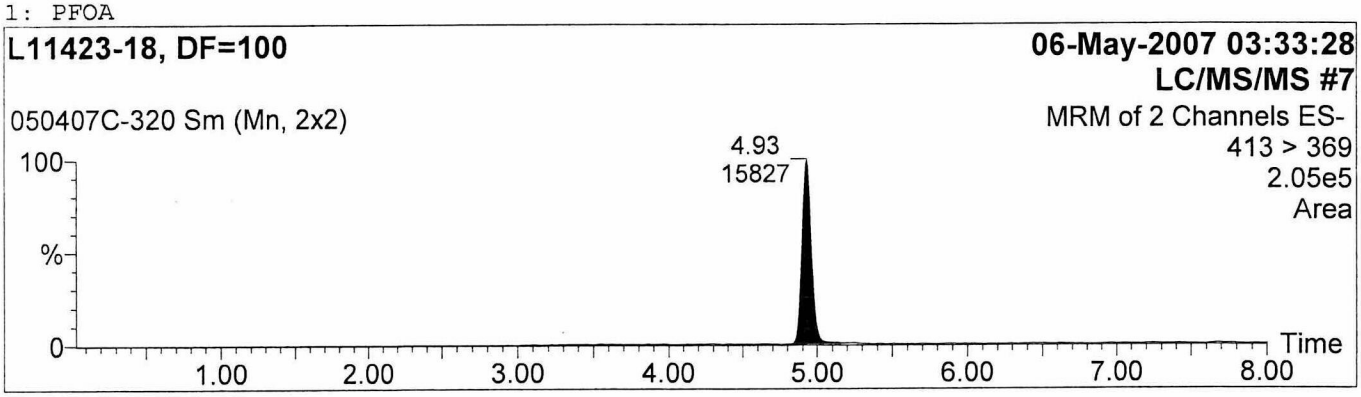


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

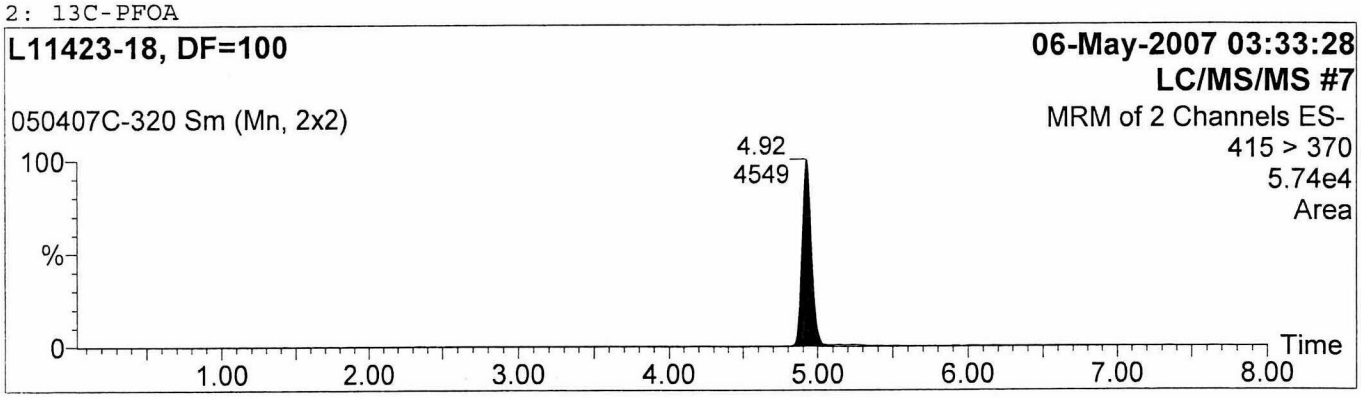
Printed: Thu May 10 09:46:10 2007

Name: 050407C-320
Text:

1: PFOA



2: 13C-PFOA



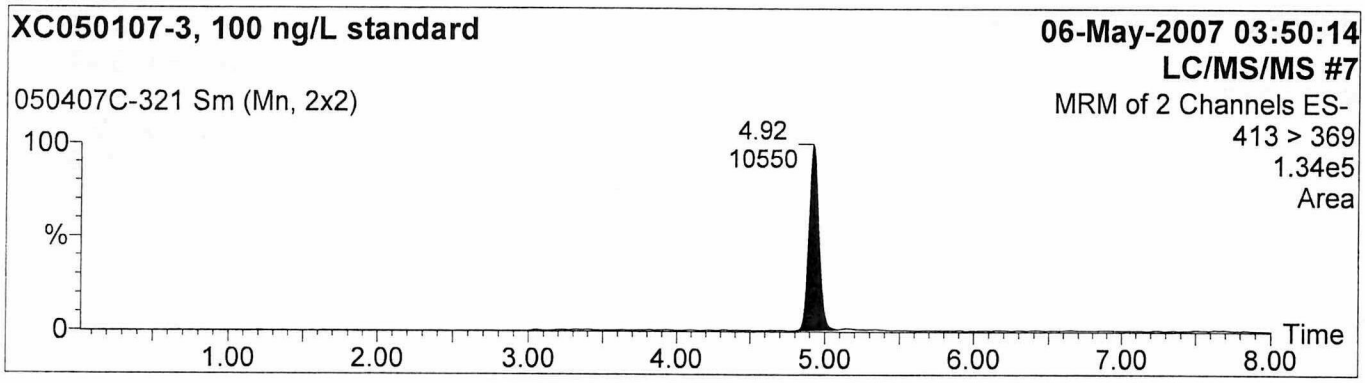
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

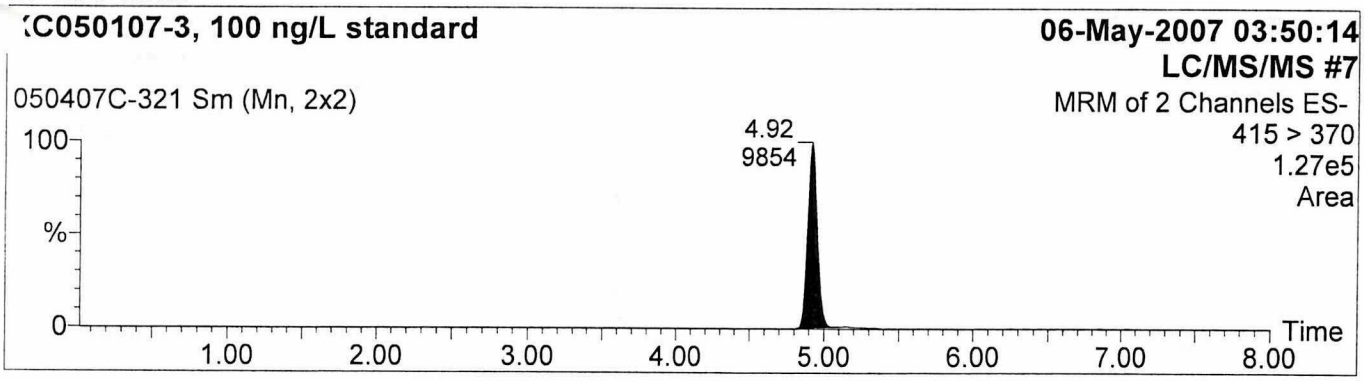
Printed: Thu May 10 09:46:10 2007

Name: 050407C-321
Text:

1: PFOA



2: 13C-PFOA



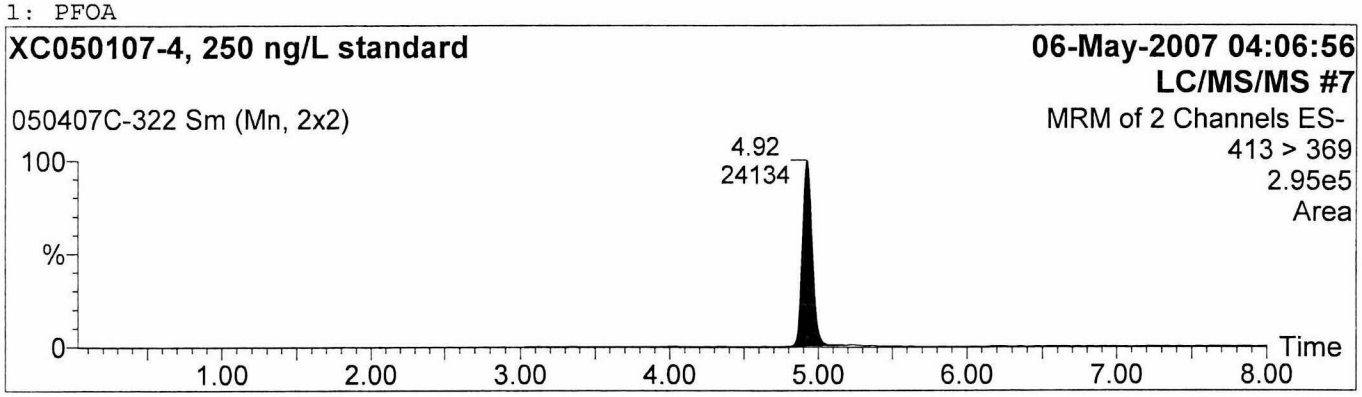
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

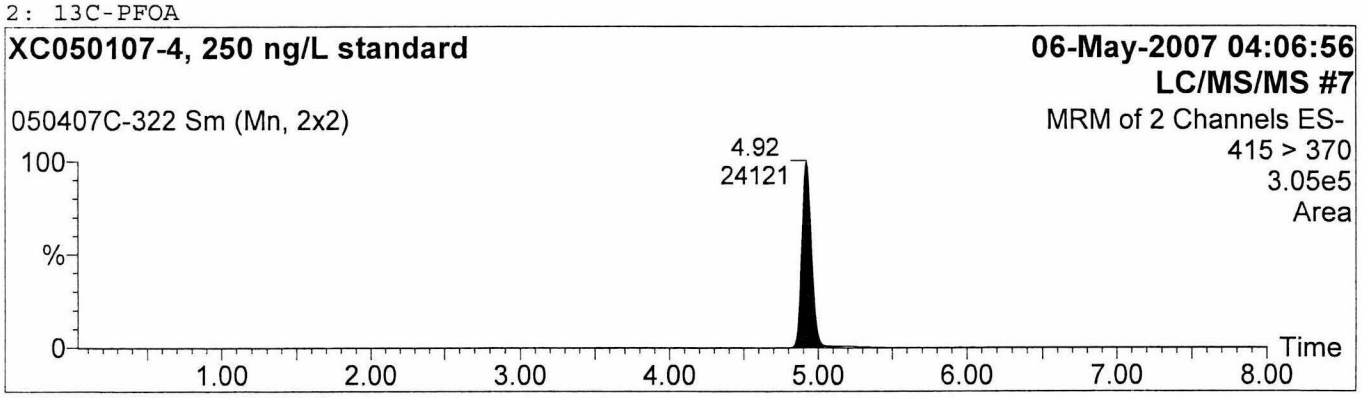
Printed: Thu May 10 09:46:10 2007

Name: 050407C-322
Text:

1: PFOA



2: 13C-PFOA



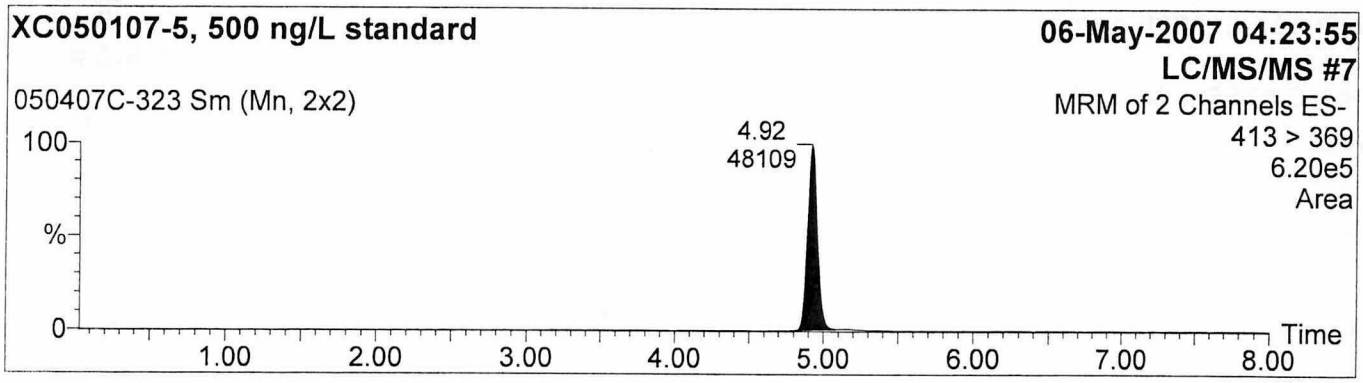
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

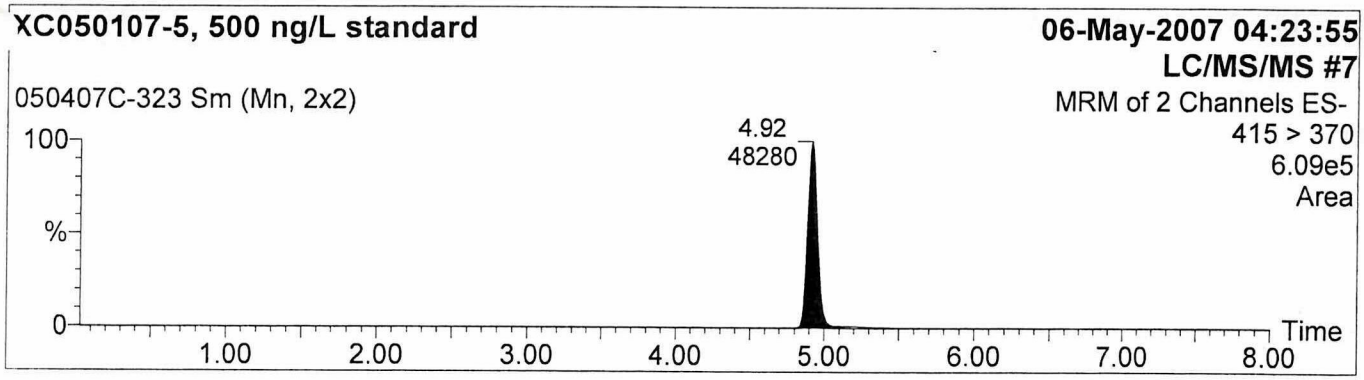
Printed: Thu May 10 09:46:10 2007

Name: 050407C-323
Text:

1: PFOA



2: 13C-PFOA



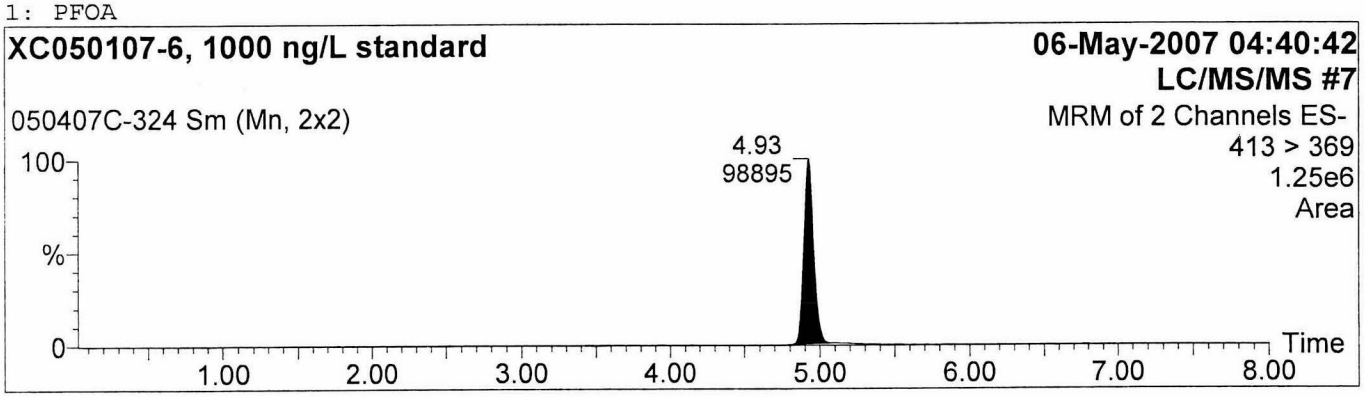
Study No: L11423, Set No: 050407C. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407C NaOH
Last modified: Thu May 10 09:40:46 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

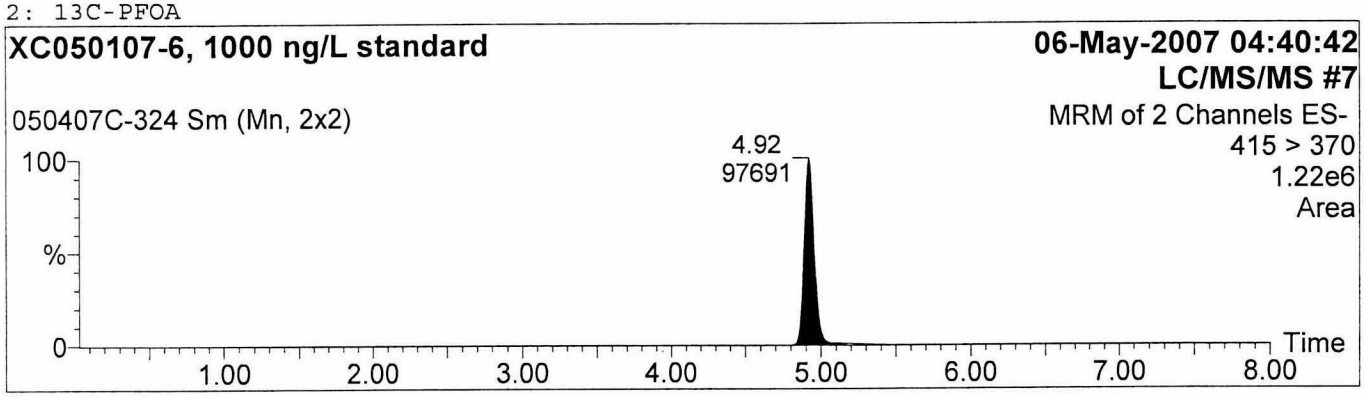
Printed: Thu May 10 09:46:10 2007

Name: 050407C-324
Text:

1: PFOA



2: 13C-PFOA



RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050207C
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	Buffer	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	4.0 mL	Extraction Date:	5/2/2007
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	5/5/2007

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050207C-301	0	-	-	0	-	-	-	-
XC050107-1	-	CS	050207C-302	25	-	-	2742	-	-	-	-
XC050107-2	-	CS	050207C-303	50	-	-	5366	-	-	-	-
XC050107-3	-	CS	050207C-304	100	-	-	10168	-	-	-	-
XC050107-4	-	CS	050207C-305	250	-	-	23900	-	-	-	-
XC050107-5	-	CS	050207C-306	500	-	-	52281	-	-	-	-
XC050107-6	-	CS	050207C-307	1000	-	-	93200	-	-	-	-
Methanol Wash	-	C	050207C-308	-	-	-	0	-	-	-	-
Reagent Control	NA	C	050207C-309	-	-	1	0	ND	ND	-	-
Reagent Spk A	NA	LCS	050207C-310	-	-	1	5872	55.3	57.6	50	111
Reagent Spk B	NA	LCS	050207C-311	-	-	1	48202	496	516	500	99
L11423-11 Spk C	SG-A-011	LF	050207C-312	-	100	10000	50096	^	-	5000000	-
L11423-11 Spk C	SG-A-011	LF	050207C-313	-	100	10	13432588	*	-	5000000	-
Methanol Wash	-	C	050207C-314	-	-	-	4974	-	-	-	-
XC050107-1	-	CS	050207C-315	25	-	-	2931	-	-	-	-
XC050107-2	-	CS	050207C-316	50	-	-	5530	-	-	-	-
L11423-3	SG-A-003	S	050207C-317	-	100	10	476	ND	ND	-	-
L11423-7	SG-A-007	S	050207C-318	-	100	10	403	ND	ND	-	-
L11423-11	SG-A-011	S	050207C-319	-	100	10	23763	2420	2520	-	-
L11423-11 Rep	SG-A-011	S	050207C-320	-	100	10	19778	2000	2080	-	-
L11423-15	SG-A-015	S	050207C-321	-	100	10	18524	1870	1950	-	-
L11423-19	SG-A-019	S	050207C-322	-	100	10	23106	2350	2450	-	-
XC050107-3	-	CS	050207C-323	100	-	-	10339	-	-	-	-
XC050107-4	-	CS	050207C-324	250	-	-	25546	-	-	-	-
XC050107-5	-	CS	050207C-325	500	-	-	48479	-	-	-	-
XC050107-6	-	CS	050207C-326	1000	-	-	95957	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 557.930
Slope = 96.0375
Coef. Of Det. = 0.997856

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.
^Sample requires a lower spiking level. For re-extraction and analysis see data set 050907C.

Spreadsheet prepared by: MLC / 5/8/07

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050207C
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	^{13}C-PFOA	Matrix:	Buffer	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	415 -> 370	Sample Volume Extracted:	4.0 mL	Extraction Date:	05/02/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/05/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	13C-PFOA Found (ng/L)	Amount 13C-PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050207C-301	0	-	-	0	-	-	-
XC050107-1	-	CS	050207C-302	25	-	-	2806	-	-	-
XC050107-2	-	CS	050207C-303	50	-	-	5010	-	-	-
XC050107-3	-	CS	050207C-304	100	-	-	9847	-	-	-
XC050107-4	-	CS	050207C-305	250	-	-	23283	-	-	-
XC050107-5	-	CS	050207C-306	500	-	-	49544	-	-	-
XC050107-6	-	CS	050207C-307	1000	-	-	94237	-	-	-
Methanol Wash	-	C	050207C-308	-	-	-	0	-	-	-
Reagent Control	NA	C	050207C-309	-	-	1	0	ND	-	-
Reagent Spk A	NA	LCS	050207C-310	-	-	1	4802	46.6	50	93
Reagent Spk B	NA	LCS	050207C-311	-	-	1	49052	512	500	102
L11423-11 Spk C	SG-A-011	LF	050207C-312	-	100	10000	76	*	5000	-
L11423-11 Spk C	SG-A-011	LF	050207C-313	-	100	10	33719	3500	5000	70
Methanol Wash	-	C	050207C-314	-	-	-	0		-	-
XC050107-1	-	CS	050207C-315	25	-	-	2634	-	-	-
XC050107-2	-	CS	050207C-316	50	-	-	5391	-	-	-
L11423-3	SG-A-003	S	050207C-317	-	100	10	42958	4480	5000	90
L11423-7	SG-A-007	S	050207C-318	-	100	10	42142	4390	5000	88
L11423-11	SG-A-011	S	050207C-319	-	100	10	48947	5100	5000	102
L11423-11 Rep	SG-A-011	S	050207C-320	-	100	10	41126	4280	5000	86
L11423-15	SG-A-015	S	050207C-321	-	100	10	44336	4620	5000	92
L11423-19	SG-A-019	S	050207C-322	-	100	10	48227	5030	5000	101
XC050107-3	-	CS	050207C-323	100	-	-	9745	-	-	-
XC050107-4	-	CS	050207C-324	250	-	-	24850	-	-	-
XC050107-5	-	CS	050207C-325	500	-	-	48081	-	-	-
XC050107-6	-	CS	050207C-326	1000	-	-	95374	-	-	-

13C-PFOA Found (ng/L) = (peak area - intercept) / slope x DF

Recovery (%) = $\frac{\text{13C-PFOA found (ng/L)}}{\text{amount 13C-PFOA added (ng/L)}} \times 100$

Standard Curve : Linear (1/x weighted)
Intercept = 370.526
Slope = 95.1573
Coef. Of Det. = 0.999558

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.

Spreadsheet prepared by: MLC / 5/8/07

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

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423 Matrix: $Na_2B_4O_7$ Impinger Sample

Sponsor Study/Protocol No: NA

The samples listed below were removed from refrigerator No. 34

Time ~~8:10 AM~~ 8:25 AM Date 5/02/2007 Initials EDD

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-A-011	L11423-11* Spk C	160 mL	40.0	20000.0
SG-A-003	L11423-3*	100 mL	40.0	-
SG-A-007	L11423-7*	100 mL	40.0	-
SG-A-011	L11423-11*	106 mL	40.0	-
SG-A-011	L11423-11 Rep*	106 mL	40.0	-
SG-A-015	L11423-15*	100 mL	40.0	-
SG-A-019	L11423-19*	100 mL	40.0	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-
-	-		-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
L11423-11* Spk C	SS20794 (100000 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-

All samples were measured:

Time 10:25 AM Date 5/02/2007 Initials EDD

After measuring samples were returned to refrigerator No. 34

Time 3:30 PM Date 5/02/2007 Initials EDD

Comments: *Sample is pre-diluted ten times by taking 4 mL of sample and bringing the volume up to 40 mL with HPLC water

Analysis Summary:

Data Set: 050207C Initials/Date: MLC / 5/4/07

Data Set: 050907C Initials/Date: MLC / 5/11/07

Data Set: - Initials/Date: - / -

Set extraction/analysis data verified by: CEE Date: 05/23/07

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

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007C$$ PROTOCOL NUMBER: NA

MATRIX: Na2B4O7 Impinger Sample ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	Reagent Control			-		-	-	Methanol	46303
na	Reagent Spk A			-		-	-	C18 SPE	0053370818
na	Reagent Spk B			-		-	-	HPLC Water	46335
SG-A-011	L11423-11* Spk C			50-1/10, 0-1/1.0		-	-	-	-
SG-A-003	L11423-3*			-		-	-	-	-
SG-A-007	L11423-7*			-		-	-	-	-
SG-A-011	L11423-11*			-		-	↓	-	-
SG-A-011	L11423-11 Rep*			-		-	-	-	-
SG-A-015	L11423-15*			-		-	-	-	-
SG-A-019	L11423-19*	↓	↓	-	↓	-	-	*Initials/Date*	EDD 5/2/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C441404
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date*	-	EDD 5/2/07	EDD 5/2/07	MLC 5/2/07	MLC ① 5/4/07	-	MLC 5/11/07	*Initials/Date*	MLC 5/4/07

① DF = 1000 MLC 5/2/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

*Sample was diluted ten times prior to extraction.
**Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:
② SAMPLES WERE PLACED ON AUTOSAMPLER ON 5/4/07, BUT DID NOT INJECT UNTIL 5/5/07. MLC 5/8/07

Final extracts stored in refrigerator 22 Initials: EDD Date: 5/2/2007

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

Exygen STUDY NO. L11423

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Protocol No.: None
Method No.: $$

Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA

Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC050107-0	Methanol	46303
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC050107-1	C18 SPE	0053370818
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC050107-2	Water	46335
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC050107-3	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC050107-4	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC050107-5	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC050107-6	-	-
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/01/07
-	-	-	-	-	-	-	-		

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) freezer # 34 Time: 9:10 AM — Initials/Date: EDD 1 5/01/07
40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD 1 5/01/07
After measuring, samples returned to (refrigerator) freezer # 34 Time: 11:10 AM — Initials/Date: EDD 1 5/01/07
Samples fortified: — Initials/Date: AMB 1 05/01/07
SPE clean-up: — Initials/Date: EDD 1 5/01/07
Final volume adjusted to 5 mL: — Initials/Date: EDD 1 5/01/07
Extracts placed in refrigerator # 32 — Initials/Date: EDD 1 5/01/07

Comments:

STANDARD EXPIRATION DATE: 5/15/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/8/07

Masslynx - Sample List

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050207C Buffer.SPL
Printed: Fri May 04 14:56:57 2007 MLC 5/4/07

Exygen STUDY NO. L11423

Page Position: (1, 1)

L @ MLC 5/4/07

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/~~mL~~)	Conc B	Conc C	Test ID	DF
1	94	050207C-301	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---	0	1
2	95	050207C-302	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
3	96	050207C-303	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
4	97	050207C-304	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
5	98	050207C-305	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
6	99	050207C-306	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
7	100	050207C-307	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1
8	92	050207C-308	---	---	Methanol Wash	---	Blank	---	---	---	0	1
9	44	050207C-309	---	---	Reagent Control	---	Blank	---	---	---	0	1
10	45	050207C-310	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---	0	1
11	46	050207C-311	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500	---	0	1
12	47	050207C-312	---	---	L11423-11 Spk C, 5,000,000 ng/L, DF=10,000	---	QC	5000000	5000	---	0	10000
13	48	050207C-313	---	---	L11423-11 Spk C, 5,000,000 ng/L, DF=10	---	QC	5000000	5000	---	0	10
14	92	050207C-314	---	---	Methanol Wash	---	Blank	---	---	---	0	1
15	95	050207C-315	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
16	96	050207C-316	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
17	49	050207C-317	---	---	L11423-3, DF=10	---	Analyte	---	5000	---	0	10
18	50	050207C-318	---	---	L11423-7, DF=10	---	Analyte	---	5000	---	0	10
19	51	050207C-319	---	---	L11423-11, DF=10	---	Analyte	---	5000	---	0	10
20	52	050207C-320	---	---	L11423-11 Rep, DF=10	---	Analyte	---	5000	---	0	10
21	53	050207C-321	---	---	L11423-15, DF=10	---	Analyte	---	5000	---	0	10
22	54	050207C-322	---	---	L11423-19, DF=10	---	Analyte	---	5000	---	0	10
23	97	050207C-323	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
24	98	050207C-324	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
25	99	050207C-325	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
26	100	050207C-326	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050207C Buffer.SPL
Printed: Fri May 04 14:56:57 2007 MLC 5/4/07

Exygen STUDY NO. L11423

	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
20	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
21	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
22	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
23	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
24	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
25	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
26	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA | Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/4/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/8/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Fri May 04 14:57:54 2007 MLC 5/4/07

Solvent Delay (mins) : 0.00

Function : 1 **MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method Report

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Friday, May 04, 2007 14:57:57

Printed: Friday, May 04, 2007 14:58:00 MLC 5/4/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

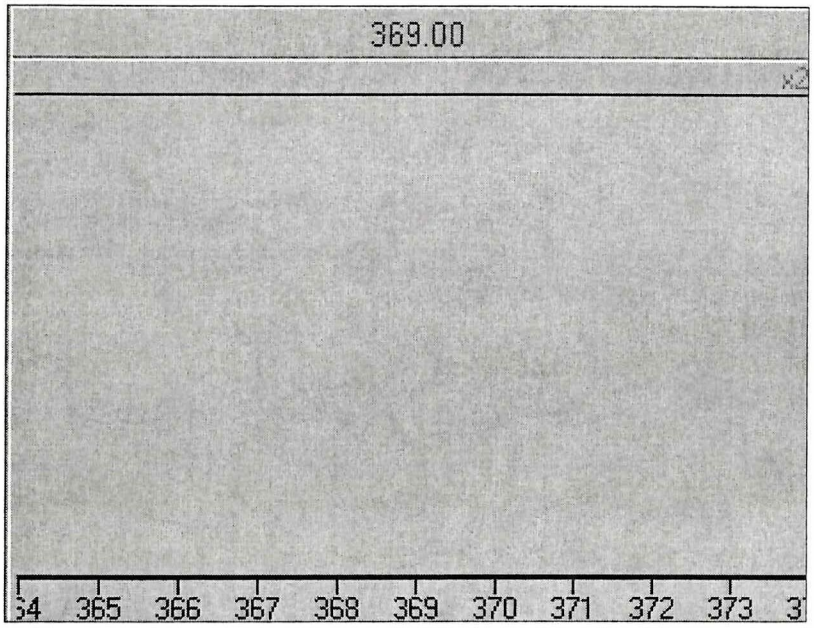
HP1100 Autosampler Initial Conditions

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	77

Tuning Method Report **Page 1**

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Fri May 04 14:58:11 2007 MLC 5/4/07



Dau 413.00

SOURCE (ESP-)	**Set**	**Rdbk**	**Analyser**	**Set**	**Rdbk**
Capillary	3.00	-2.92	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	102	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	89.7
Gas Cell	2.3e-3	Desolvation	681.0

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050207C
Extraction Date: 05/02/07
Analyzed on: 05/05/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2742
XC050107-2	50	5366
XC050107-3	100	10168
XC050107-4	250	23900
XC050107-5	500	52281
XC050107-6	1000	93200

Coefficient of Determination: 0.996427735

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.998212269
R Square	0.996427735
Adjusted R Square	0.995534668
Standard Error	2364.68234
Observations	6

MLC
5/8/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	6238895207	6238895207	1115.737616	4.79111E-06
Residual	4	22366890.27	5591722.57		
Total	5	6261262097			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	1159.263646	1320.944306	0.87760221	0.429710513	-2508.265707	4826.792999	-2508.265707	4826.792999
X Variable 1	93.87086656	2.810281274	33.4026588	4.79111E-06	86.06827487	101.6734582	86.06827487	101.6734582

Initial Calibration Curve for ^{13}C-PFOA

Exygen Study No.: L11423
Set No: 050207C
Extraction Date: 05/02/07
Analyzed on: 05/05/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2806
XC050107-2	50	5010
XC050107-3	100	9847
XC050107-4	250	23283
XC050107-5	500	49544
XC050107-6	1000	94237

Coefficient of Determination: 0.999278208

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999639039
R Square	0.999278208
Adjusted R Square	0.99909776
Standard Error	1067.902423
Observations	6

MLC
5/8/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	6315349588	6315349588	5537.761562	1.95416E-07
Residual	4	4561662.338	1140415.58		
Total	5	6319911251			

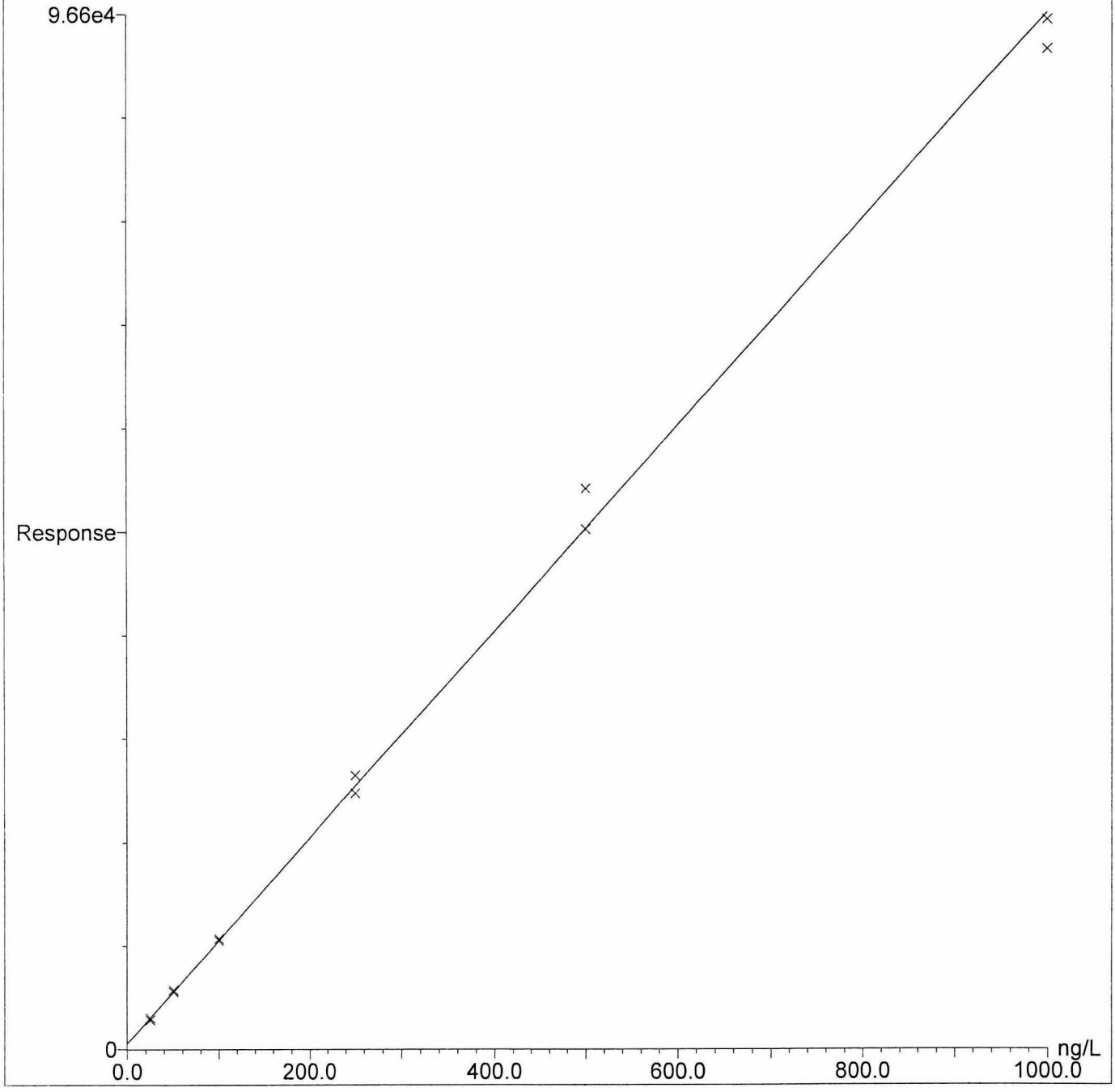
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	486.9588054	596.5450839	0.81629841	0.460151856	-1169.315873	2143.233484	-1169.315873	2143.233484
X Variable 1	94.44428424	1.26913714	74.4161378	1.95416E-07	90.92059464	97.96797384	90.92059464	97.96797384

Quantify Calibration Report
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050207C Buffer
Last modified: Tue May 08 13:36:14 2007
Printed: Tue May 08 13:55:56 2007

MLC 5/8/07

Compound 1 name: PFOA
Coefficient of Determination: 0.997856
Calibration curve: 96.0375 * x + 557.930
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

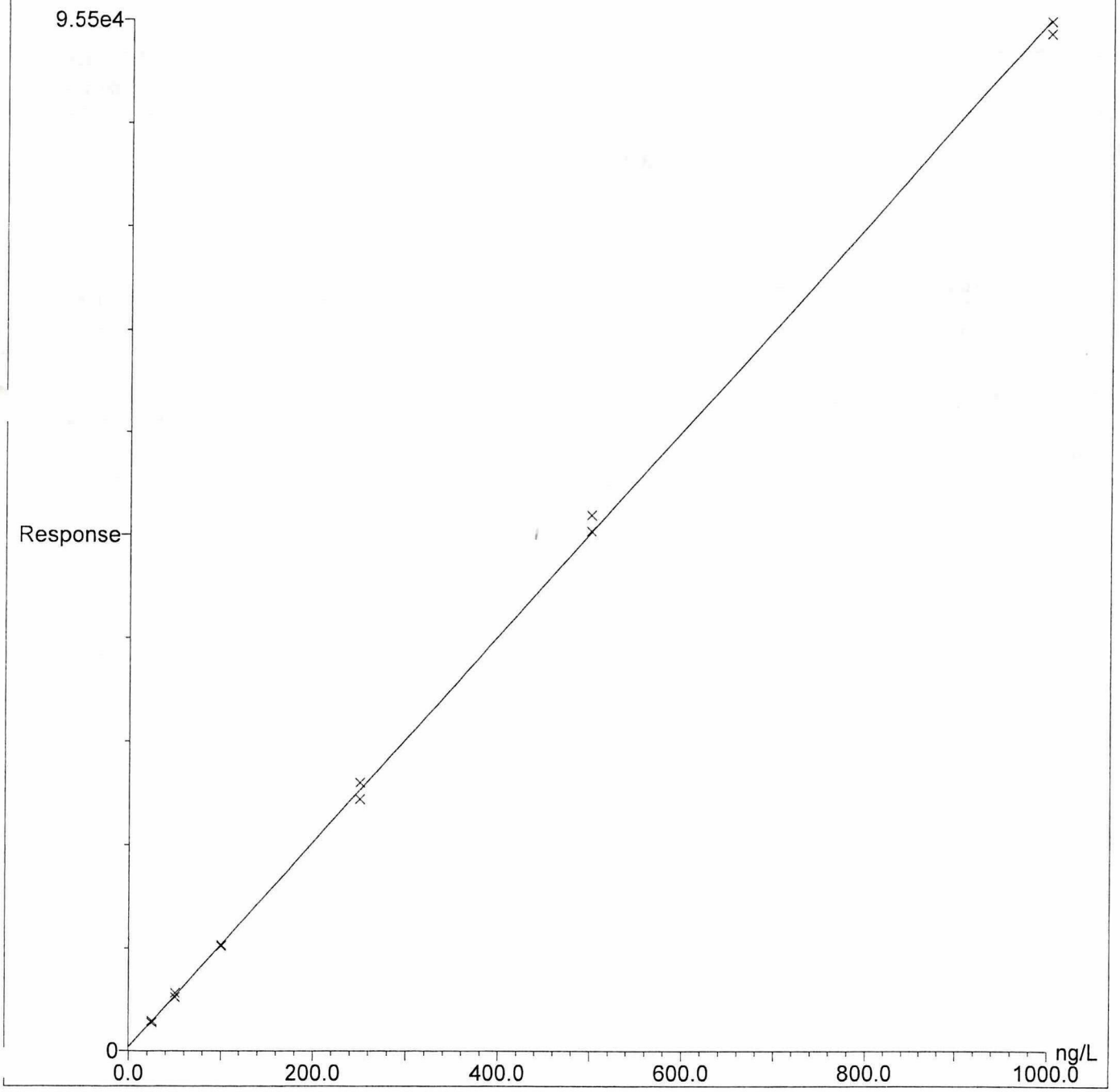


Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050207C Buffer
Last modified: Tue May 08 13:36:14 2007
Printed: Tue May 08 13:55:56 2007

MLC 5/8/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.999558
Calibration curve: 95.1573 * x + 370.526
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

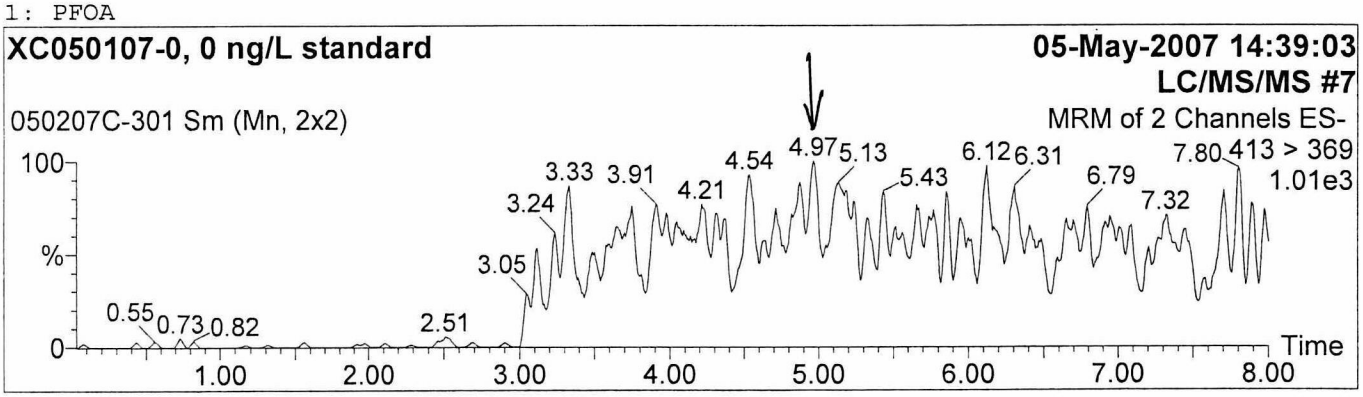
Initials MLC
Date 5/8/07

Printed: Tue May 08 13:55:58 2007

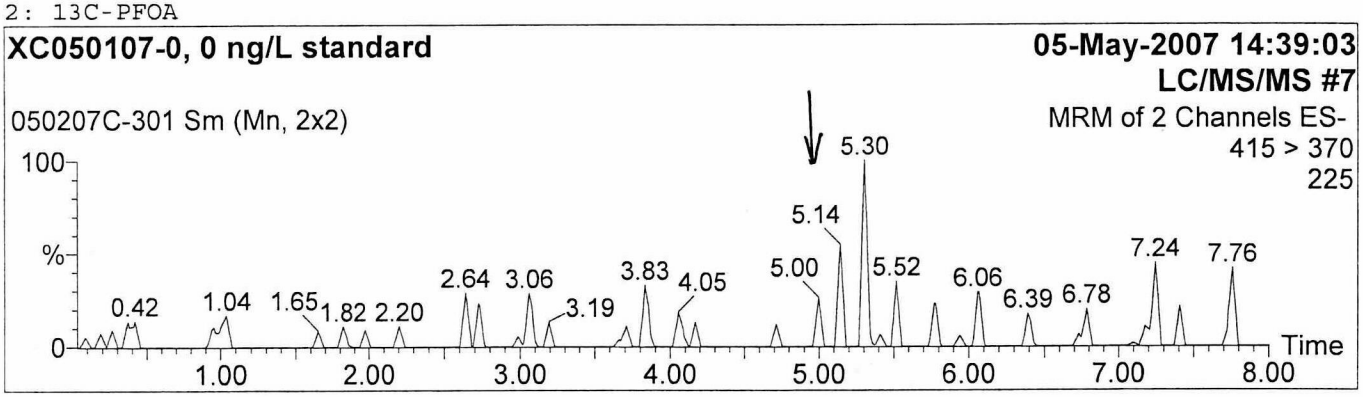
Run# 050207C-301 To 050207C-326

Name: 050207C-301
Text:

1: PFOA



2: 13C-PFOA



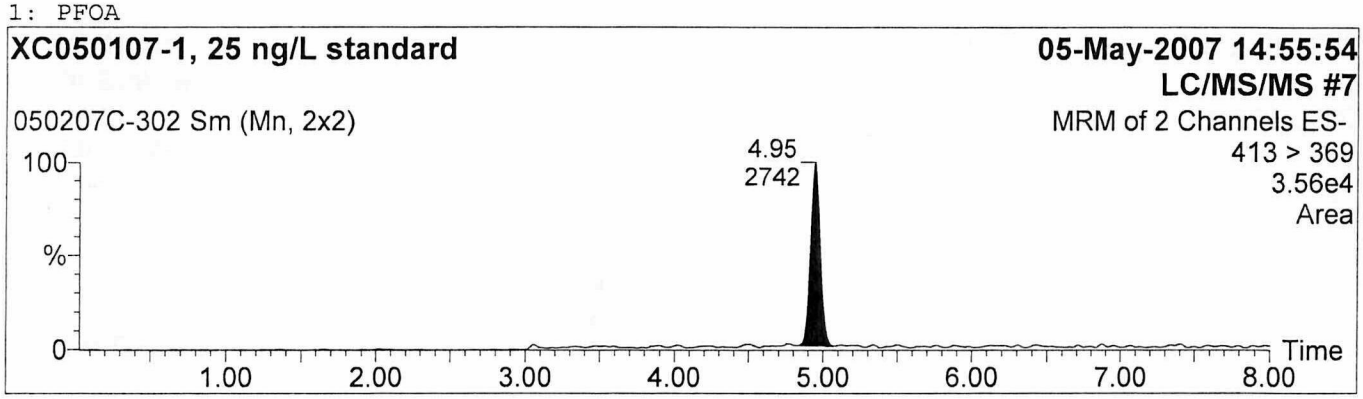
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

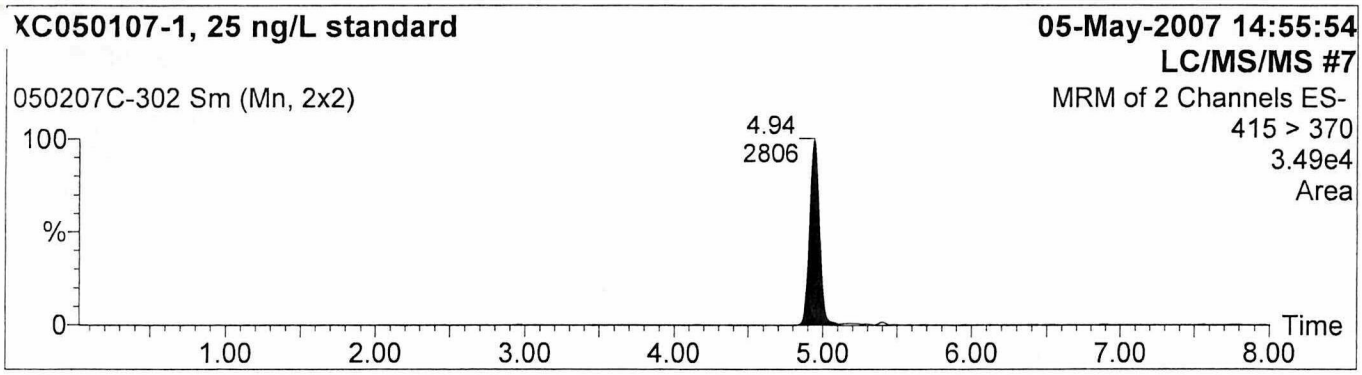
Printed: Tue May 08 13:55:58 2007

Name: 050207C-302
Text:

1: PFOA



2: 13C-PFOA



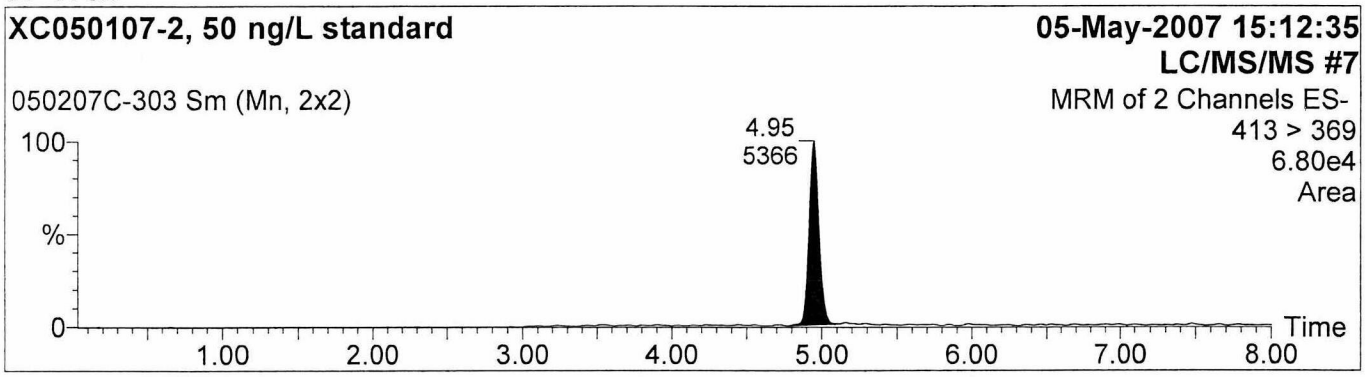
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

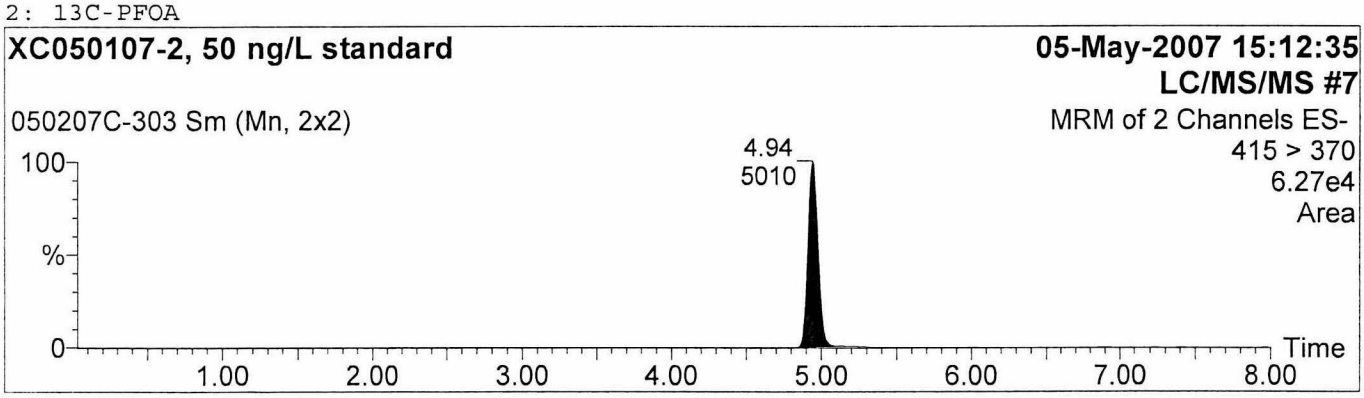
Printed: Tue May 08 13:55:58 2007

Name: 050207C-303
Text:

1: PFOA



2: 13C-PFOA



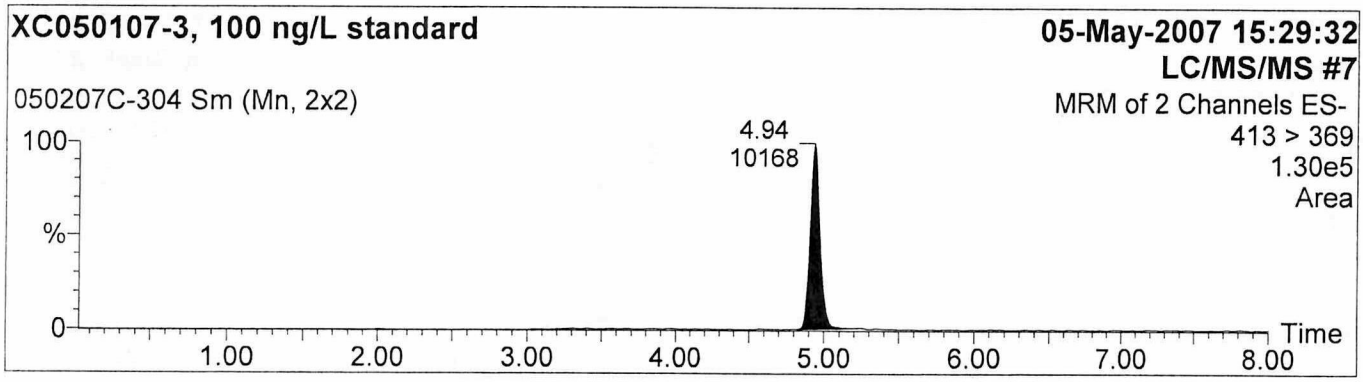
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

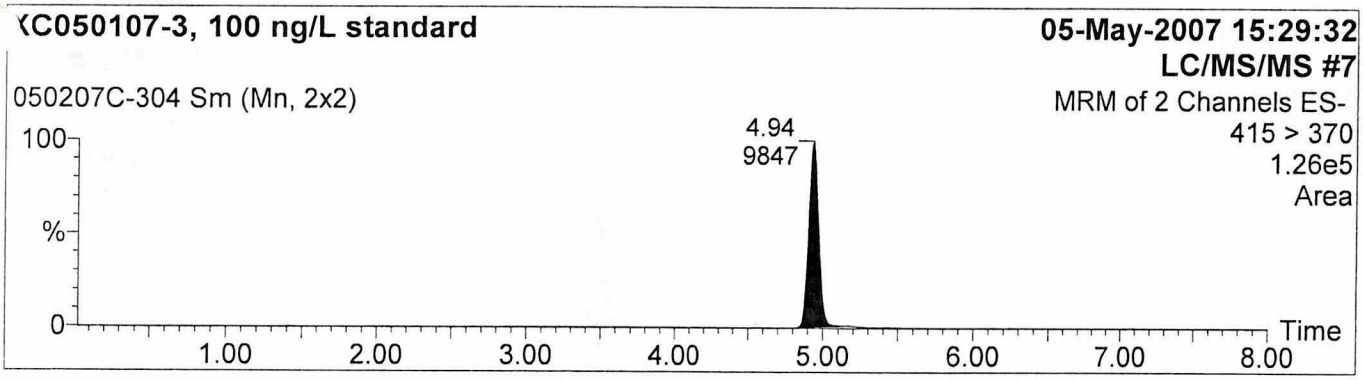
Printed: Tue May 08 13:55:58 2007

Name: 050207C-304
Text:

1: PFOA



2: 13C-PFOA

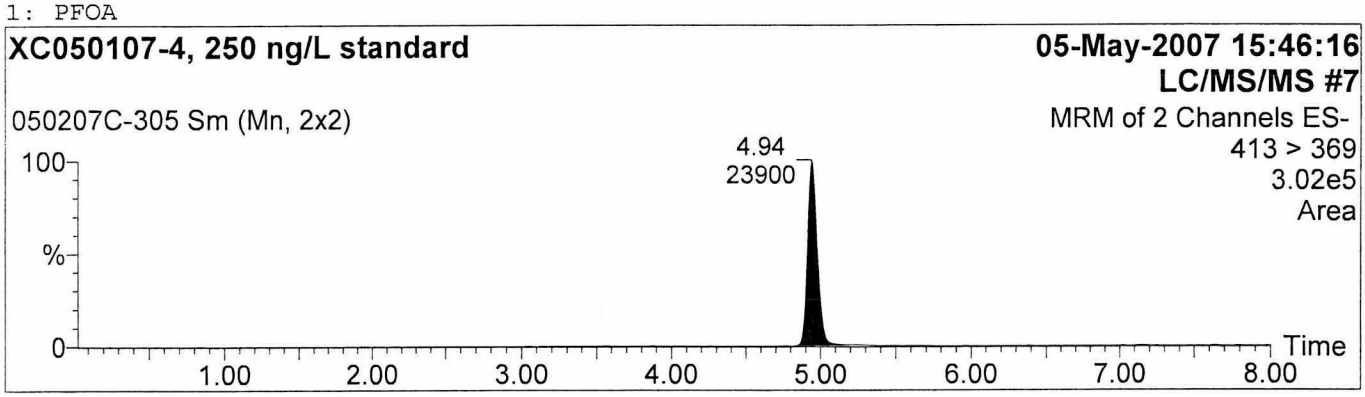


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

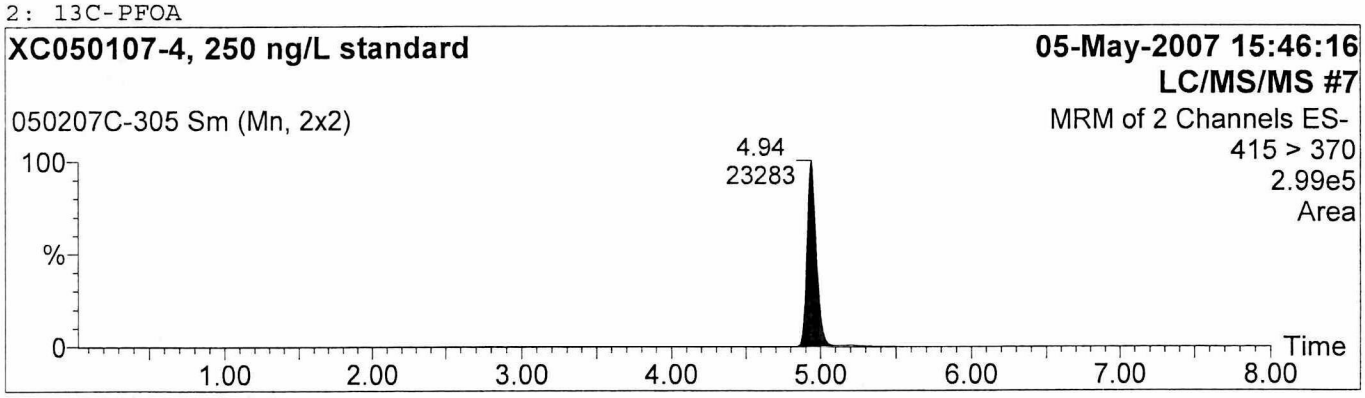
Printed: Tue May 08 13:55:58 2007

Name: 050207C-305
Text:

1: PFOA



2: 13C-PFOA



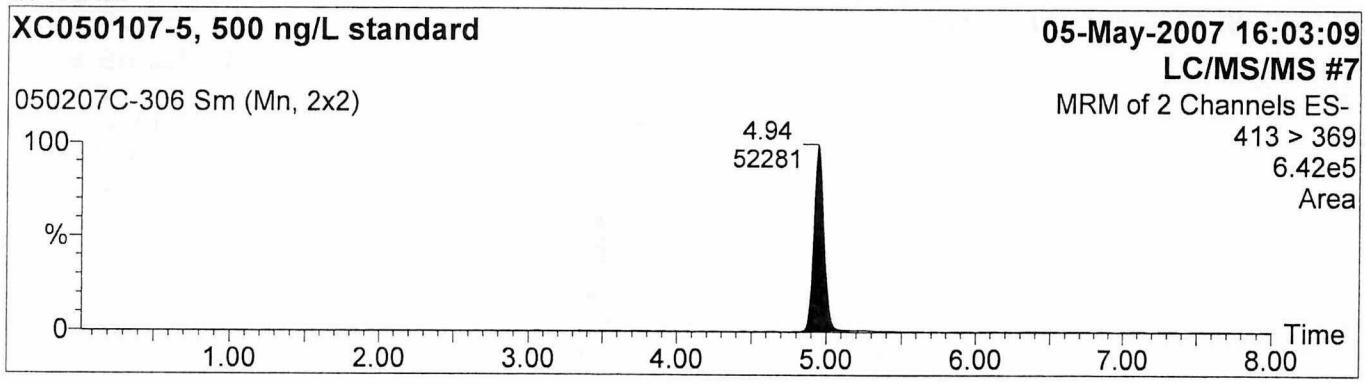
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

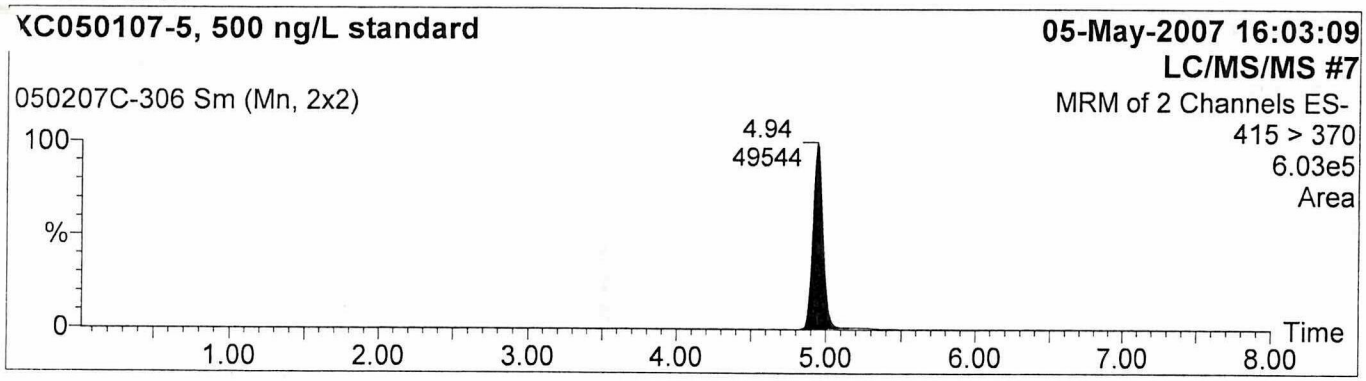
Printed: Tue May 08 13:55:58 2007

Name: 050207C-306
Text:

1: PFOA



2: 13C-PFOA



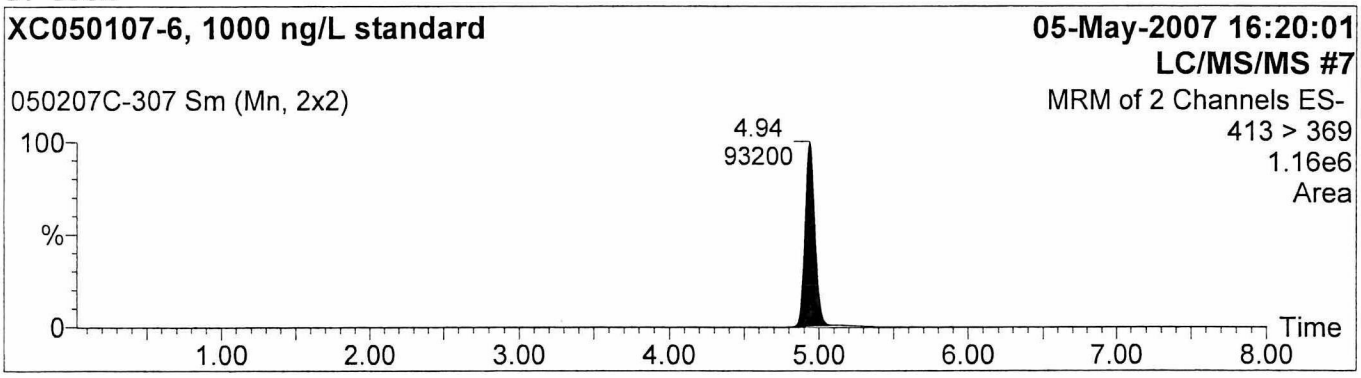
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

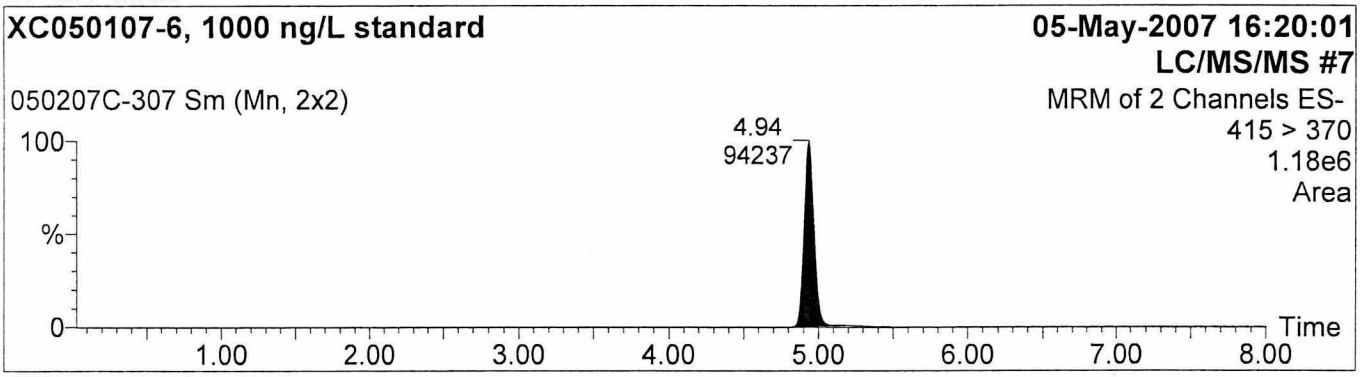
Printed: Tue May 08 13:55:58 2007

Name: 050207C-307
Text:

1: PFOA



2: 13C-PFOA



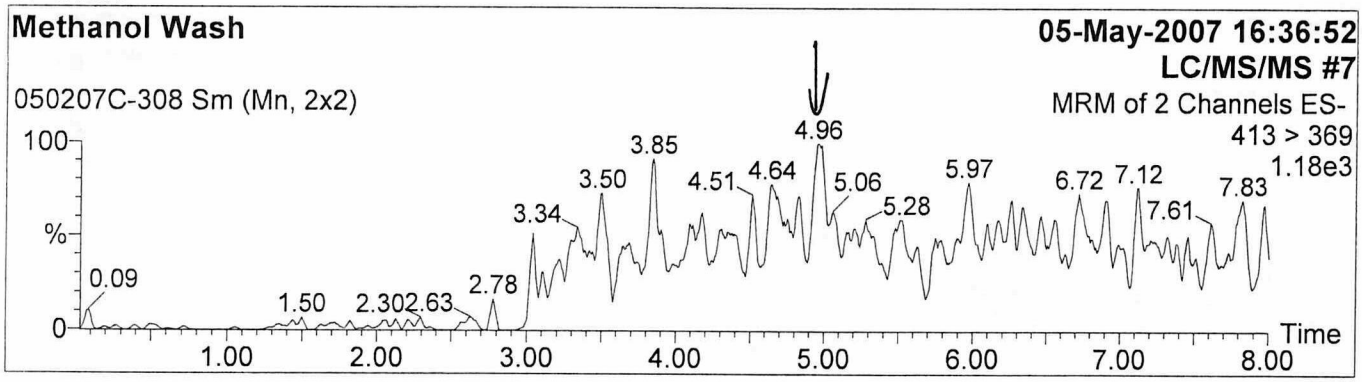
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

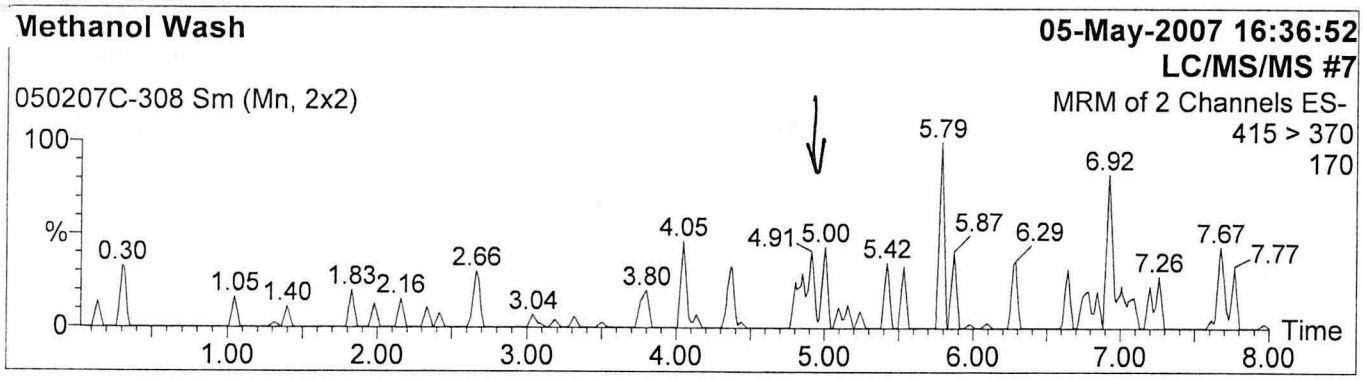
Printed: Tue May 08 13:55:58 2007

Name: 050207C-308
Text:

1: PFOA



2: 13C-PFOA



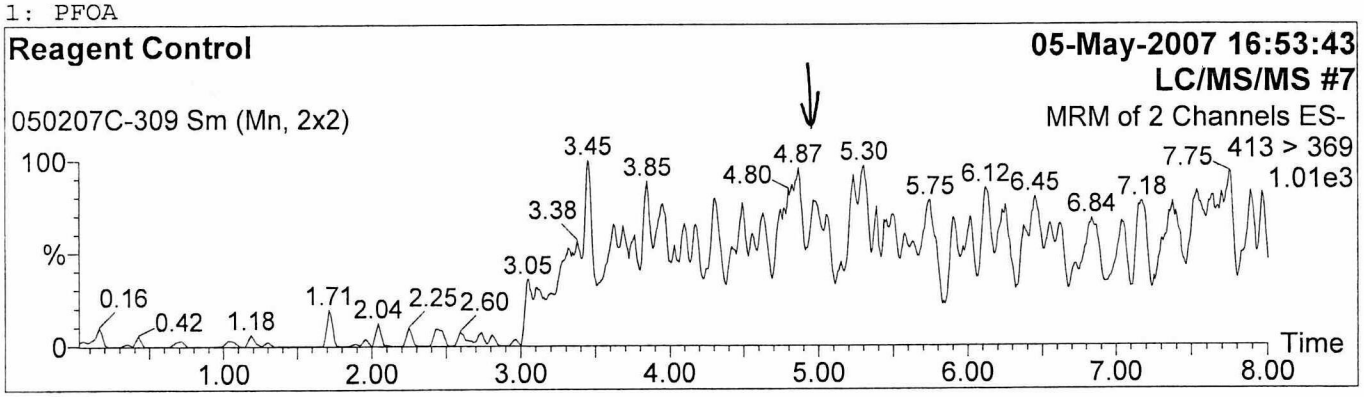
Quantify Sample Report
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

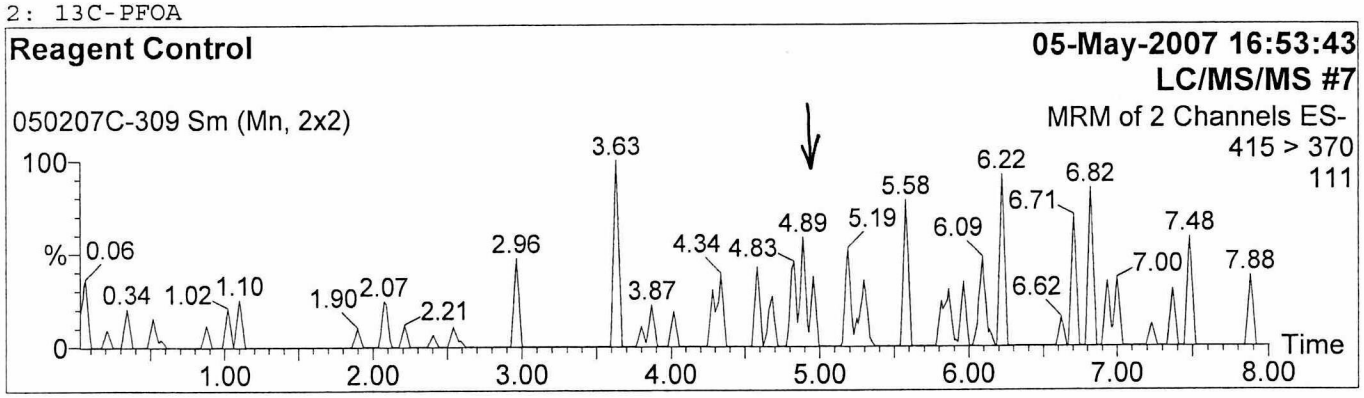
Printed: Tue May 08 13:55:58 2007

Name: 050207C-309
Text:

1: PFOA



2: 13C-PFOA

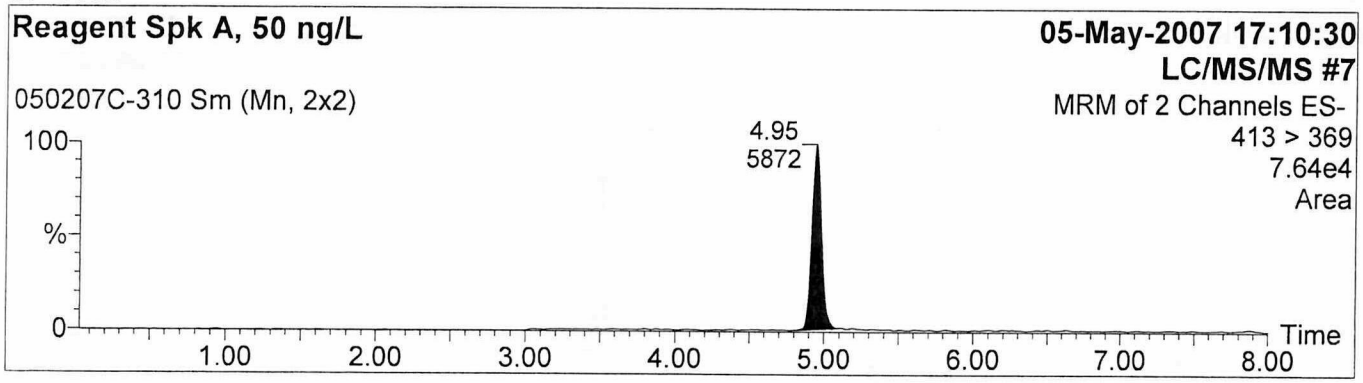


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

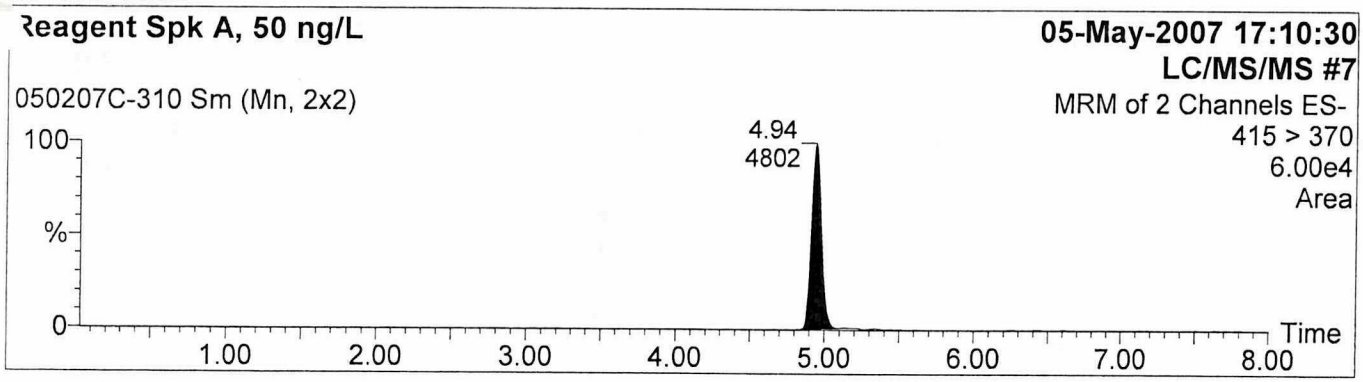
Printed: Tue May 08 13:55:58 2007

Name: 050207C-310
Text:

1: PFOA



2: 13C-PFOA



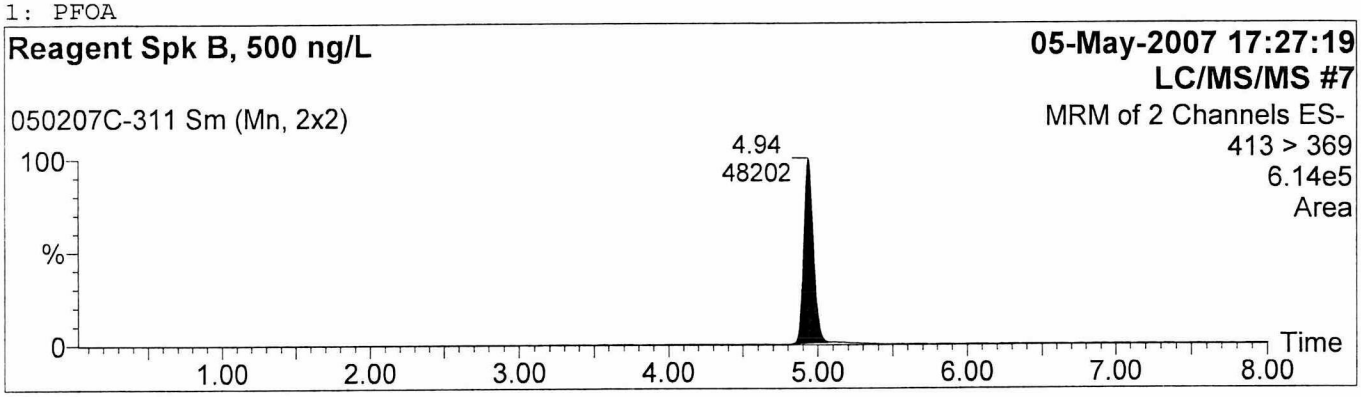
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

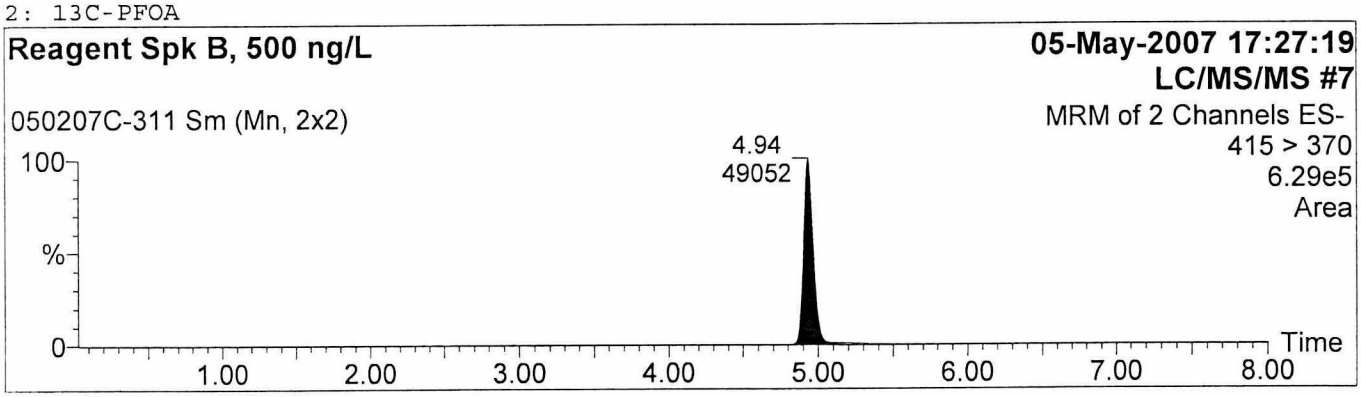
Printed: Tue May 08 13:55:58 2007

Name: 050207C-311
Text:

1: PFOA



2: 13C-PFOA



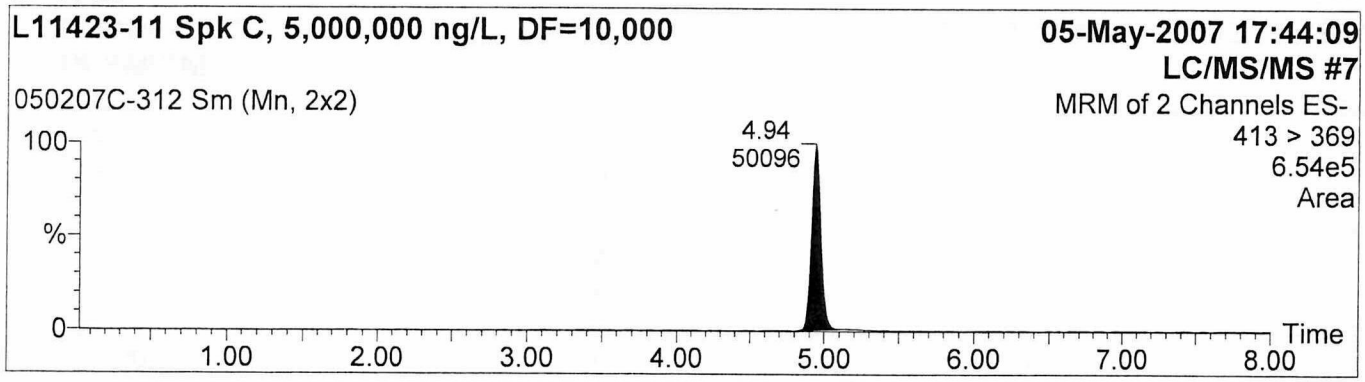
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

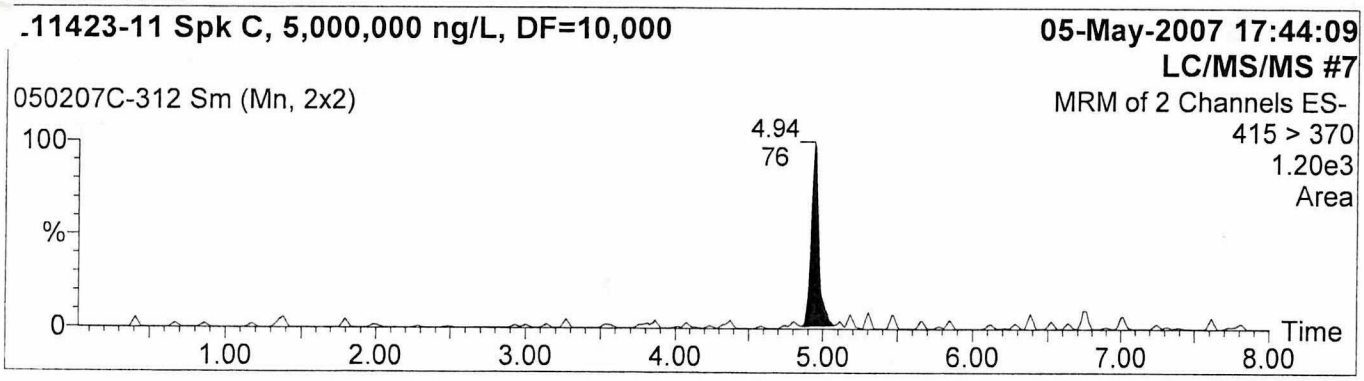
Printed: Tue May 08 13:55:58 2007

Name: 050207C-312
Text:

1: PFOA



2: 13C-PFOA



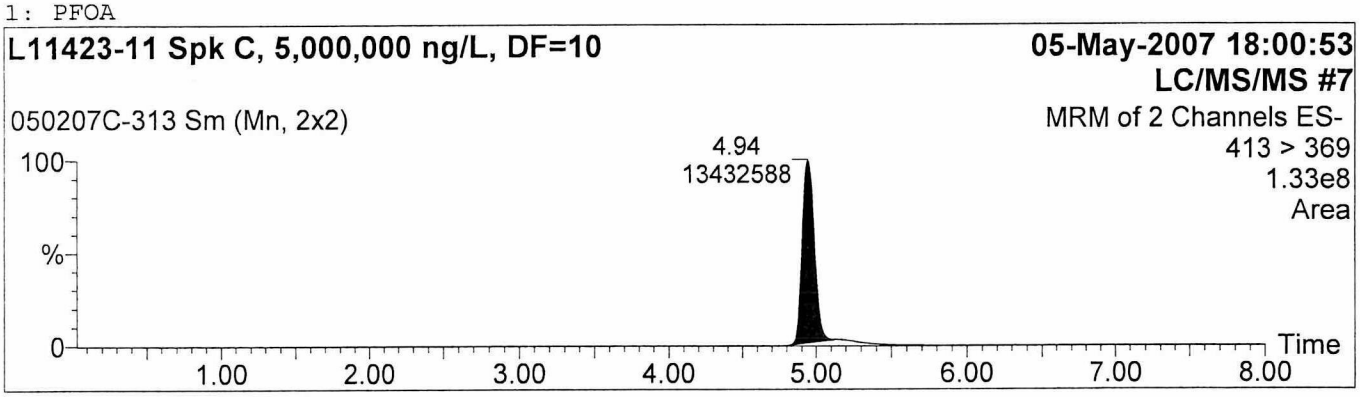
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

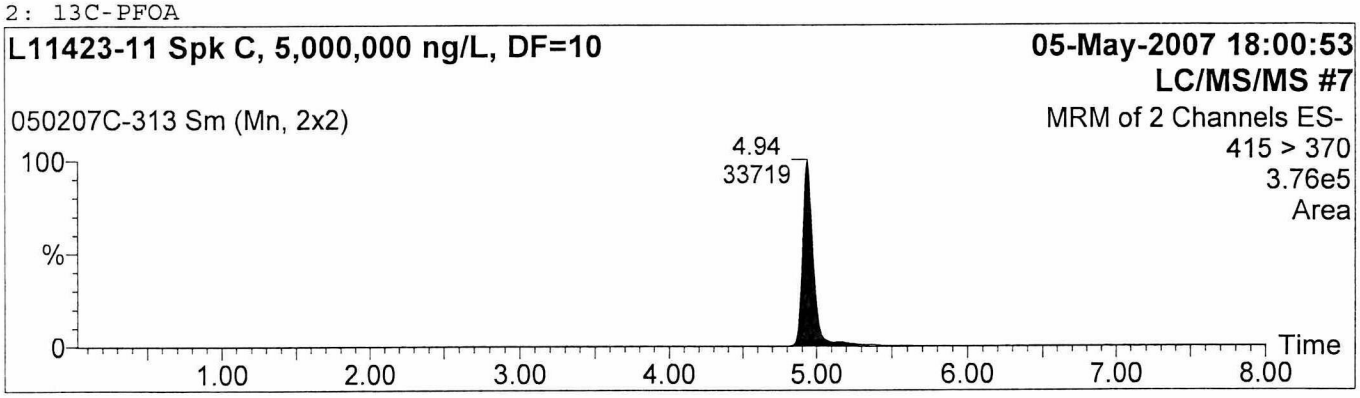
Printed: Tue May 08 13:55:58 2007

Name: 050207C-313
Text:

1: PFOA



2: 13C-PFOA



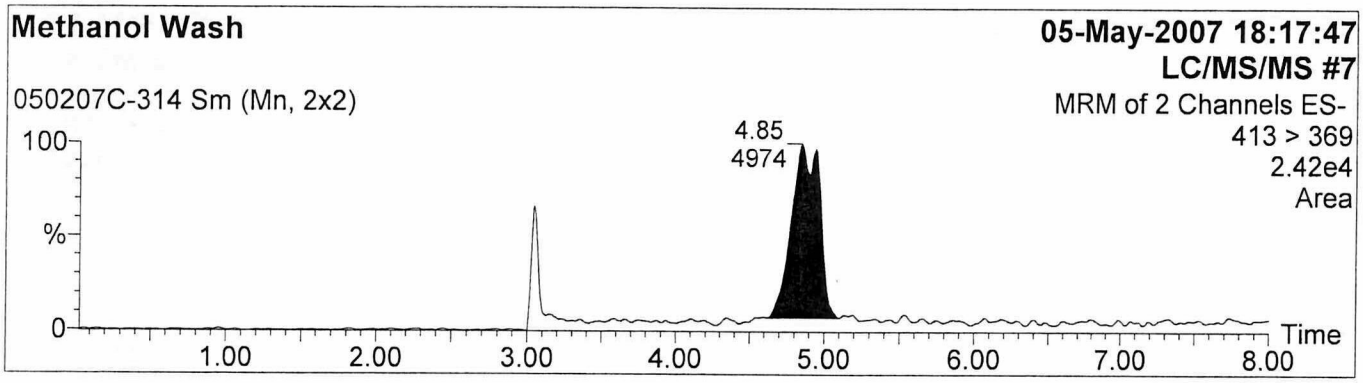
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

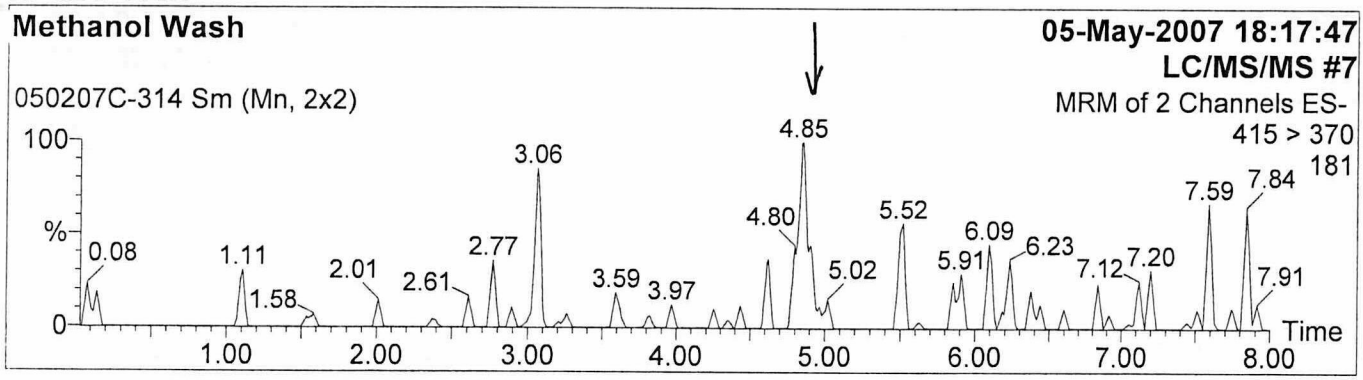
Printed: Tue May 08 13:55:58 2007

Name: 050207C-314
Text:

1: PFOA



2: 13C-PFOA

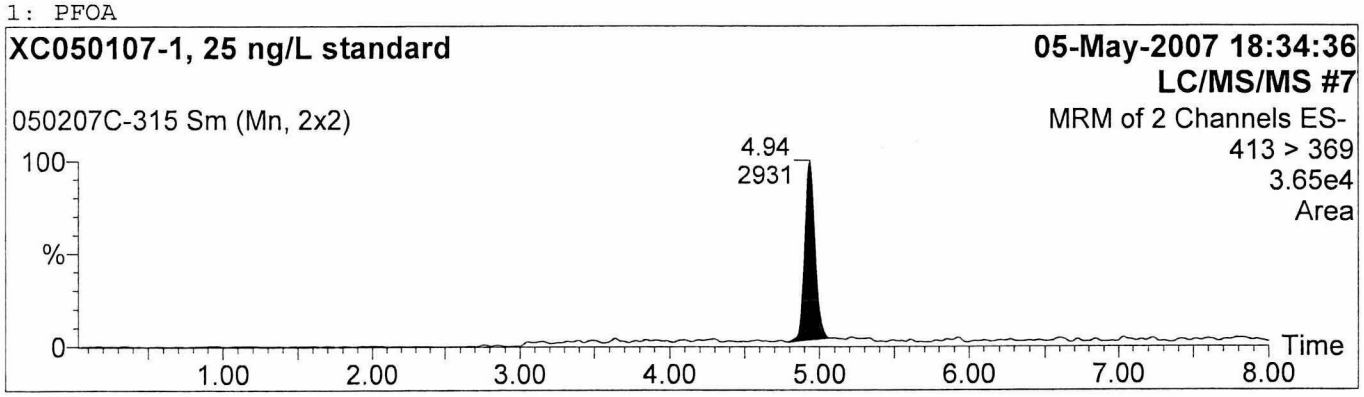


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

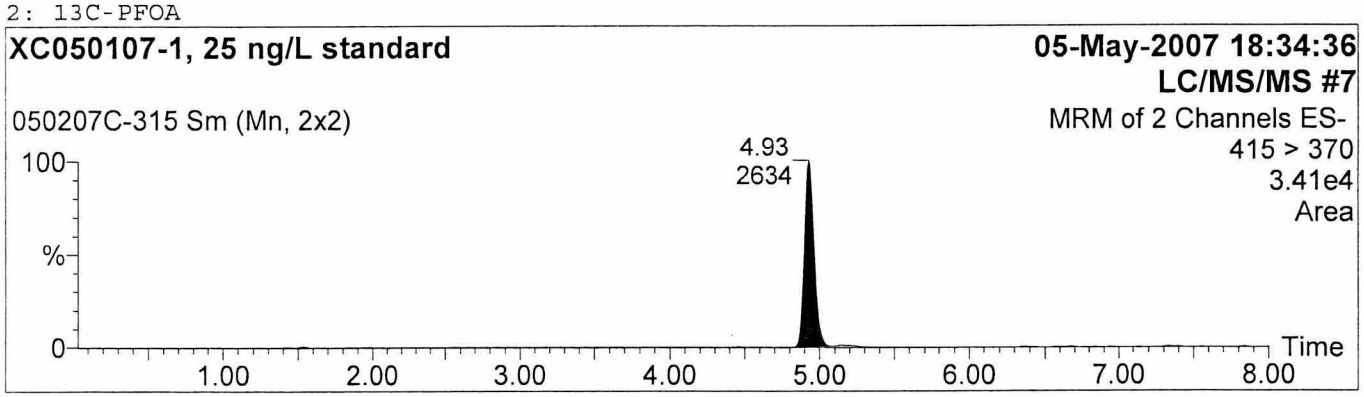
Printed: Tue May 08 13:55:58 2007

Name: 050207C-315
Text:

1: PFOA



2: 13C-PFOA



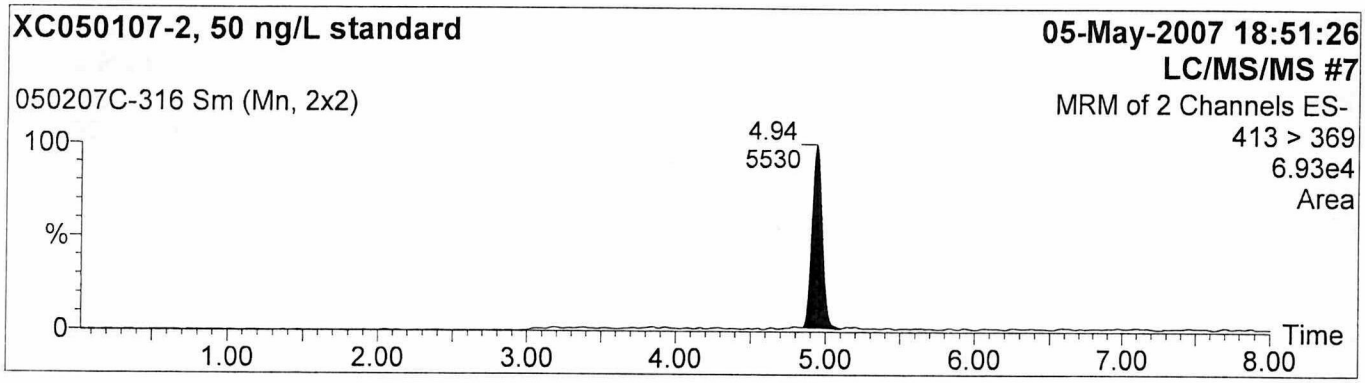
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

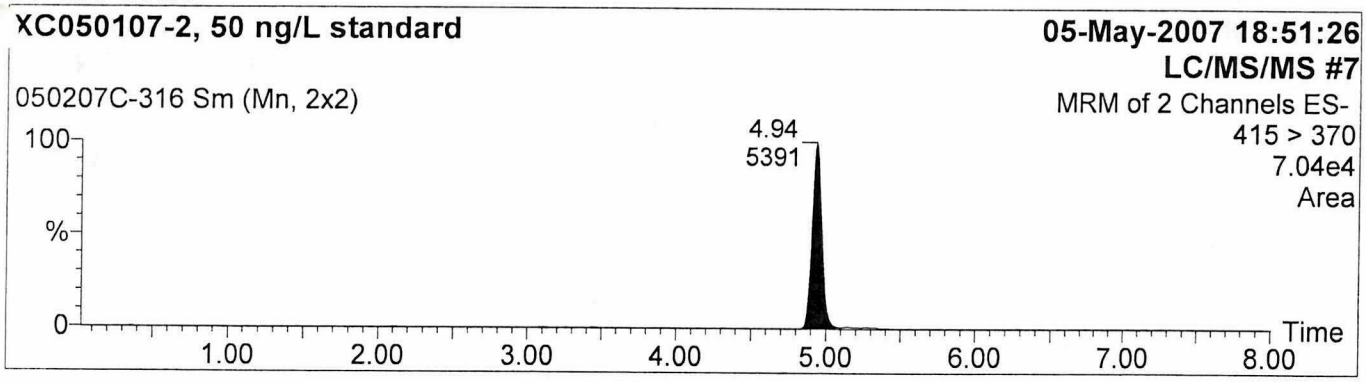
Printed: Tue May 08 13:55:58 2007

Name: 050207C-316
Text:

1: PFOA



2: 13C-PFOA



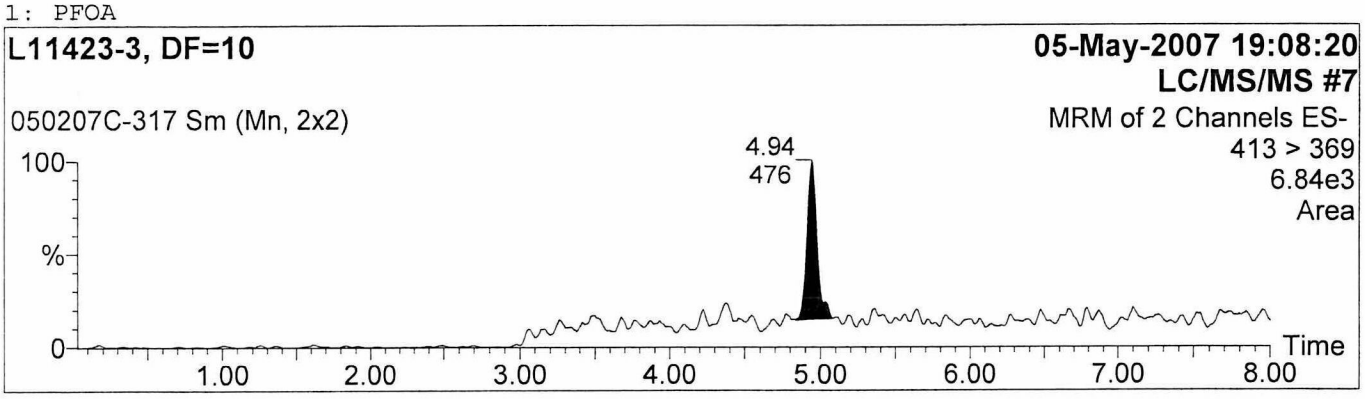
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

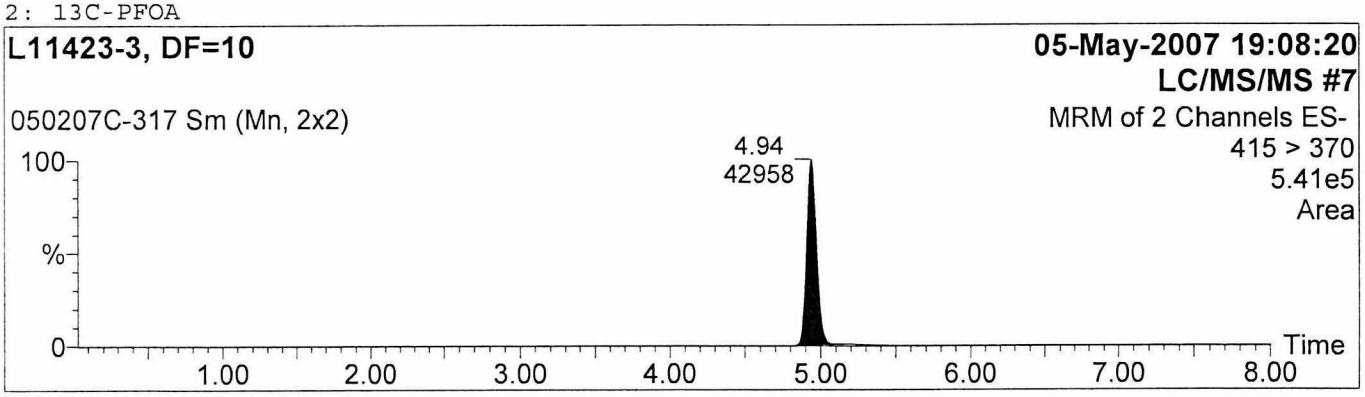
Printed: Tue May 08 13:55:58 2007

Name: 050207C-317
Text:

1: PFOA



2: 13C-PFOA



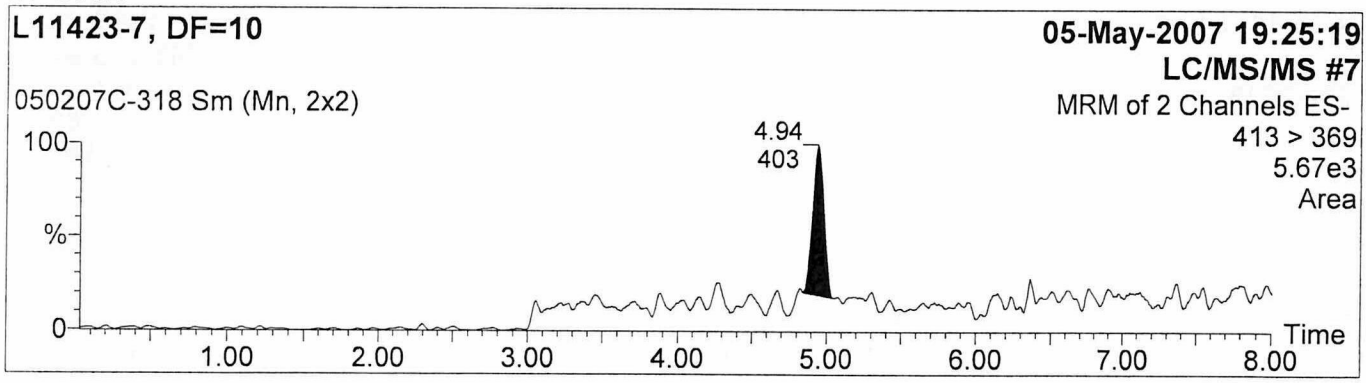
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

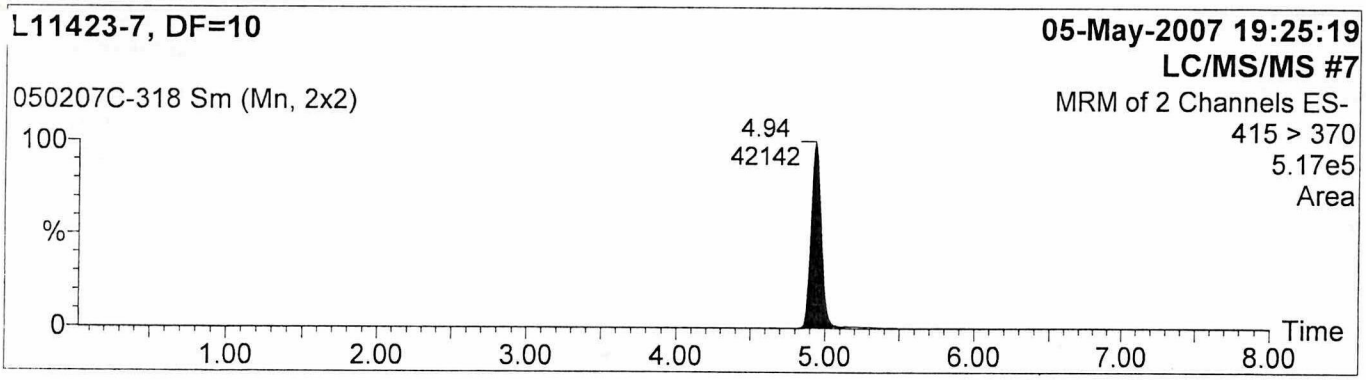
Printed: Tue May 08 13:55:58 2007

Name: 050207C-318
Text:

1: PFOA



2: 13C-PFOA



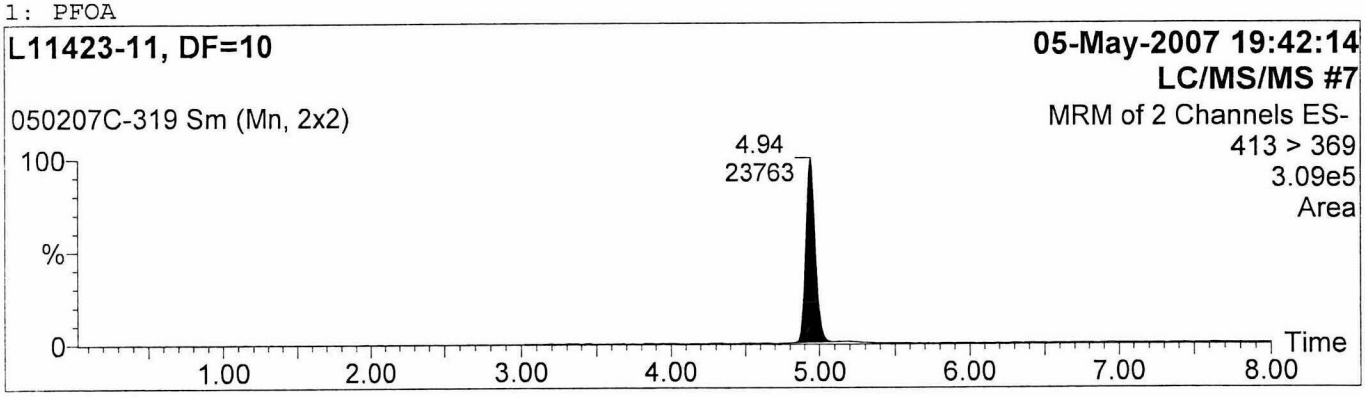
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

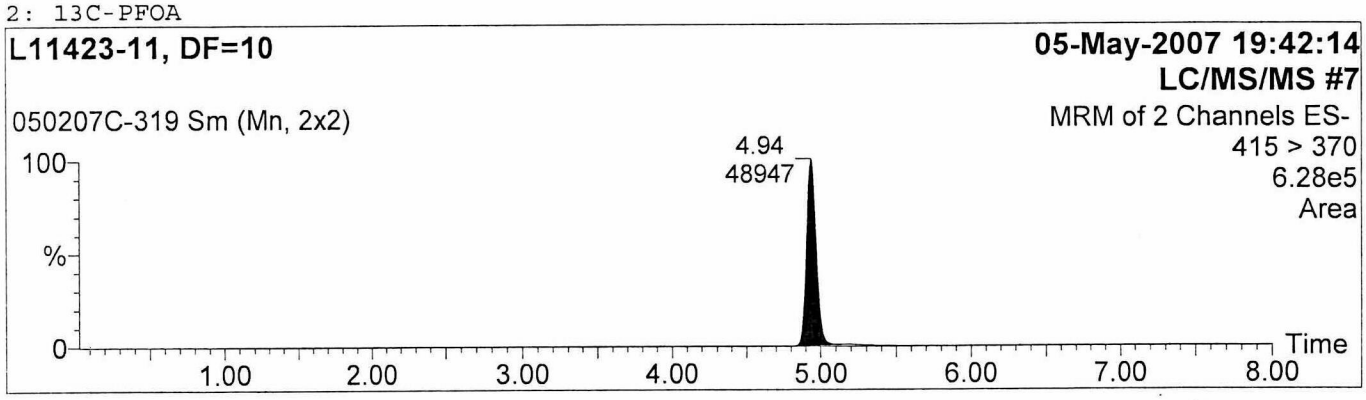
Printed: Tue May 08 13:55:58 2007

Name: 050207C-319
Text:

1: PFOA



2: 13C-PFOA



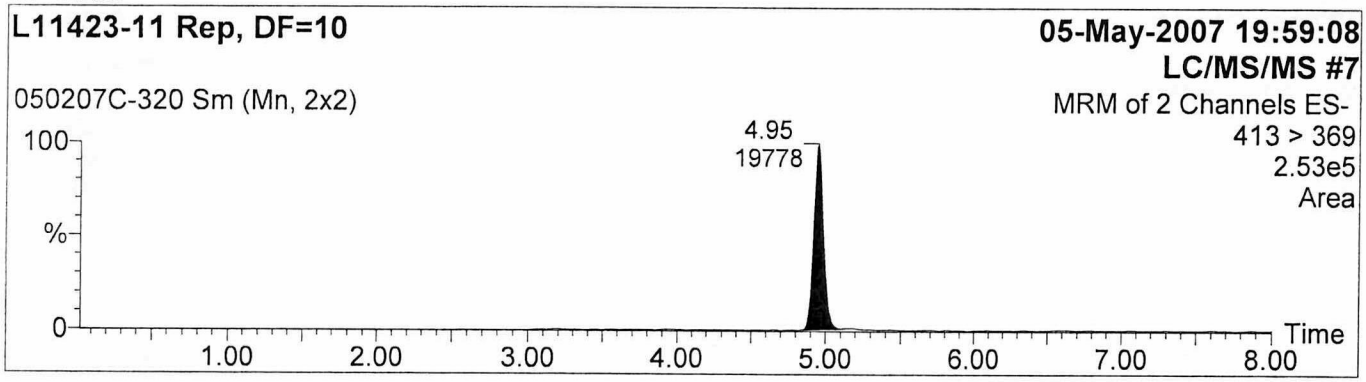
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

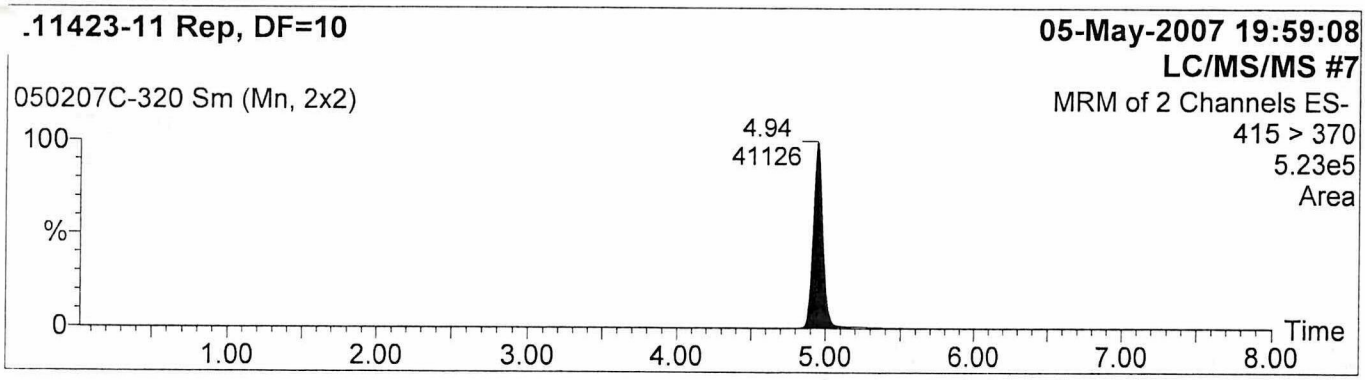
Printed: Tue May 08 13:55:58 2007

Name: 050207C-320
Text:

1: PFOA



2: 13C-PFOA

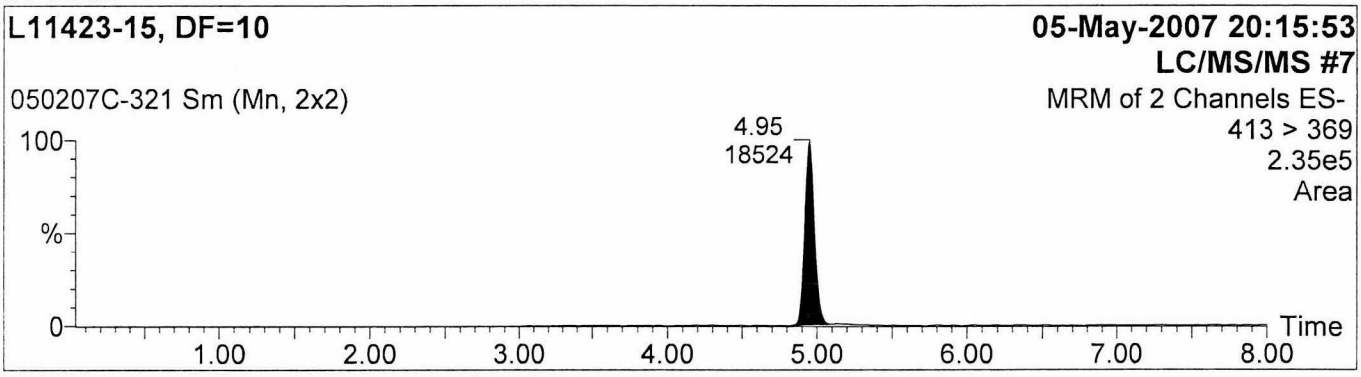


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

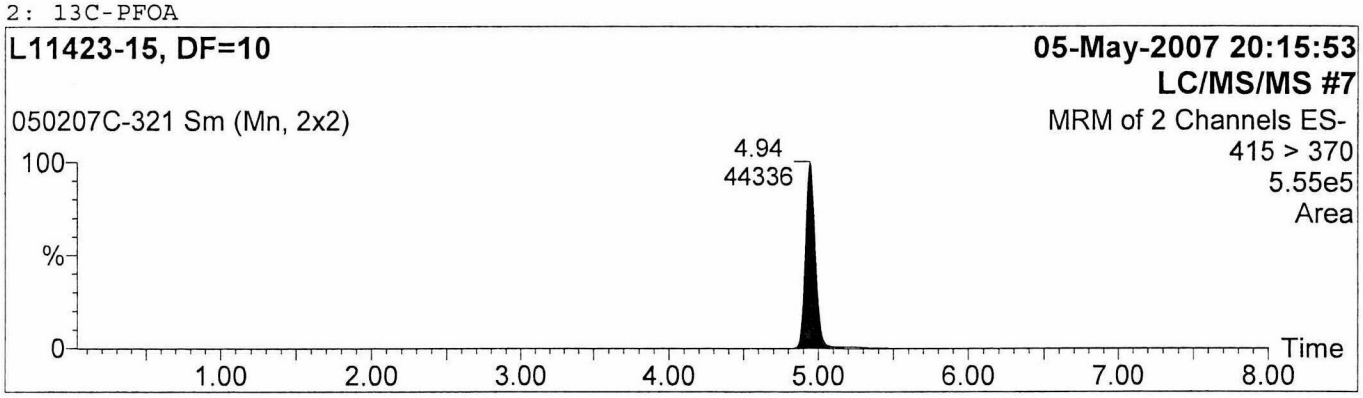
Printed: Tue May 08 13:55:58 2007

Name: 050207C-321
Text:

1: PFOA



2: 13C-PFOA



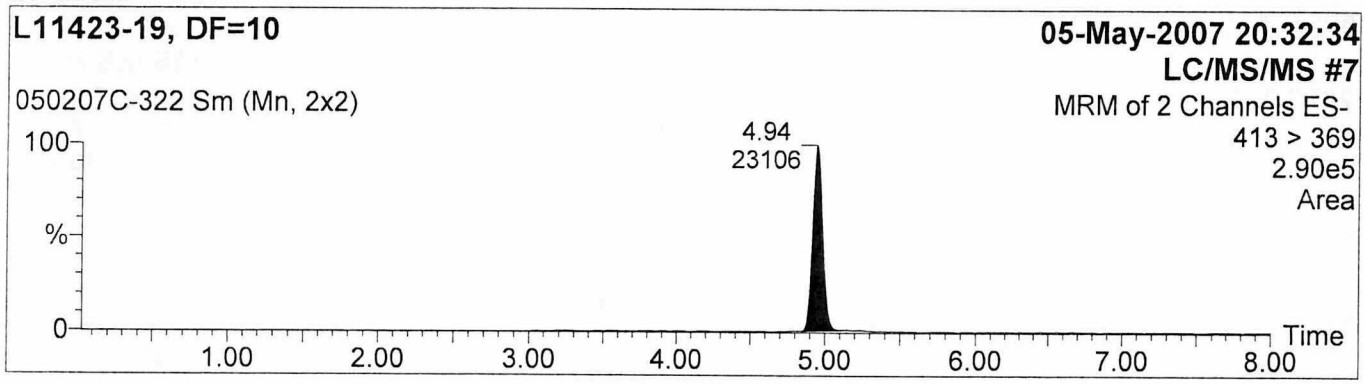
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

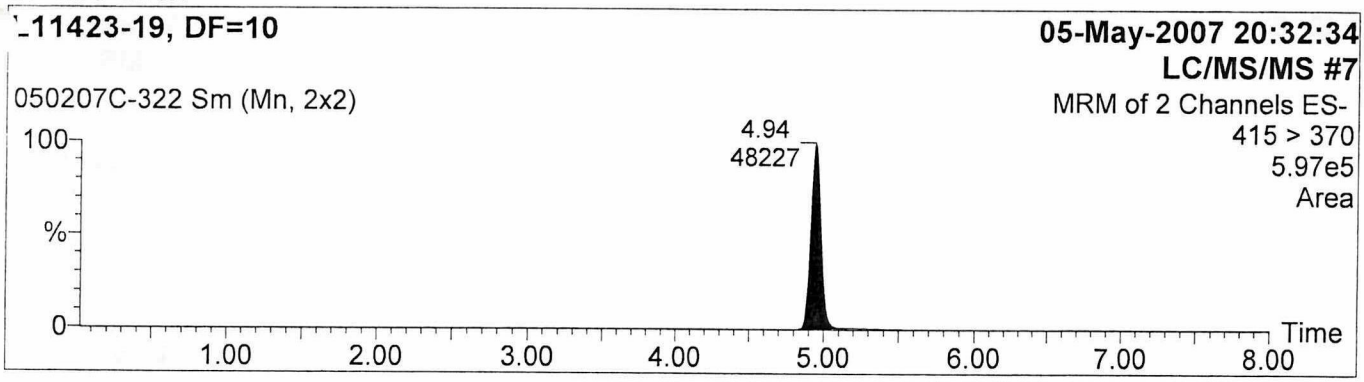
Printed: Tue May 08 13:55:58 2007

Name: 050207C-322
Text:

1: PFOA



2: 13C-PFOA



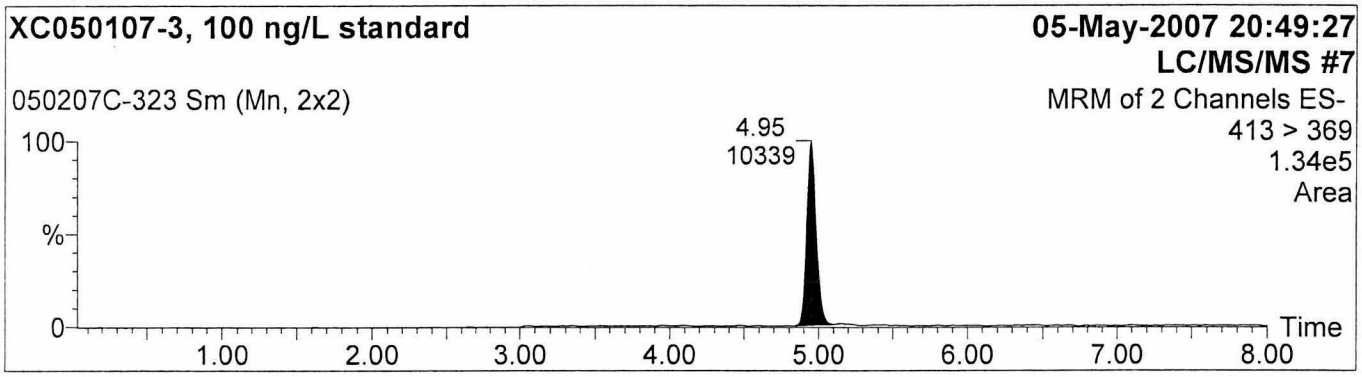
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

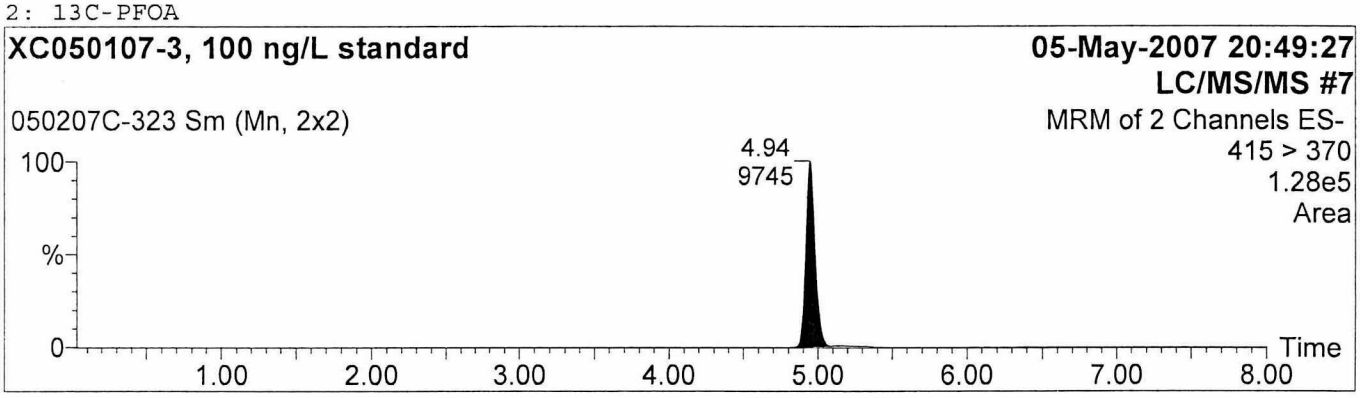
Printed: Tue May 08 13:55:58 2007

Name: 050207C-323
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
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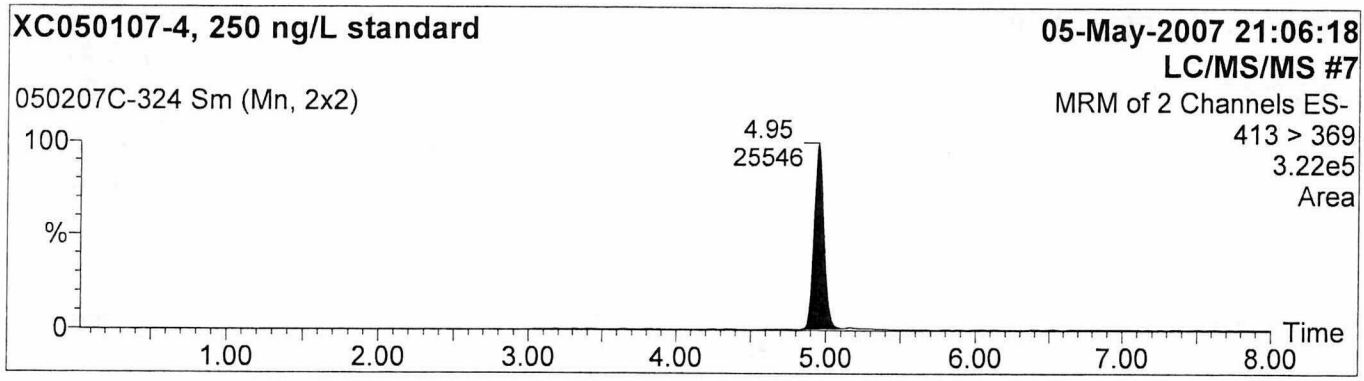
Last modified: Thu May 03 10:45:11 2007
Job Code:

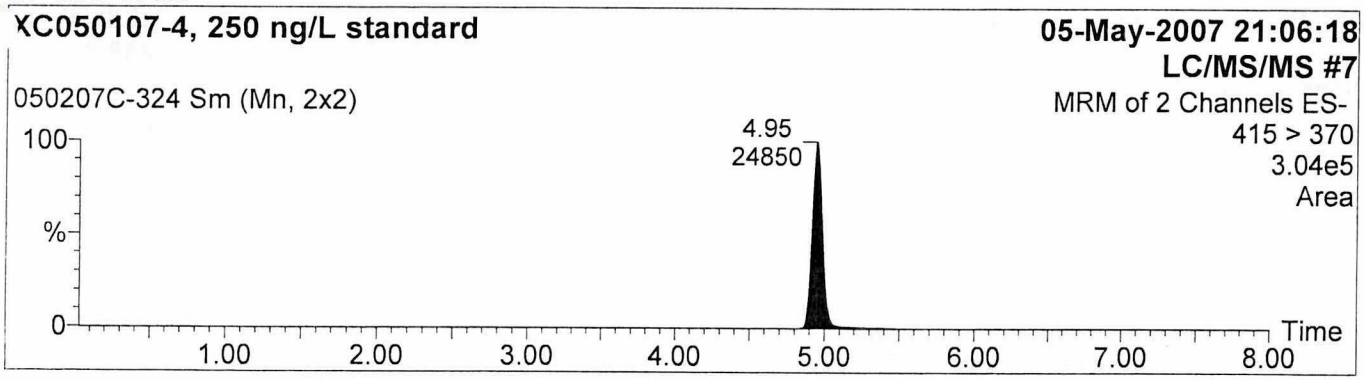
Printed: Tue May 08 13:55:58 2007

Name: 050207C-324
Text:

1: PFOA



2: 13C-PFOA



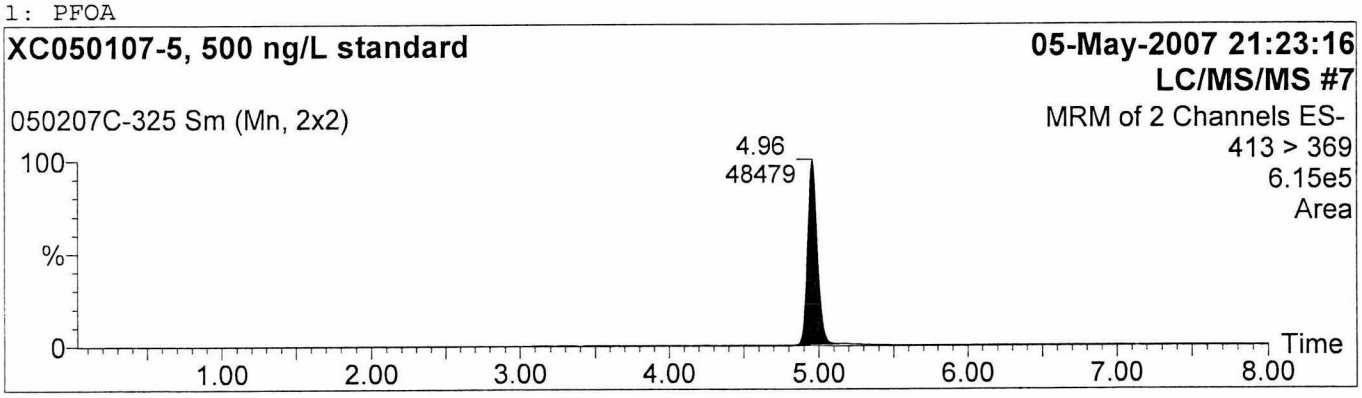
Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

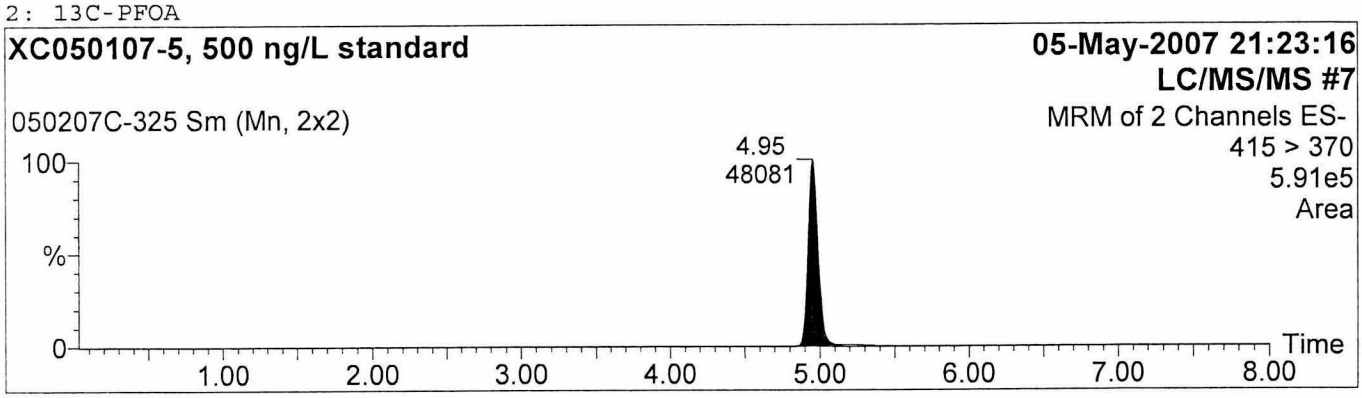
Printed: Tue May 08 13:55:58 2007

Name: 050207C-325
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050207C. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207C Buffer
Last modified: Tue May 08 13:30:37 2007
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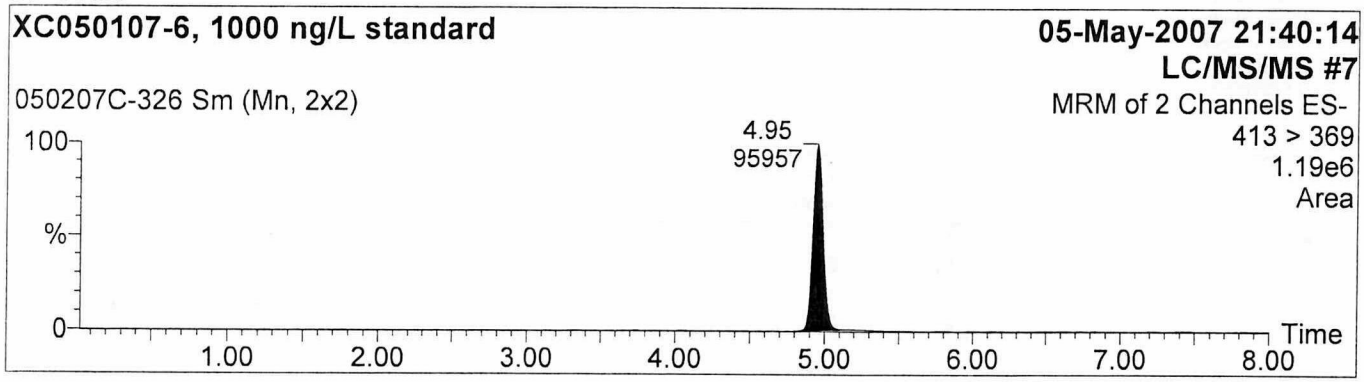
Last modified: Thu May 03 10:45:11 2007
Job Code:

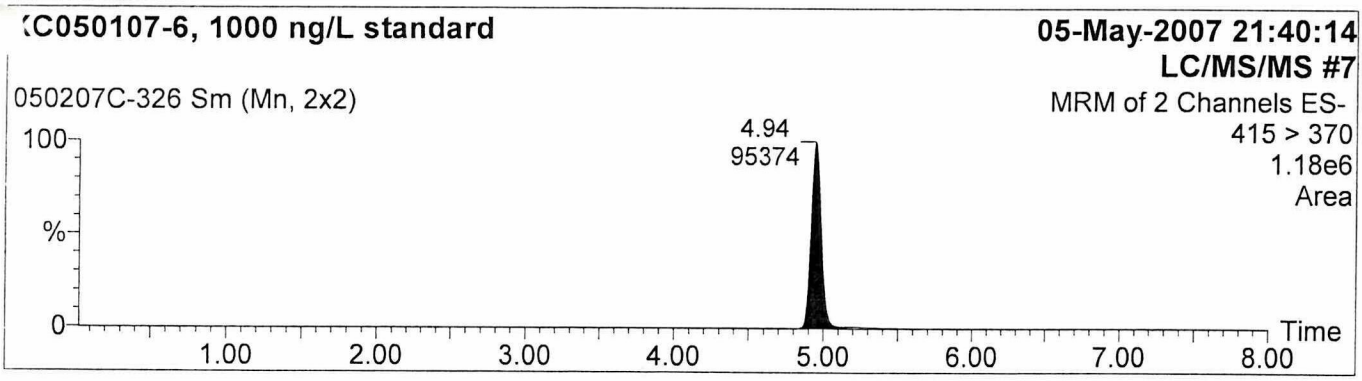
Printed: Tue May 08 13:55:58 2007

Name: 050207C-326
Text:

1: PFOA



2: 13C-PFOA



RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050907C
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	Buffer	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	4.0 mL	Extraction Date:	5/9/2007
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	5/11/2007

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)	
XC050107-0	-	CS	050907C-301	0	-	-	0	-	-	-	-	
XC050107-1	-	CS	050907C-302	25	-	-	2610	-	-	-	-	
XC050107-2	-	CS	050907C-303	50	-	-	4691	-	-	-	-	
XC050107-3	-	CS	050907C-304	100	-	-	9950	-	-	-	-	
XC050107-4	-	CS	050907C-305	250	-	-	24207	-	-	-	-	
XC050107-5	-	CS	050907C-306	500	-	-	48261	-	-	-	-	
XC050107-6	-	CS	050907C-307	1000	-	-	95436	-	-	-	-	
Methanol Wash	-	C	050907C-308	-	-	-	0	-	-	-	-	
Reagent Control	NA	C	050907C-309	-	-	1	0	ND	ND	-	-	
Reagent Spk A	NA	LCS	050907C-310	-	-	1	6169	59.1	61.5	50	118	
Reagent Spk B	NA	LCS	050907C-311	-	-	1	49371	494	514	500	99	
L11423-11 Spk C	SG-A-011	LF	050907C-312	-	100	100	8200	7950	8280	5000	111	*
Methanol Wash	-	C	050907C-314	-	-	-	0	-	-	-	-	
XC050107-1	-	CS	050907C-315	25	-	-	2885	-	-	-	-	
XC050107-2	-	CS	050907C-316	50	-	-	5665	-	-	-	-	
XC050107-3	-	CS	050907C-317	100	-	-	10872	-	-	-	-	
XC050107-4	-	CS	050907C-318	250	-	-	26221	-	-	-	-	
XC050107-5	-	CS	050907C-319	500	-	-	54103	-	-	-	-	
XC050107-6	-	CS	050907C-320	1000	-	-	101436	-	-	-	-	

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 294.123
Slope = 99.4304
Coef. Of Det. = 0.996793

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Lab fortified sample corrected using results found in data set 050207C.

Spreadsheet prepared by: MLC / 5/15/07

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

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423 Matrix: $Na_2B_4O_7$ Impinger Sample
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 34
Time 8:30 AM Date 5/9/2007 Initials EDD

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-A-011	L11423-11* Spk C	100 mL	40.0	20.0
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/9/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/9/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/9/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/9/07
L11423-11* Spk C	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/9/07
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-

All samples were measured:
Time 10:30 AM Date 5/9/2007 Initials EDD

After measuring samples were returned to refrigerator No. 34
Time 3:00 PM Date 5/9/2007 Initials EDD

Comments: *Sample is pre-diluted ten times by taking 4 mL of sample and bringing the volume up to 40 mL with HPLC water

Analysis Summary:
Data Set: 050907C Initials/Date: MLC / 5/11/07
Data Set: - Initials/Date: - / -
Data Set: - Initials/Date: - / -

Set extraction/analysis data verified by: CEE Date: 05/23/07

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

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007C[$$] PROTOCOL NUMBER: NA

MATRIX: Na2B4O7 Impinger Sample ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	Reagent Control	↓	↓	–	↓	–	–	Methanol	46314
na	Reagent Spk A	↓	↓	–	↓	–	–	C18 SPE	0053370818
na	Reagent Spk B	↓	↓	–	↓	–	–	HPLC Water	46352
SG-A-011	L11423-11* Spk C	√	√	0.1/1.0	↓	–	–	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	*Initials/Date*	EDD 5/9/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C441404
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date	-	EDD 5/9/07	EDD 5/9/07	MLC 5/9/07	MLC 5/11/07	–	–	*Initials/Date*	MLC 5/11/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

*Sample was diluted ten times prior to extraction.
**Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:

Final extracts stored in refrigerator 32 Initials: EDD Date: 5/9/2007

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

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Exygen STUDY NO. L11423

Protocol No.: None
Method No.: $$

Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA

Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC050107-0	Methanol	46303
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC050107-1	C18 SPE	0053370818
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC050107-2	Water	46335
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC050107-3	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC050107-4	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC050107-5	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC050107-6	-	-
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/01/07
-	-	-	-	-	-	-	-		

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) freezer # 34 Time: 9:10 AM — Initials/Date: EDD 1 5/01/07

40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD 1 5/01/07

After measuring, samples returned to (refrigerator) freezer # 34 Time: 11:10 AM — Initials/Date: EDD 1 5/01/07

Samples fortified: — Initials/Date: AMB 1 05/01/07

SPE clean-up: — Initials/Date: EDD 1 5/01/07

Final volume adjusted to 5 mL: — Initials/Date: EDD 1 5/01/07

Extracts placed in refrigerator # 32 — Initials/Date: EDD 1 5/01/07

Comments:

STANDARD EXPIRATION DATE: 5/15/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/15/07

Exygen STUDY NO. L11423

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050907C Buffer.SPL
Printed: Fri May 11 09:07:40 2007 MLC 5/11/07

Page Position: (1, 1)

L (KE) MLC 5/11/07

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/~~mL~~)	Conc B	Conc C	Test ID	DF
1	94	050907C-301	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---	0	1
2	95	050907C-302	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
3	96	050907C-303	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
4	97	050907C-304	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
5	98	050907C-305	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
6	99	050907C-306	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
7	100	050907C-307	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1
8	92	050907C-308	---	---	Methanol Wash	---	Blank	---	---	---	0	1
9	31	050907C-309	---	---	Reagent Control	---	Blank	---	---	---	0	1
10	32	050907C-310	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---	0	1
11	33	050907C-311	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500	---	0	1
12	34	050907C-312	---	---	L11423-11 Spk C, 5,000 ng/L, DF=100	---	QC	5000	5000	---	0	100
13	35	050907C-313	---	---	L11423-11, DF=10	---	Analyte	---	5000	---	0	10
14	92	050907C-314	---	---	Methanol Wash	---	Blank	---	---	---	0	1
15	95	050907C-315	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
16	96	050907C-316	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
17	97	050907C-317	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
18	98	050907C-318	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
19	99	050907C-319	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
20	100	050907C-320	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050907C Buffer.SPL
Printed: Fri May 11 09:07:40 2007

MLC 5/11/07

Exygen STUDY NO. 111423

Page Position: (2, 1)

	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
20	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA

Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/11/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/15/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Fri May 11 09:08:04 2007

MLC 5/11/07

Solvent Delay (mins) : 0.00

Function : 1 MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Friday, May 11, 2007 08:57:10

Printed: Friday, May 11, 2007 09:08:27 MLC 5/11/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

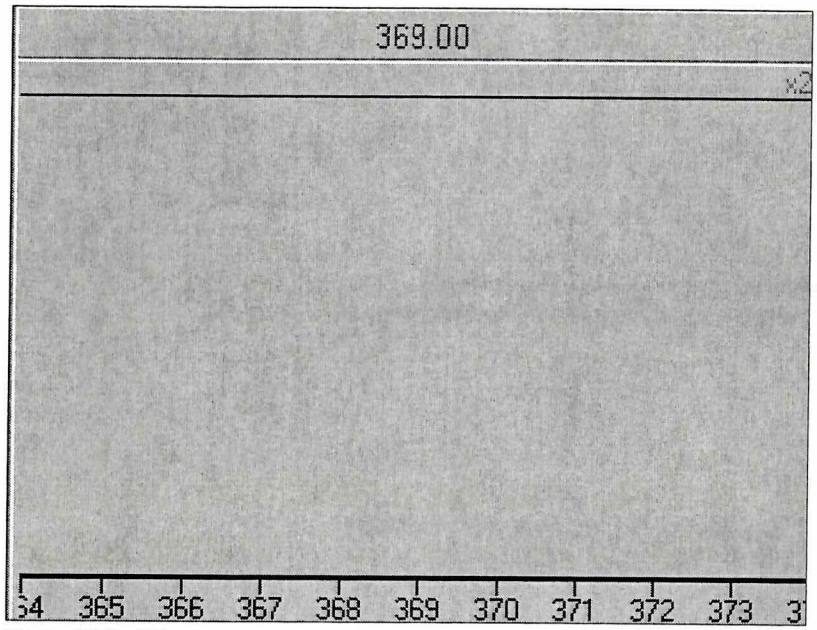
HP1100 Autosampler Initial Conditions

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	21

Tuning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Fri May 11 09:08:39 2007 MLC 5/11/07



Dau 413.00

SOURCE (ESP-)	**Set**	**Rdbk**
Capillary	3.00	-2.94
Cone	10	-10
Hexapole 1	0.0	
Aperture 1	0.0	
Hexapole 2	0.0	
Source Block Temp.	100	102
Desolvation Temp.	300	300

Analyser	**Set**	**Rdbk**
LM Res 1	14.0	
HM Res 1	14.0	
IEnergy 1	1.0	
Entrance	-2	8
Collision	10	9
Exit	2	12
LM Res 2	14.0	
HM Res 2	14.0	
IEnergy 2	2.0	
Multiplier	700	-695

Pressures	**Rdbk**
Analyser Vacuum	OFF
Gas Cell	2.2e-3

Gas Flows	**Rdbk**
Cone Gas	94.0
Desolvation	681.0

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050907C
Extraction Date: 05/09/07
Analyzed on: 05/11/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2610
XC050107-2	50	4691
XC050107-3	100	9950
XC050107-4	250	24207
XC050107-5	500	48261
XC050107-6	1000	95436

Coefficient of Determination: 0.999955481

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.99997774
R Square	0.999955481
Adjusted R Square	0.999944351
Standard Error	267.6665311
Observations	6

MCC
5/15/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	6436937561	6436937561	89844.42951	7.43253E-10
Residual	4	286581.4874	71645.3719		
Total	5	6437224143			

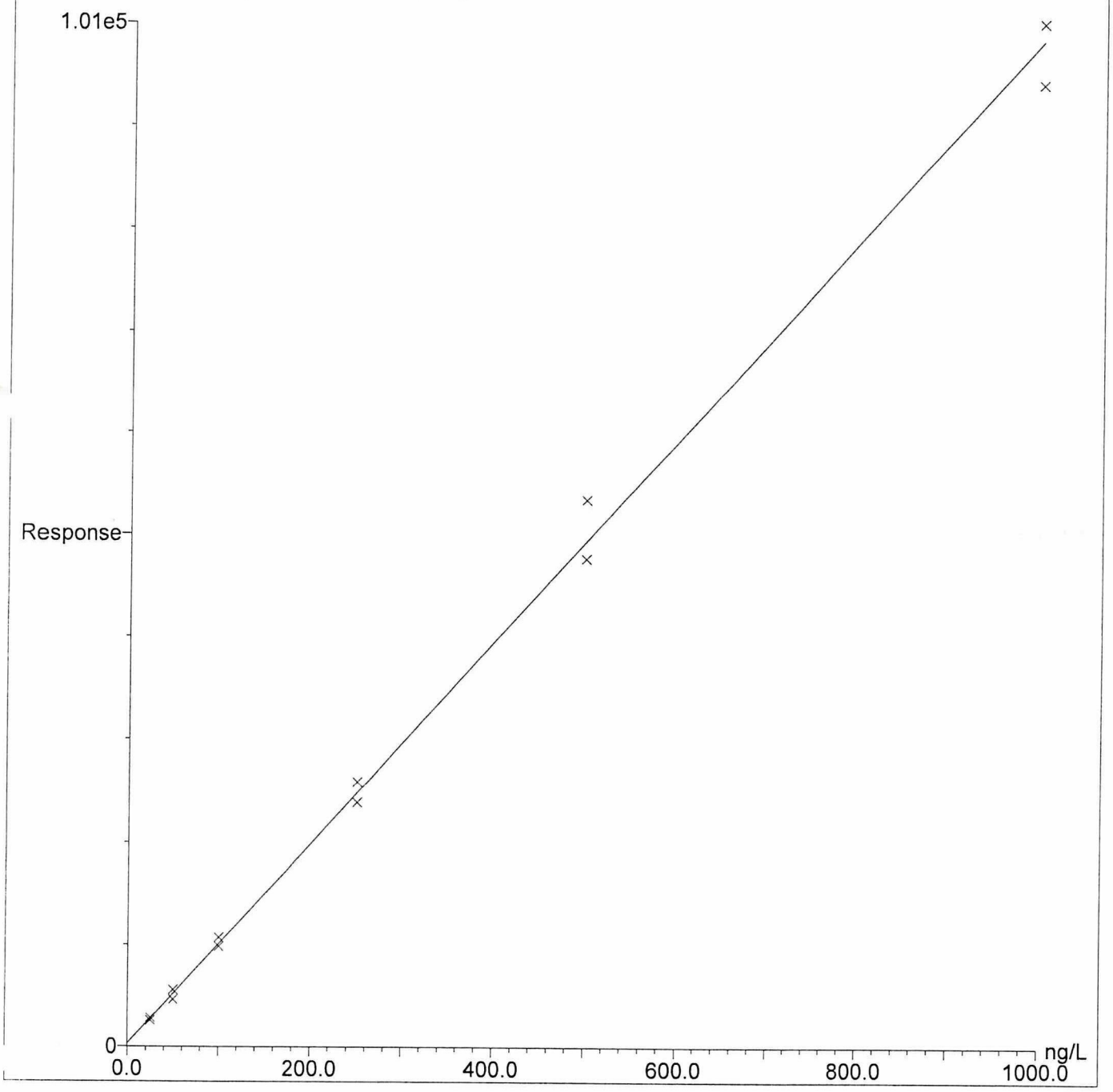
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	267.9948507	149.5222315	1.79234117	0.147547853	-147.145417	683.1351183	-147.145417	683.1351183
X Variable 1	95.34910696	0.318105408	299.740604	7.43253E-10	94.46590476	96.23230916	94.46590476	96.23230916

Quantify Calibration Report
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050907C Buffer
Last modified: Tue May 15 10:26:48 2007
Printed: Tue May 15 10:27:52 2007

MLC 5/15/07

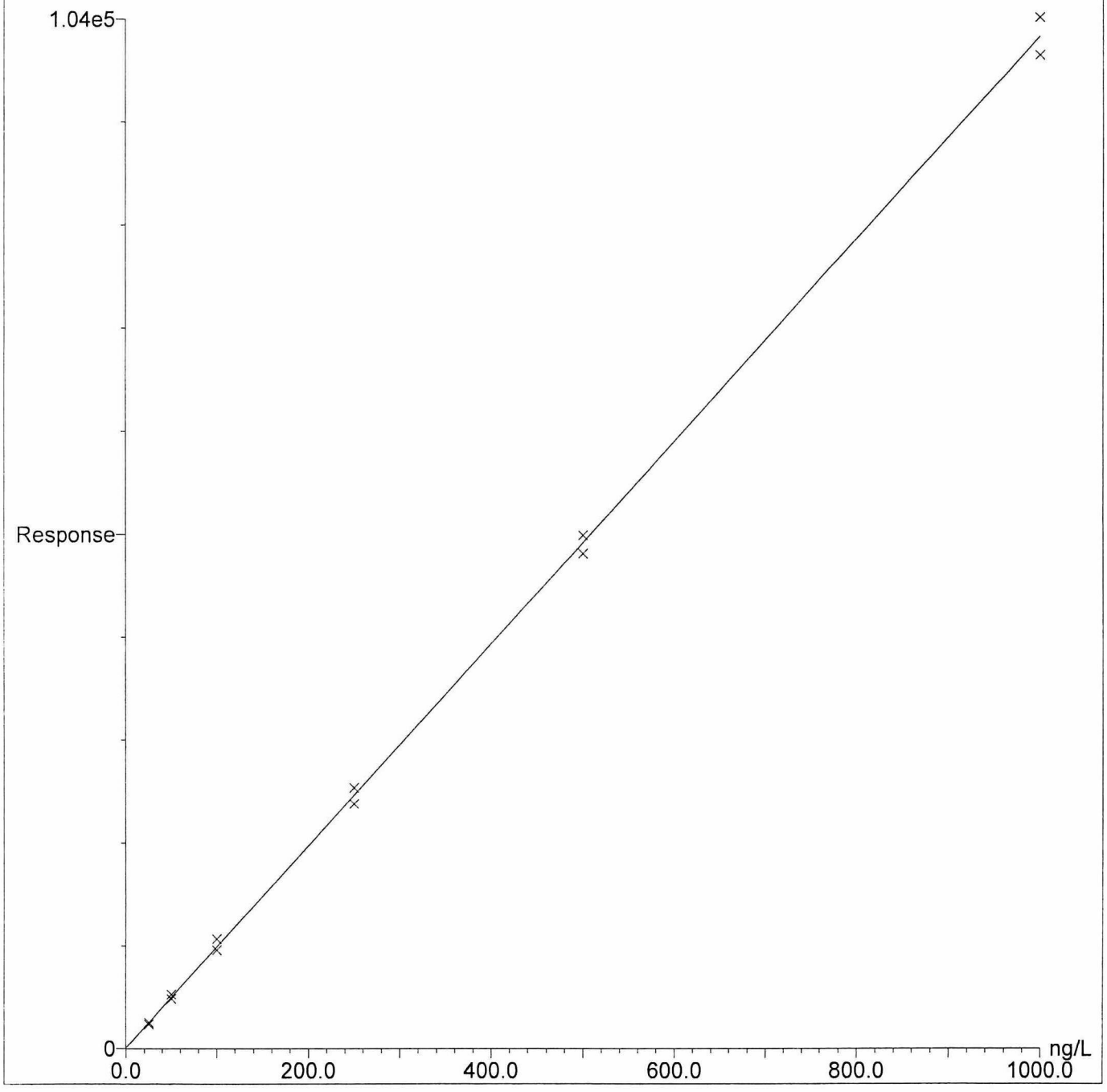
Compound 1 name: PFOA
Coefficient of Determination: 0.996793
Calibration curve: 99.4304 * x + 294.123
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050907C Buffer
Last modified: Tue May 15 10:26:48 2007
Printed: Tue May 15 10:27:52 2007 MLC 5/15/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.999238
Calibration curve: 102.127 * x + 78.9653
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



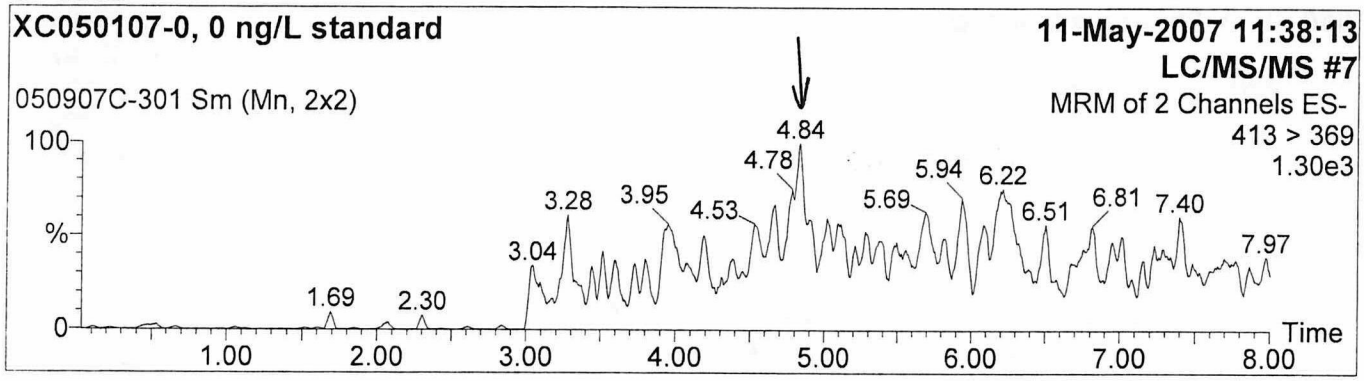
Quantify Sample Report
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

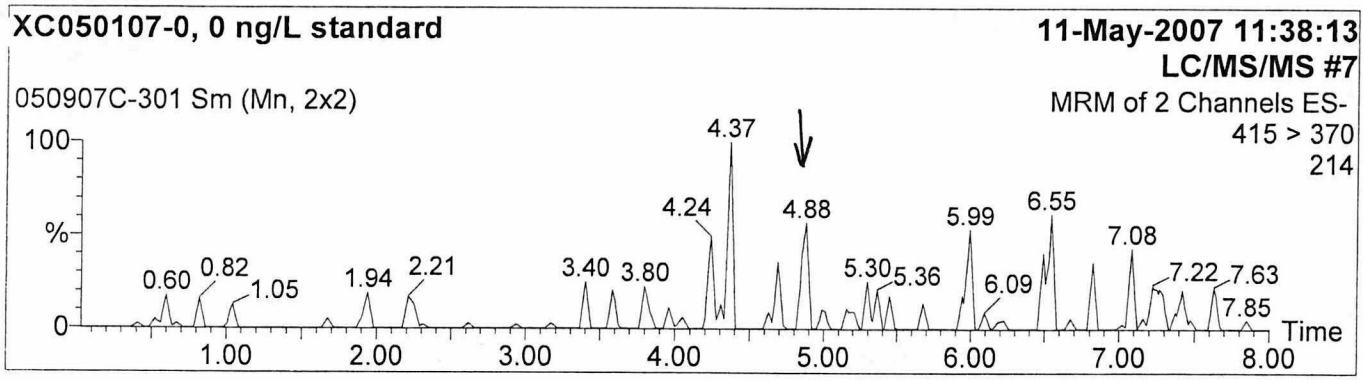
Initials MLC
Date 5/15/07
Printed: Tue May 15 10:27:53 2007 Run# 050907C-301 To 050907C-320

Name: 050907C-301
Text:

1: PFOA



2: 13C-PFOA



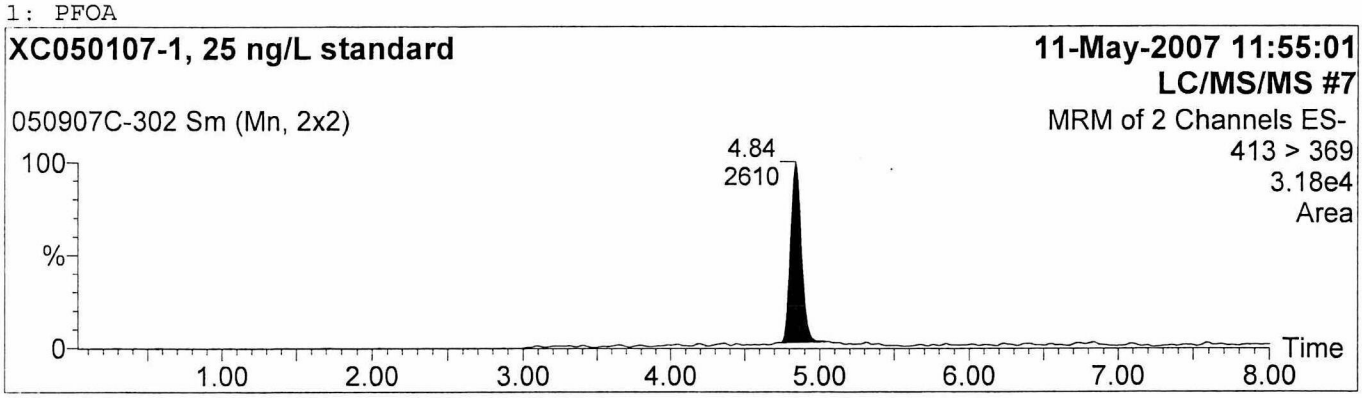
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

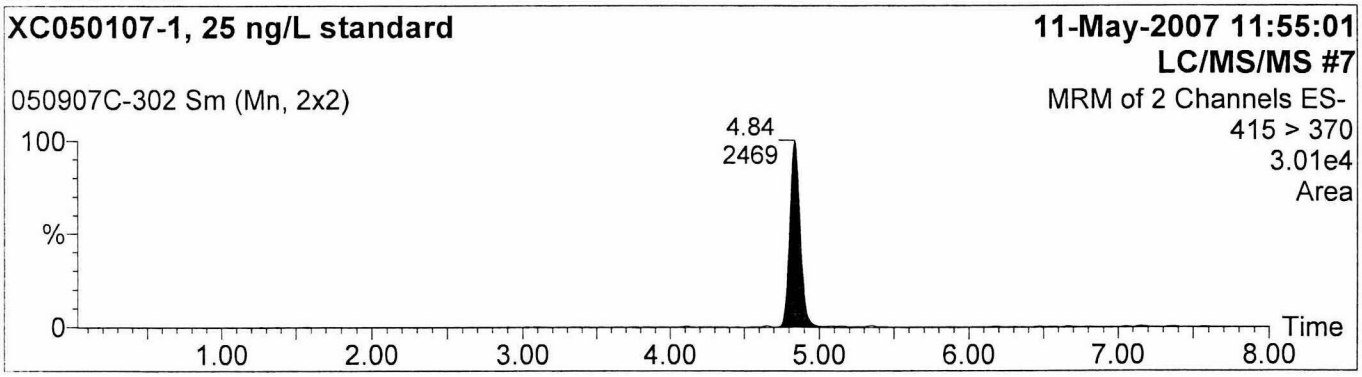
Printed: Tue May 15 10:27:53 2007

Name: 050907C-302
Text:

1: PFOA



2: 13C-PFOA



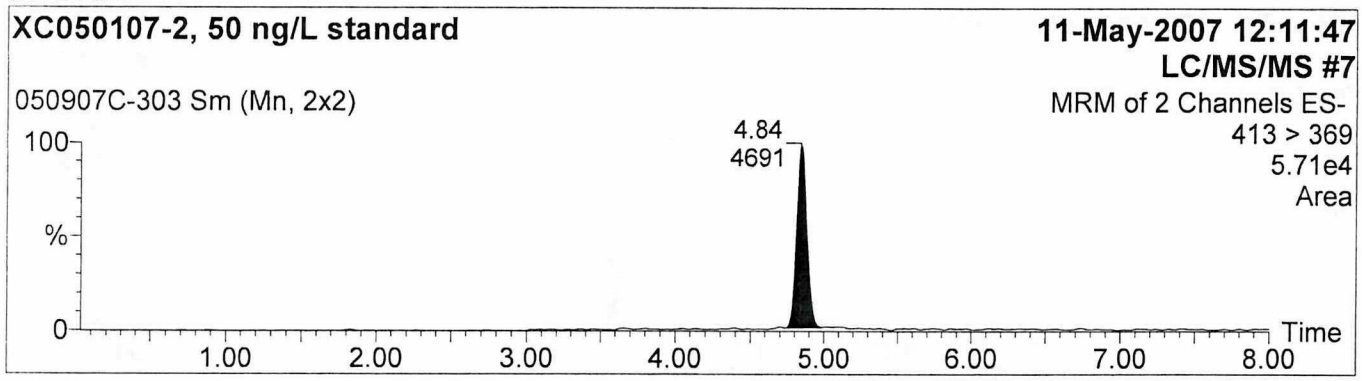
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

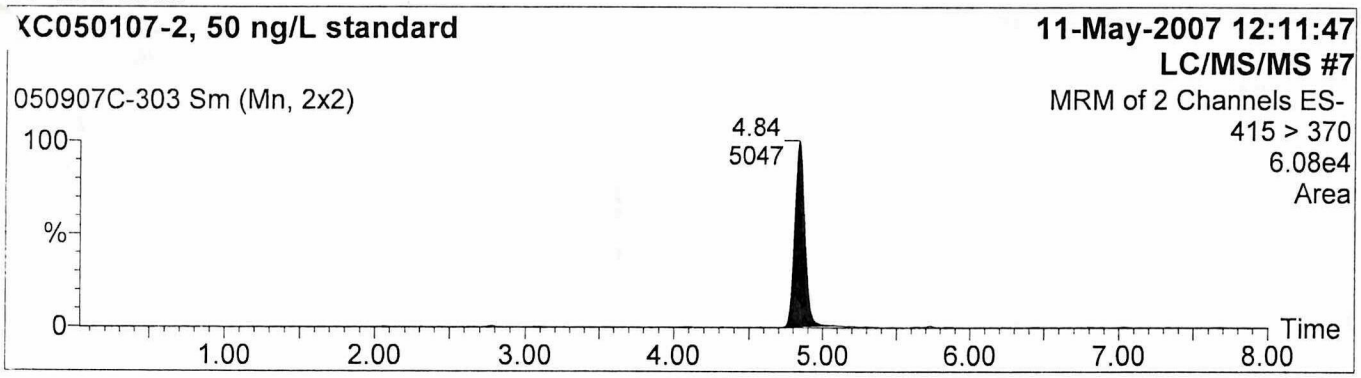
Printed: Tue May 15 10:27:53 2007

Name: 050907C-303
Text:

1: PFOA



2: 13C-PFOA



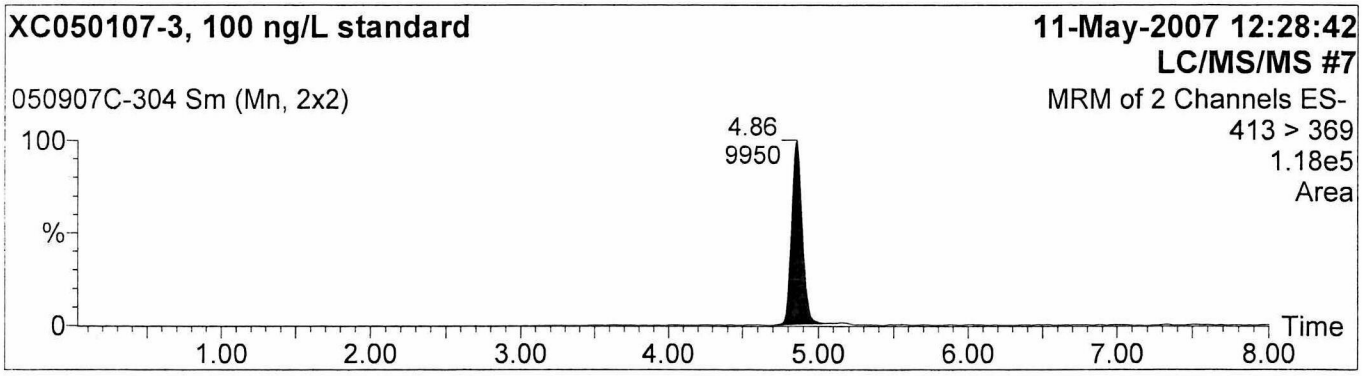
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

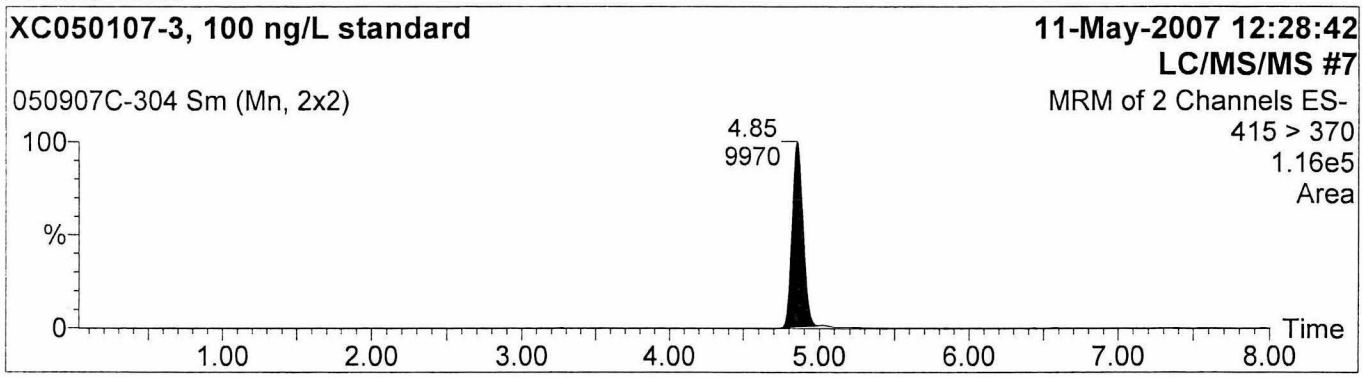
Printed: Tue May 15 10:27:53 2007

Name: 050907C-304
Text:

1: PFOA



2: 13C-PFOA



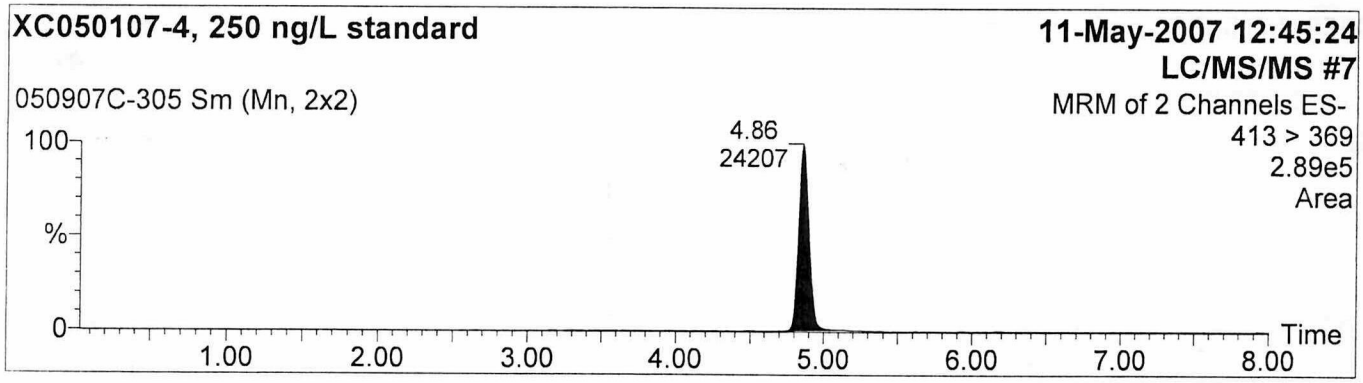
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

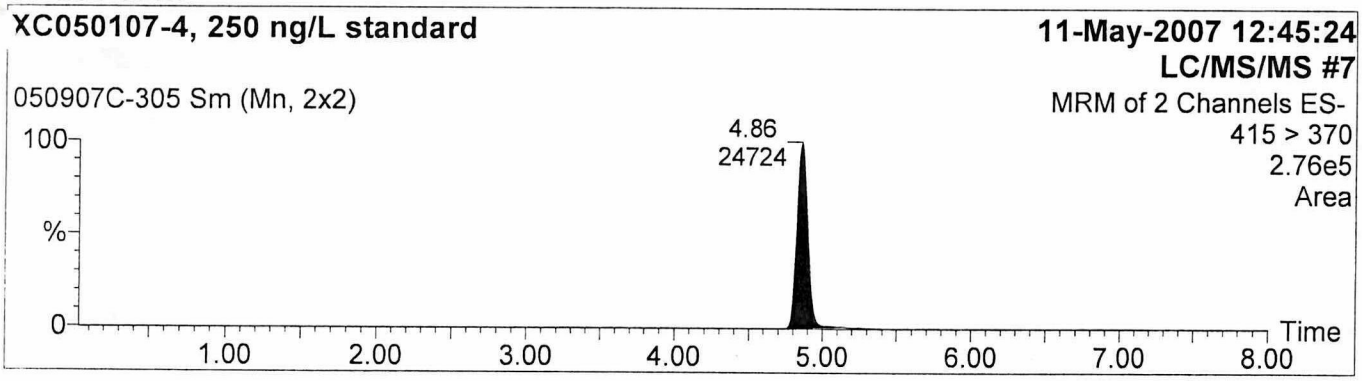
Printed: Tue May 15 10:27:53 2007

Name: 050907C-305
Text:

1: PFOA



2: 13C-PFOA



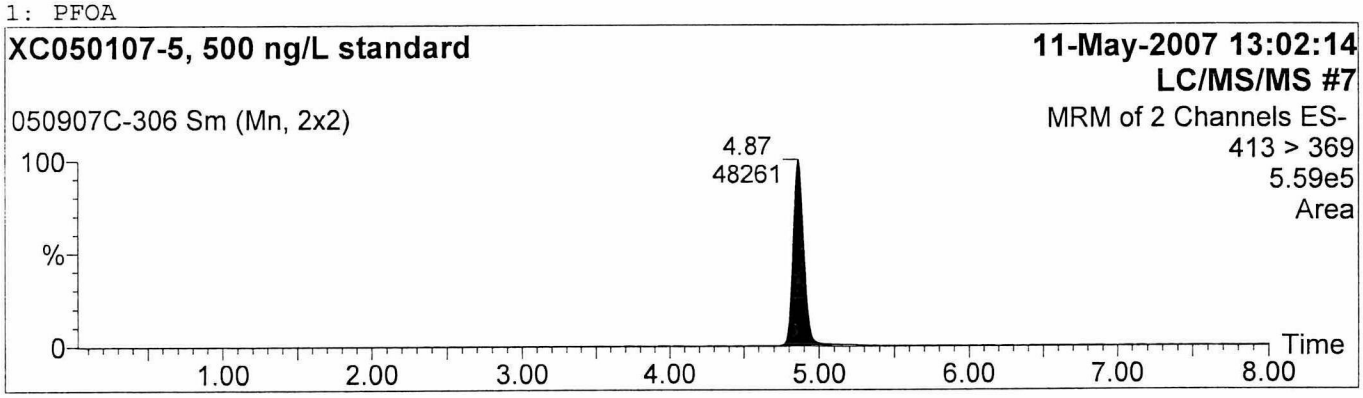
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

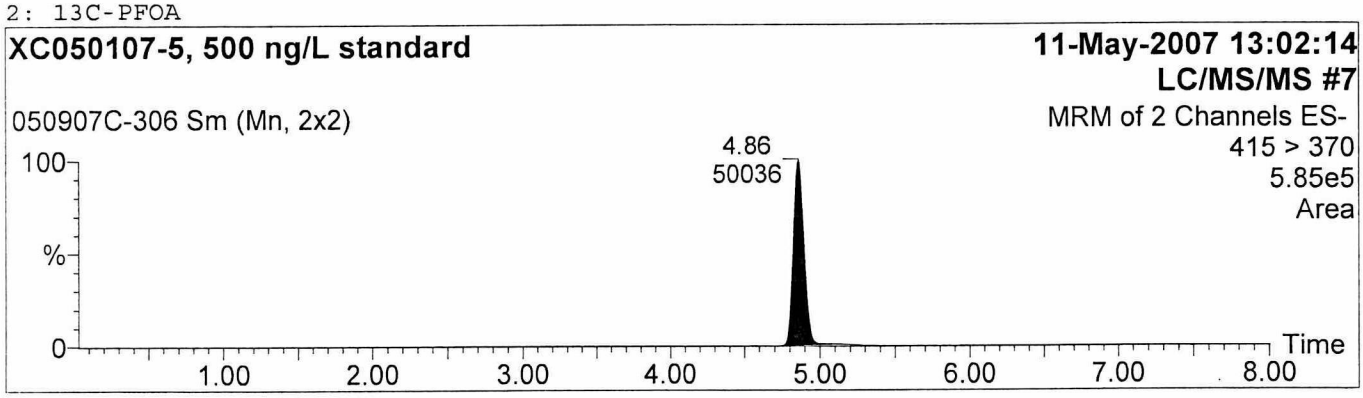
Printed: Tue May 15 10:27:53 2007

Name: 050907C-306
Text:

1: PFOA



2: 13C-PFOA



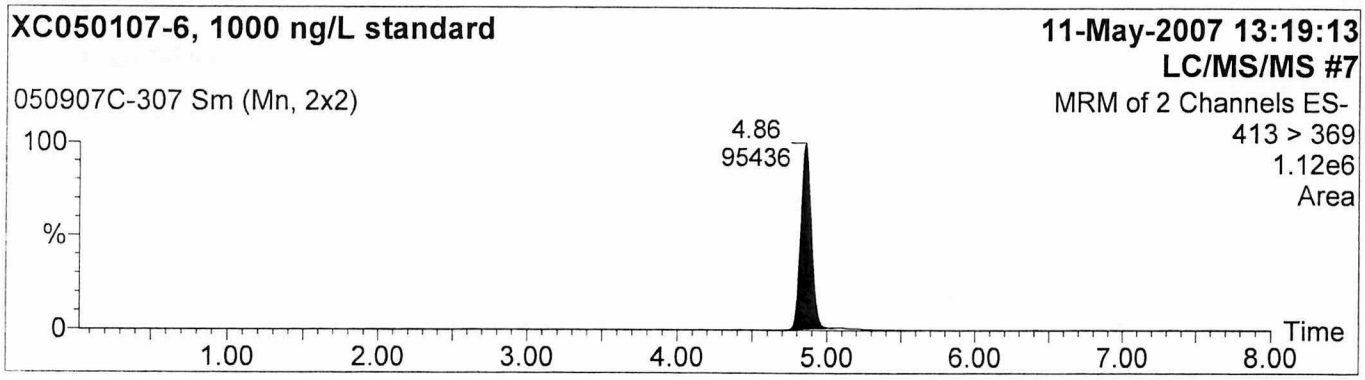
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

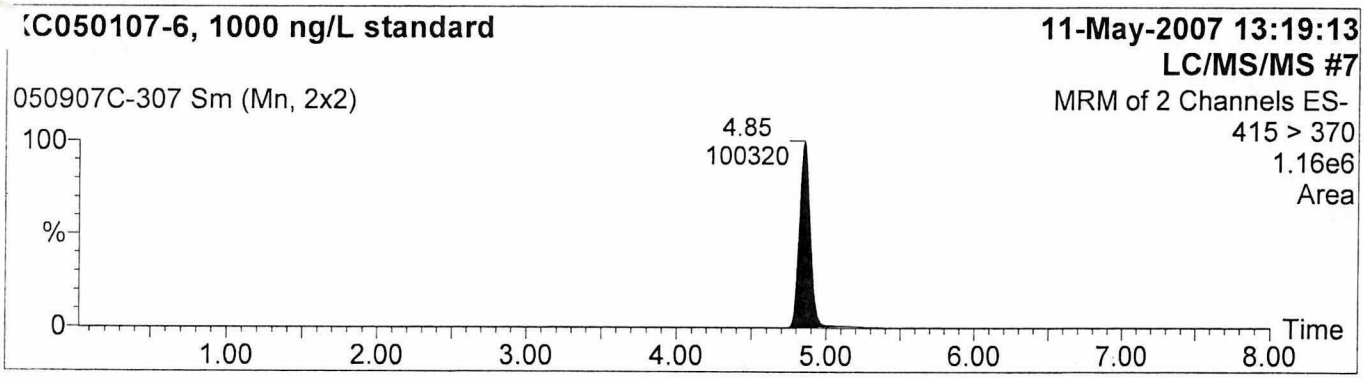
Printed: Tue May 15 10:27:53 2007

Name: 050907C-307
Text:

1: PFOA



2: 13C-PFOA



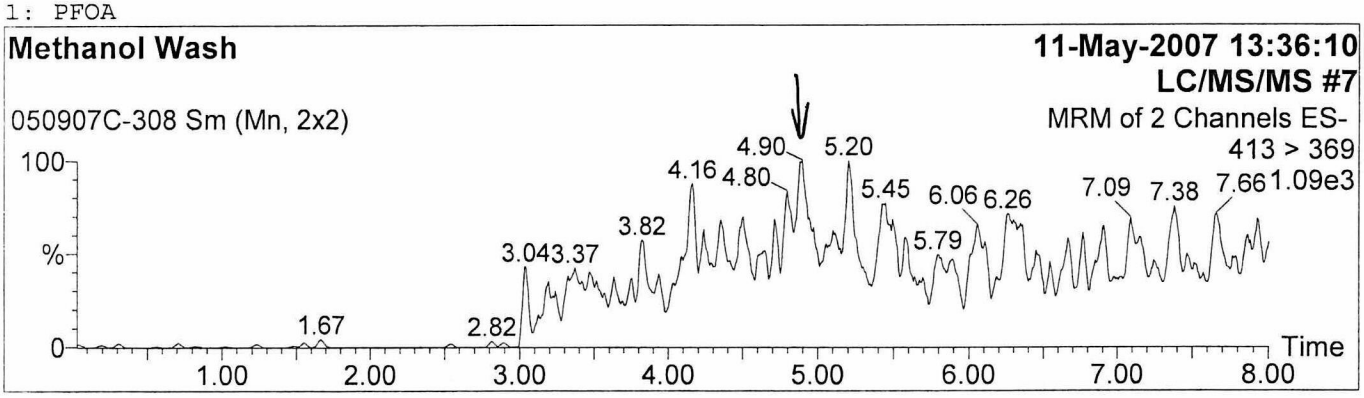
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

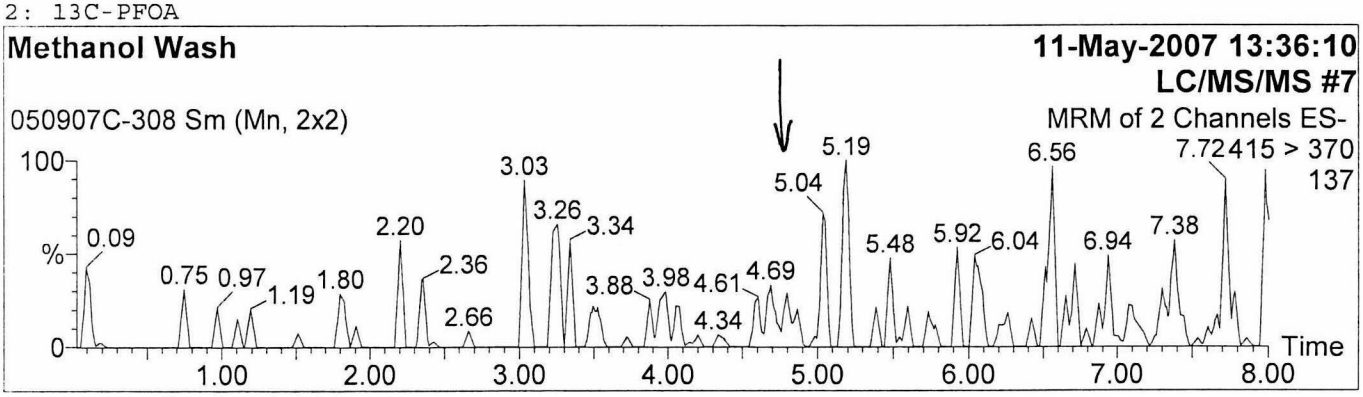
Printed: Tue May 15 10:27:53 2007

Name: 050907C-308
Text:

1: PFOA



2: 13C-PFOA



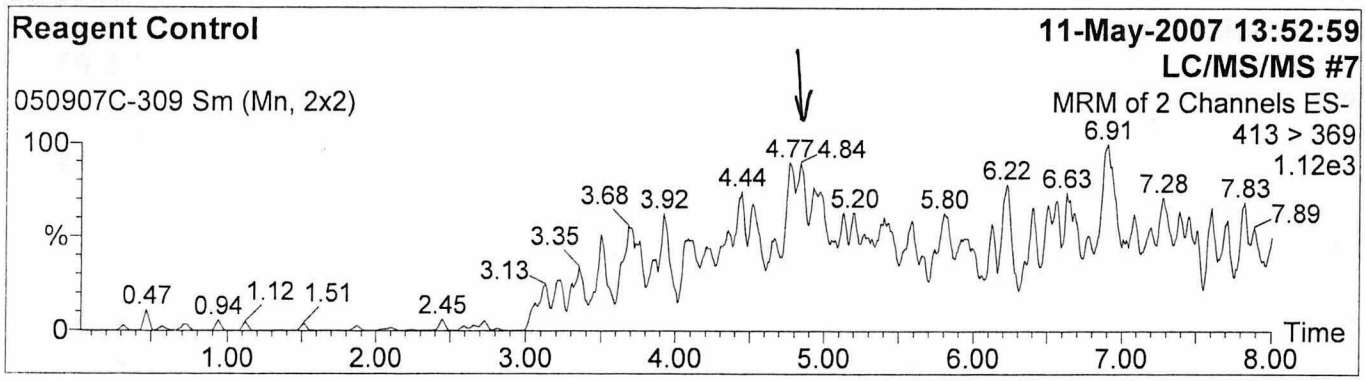
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

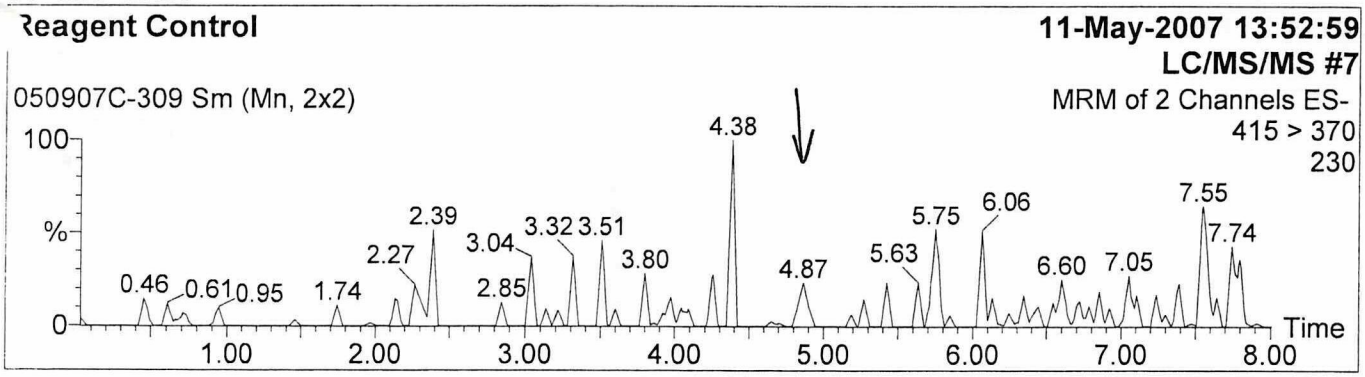
Printed: Tue May 15 10:27:53 2007

Name: 050907C-309
Text:

1: PFOA



2: 13C-PFOA

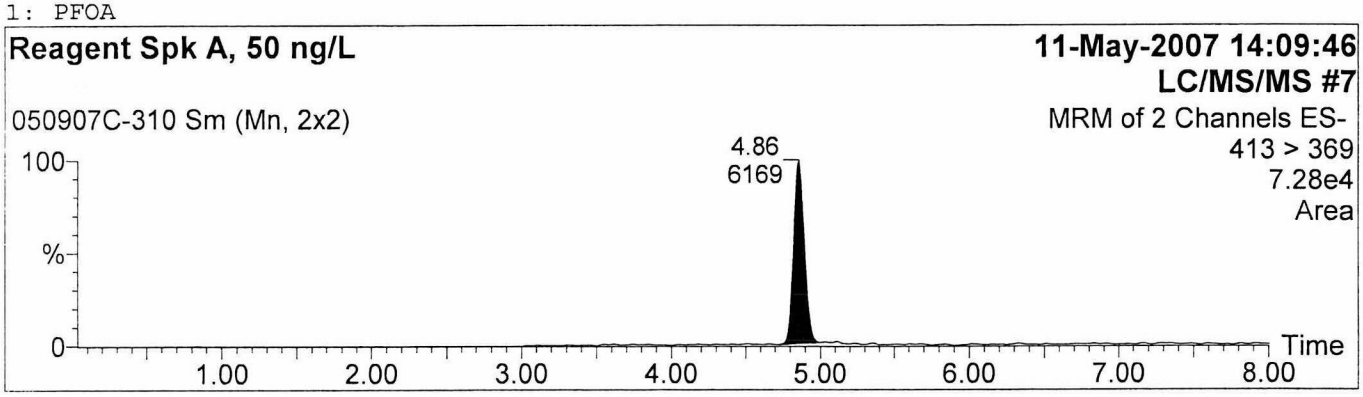


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

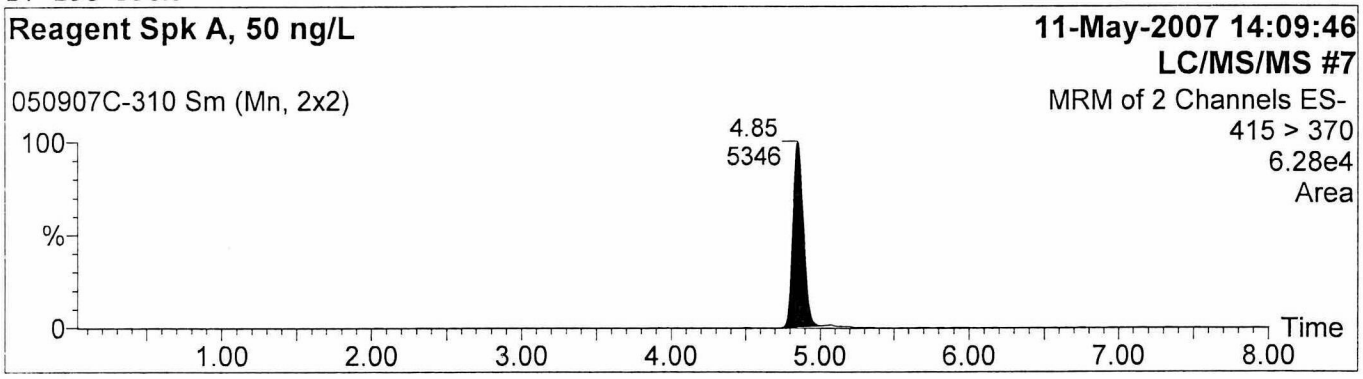
Printed: Tue May 15 10:27:53 2007

Name: 050907C-310
Text:

1: PFOA



2: 13C-PFOA



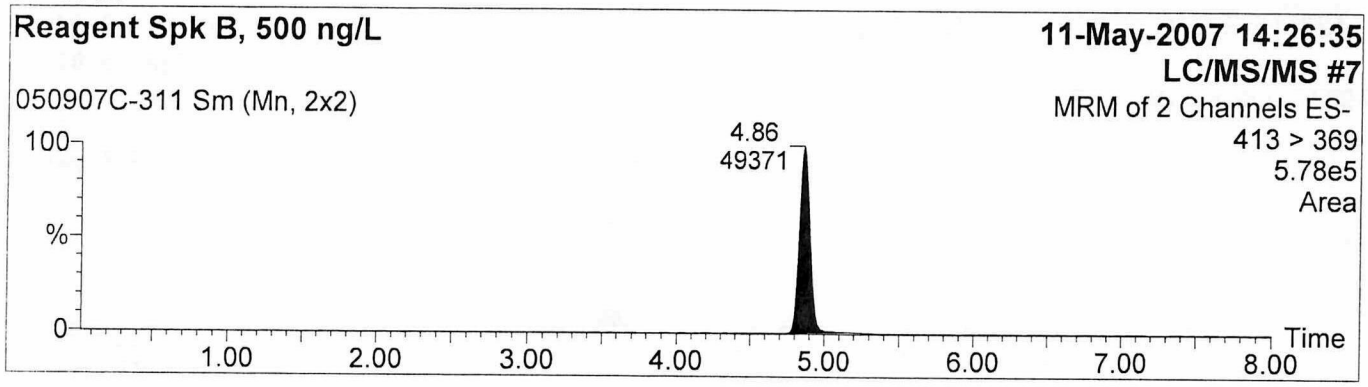
Quantify Sample Report
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

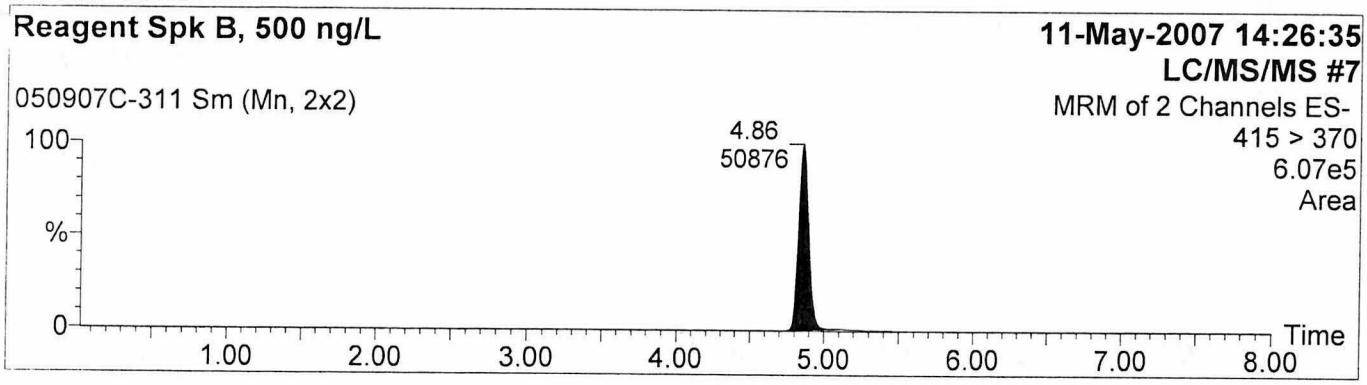
Printed: Tue May 15 10:27:53 2007

Name: 050907C-311
Text:

1: PFOA



2: 13C-PFOA



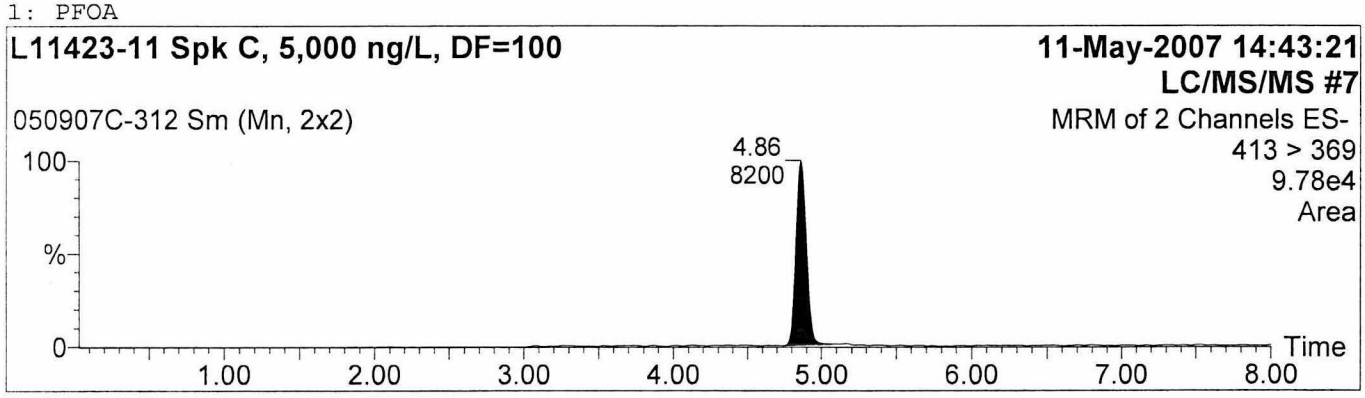
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

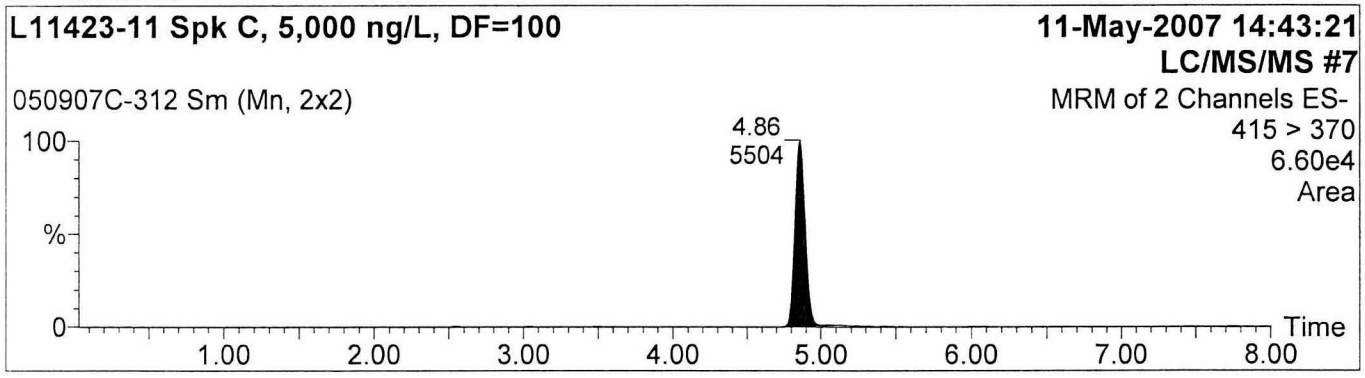
Printed: Tue May 15 10:27:53 2007

Name: 050907C-312
Text:

1: PFOA



2: 13C-PFOA



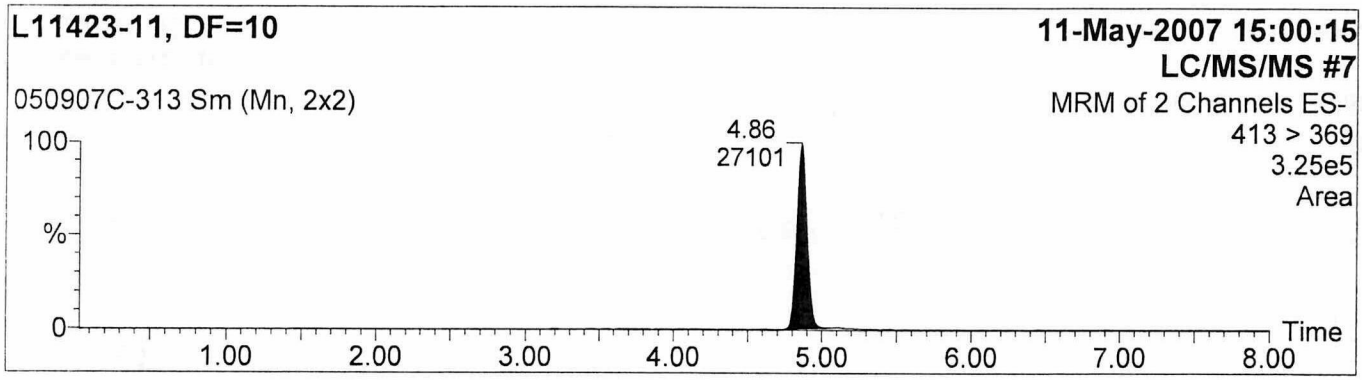
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

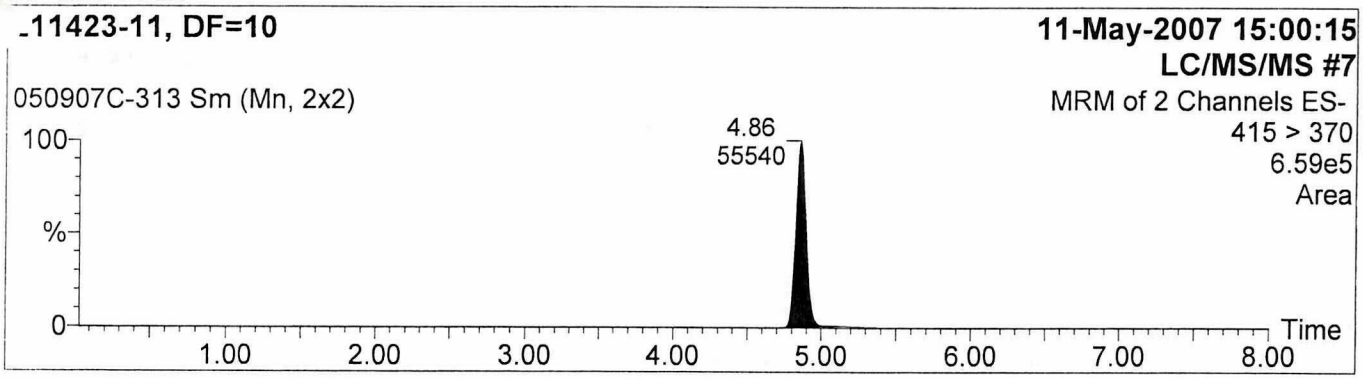
Printed: Tue May 15 10:27:53 2007

Name: 050907C-313
Text:

1: PFOA



2: 13C-PFOA



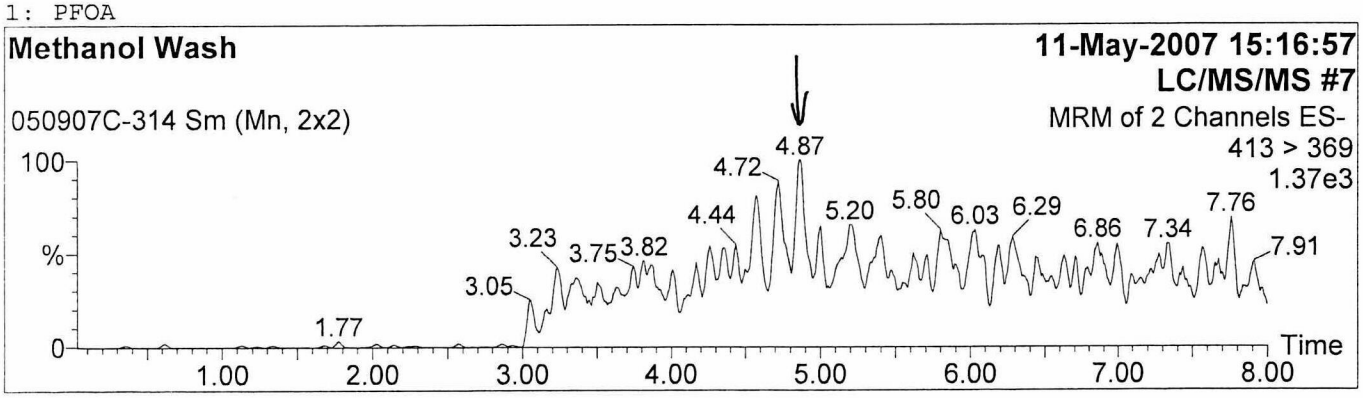
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

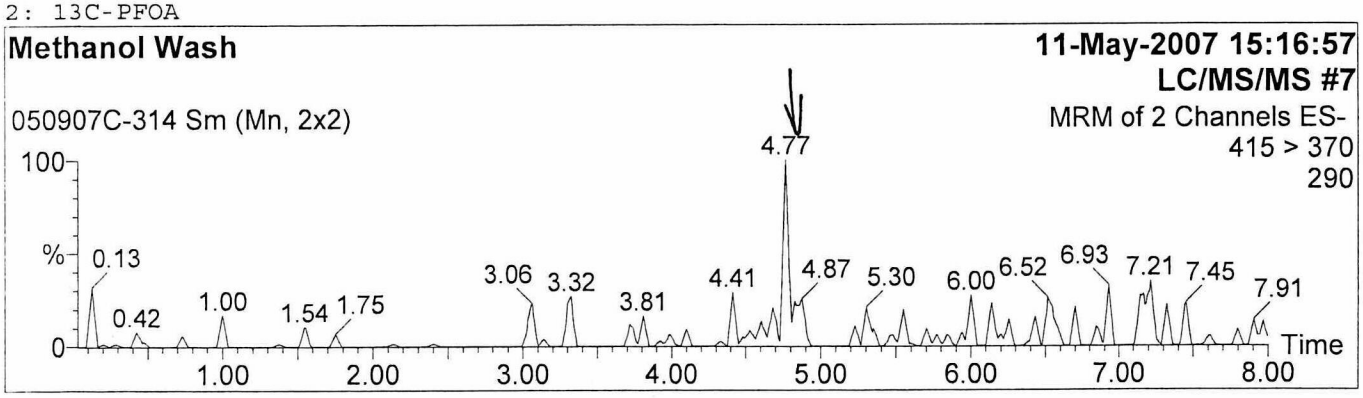
Printed: Tue May 15 10:27:53 2007

Name: 050907C-314
Text:

1: PFOA



2: 13C-PFOA



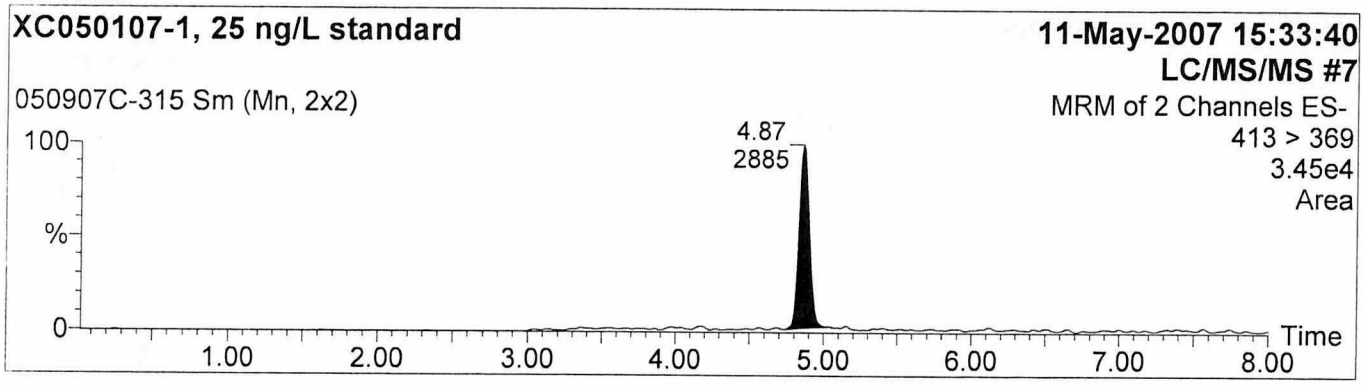
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

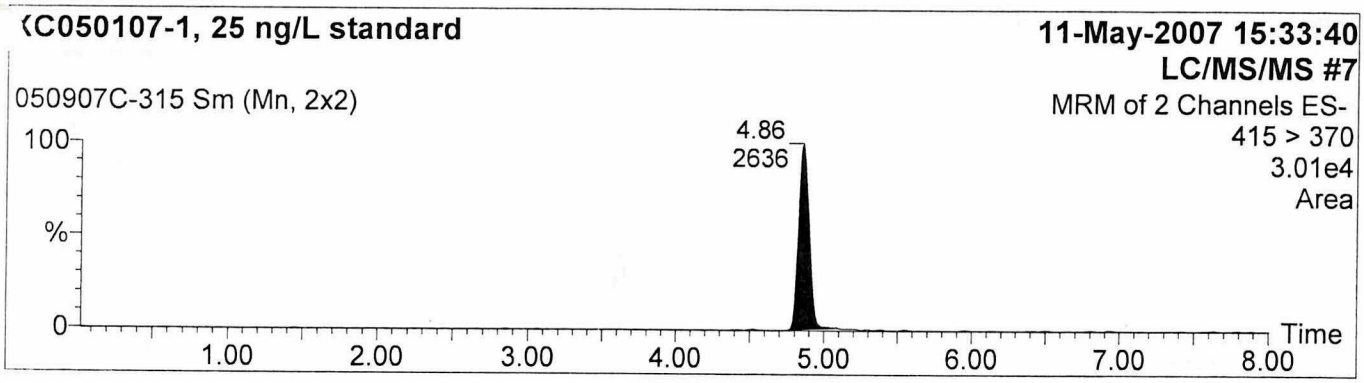
Printed: Tue May 15 10:27:53 2007

Name: 050907C-315
Text:

1: PFOA



2: 13C-PFOA



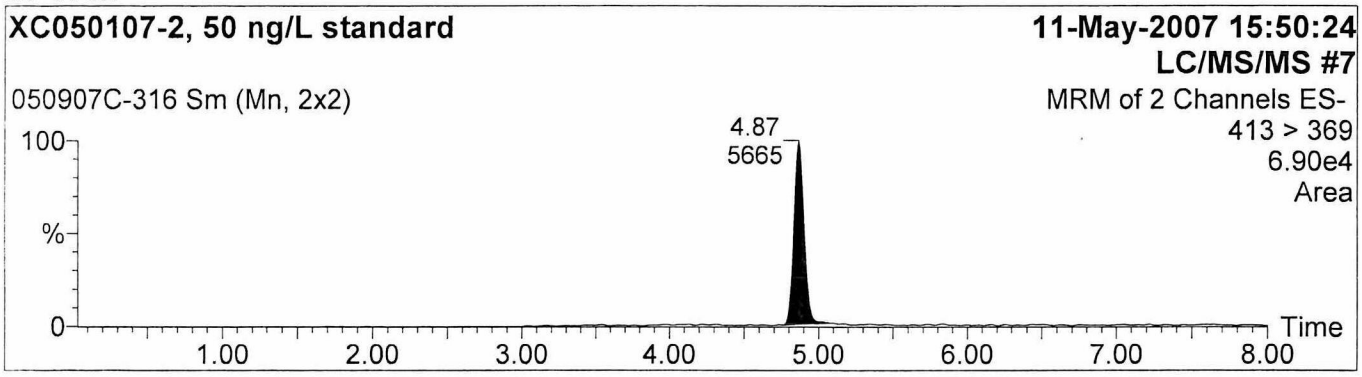
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

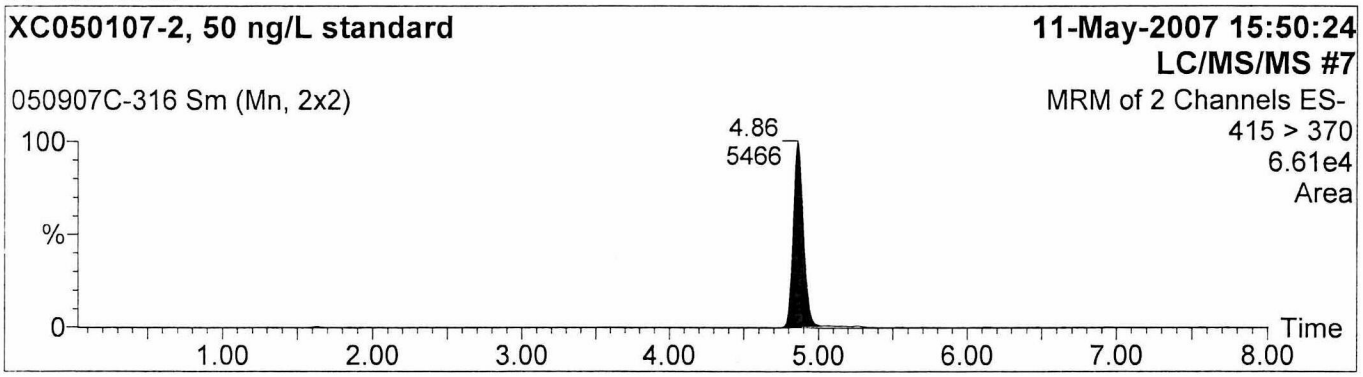
Printed: Tue May 15 10:27:53 2007

Name: 050907C-316
Text:

1: PFOA



2: 13C-PFOA



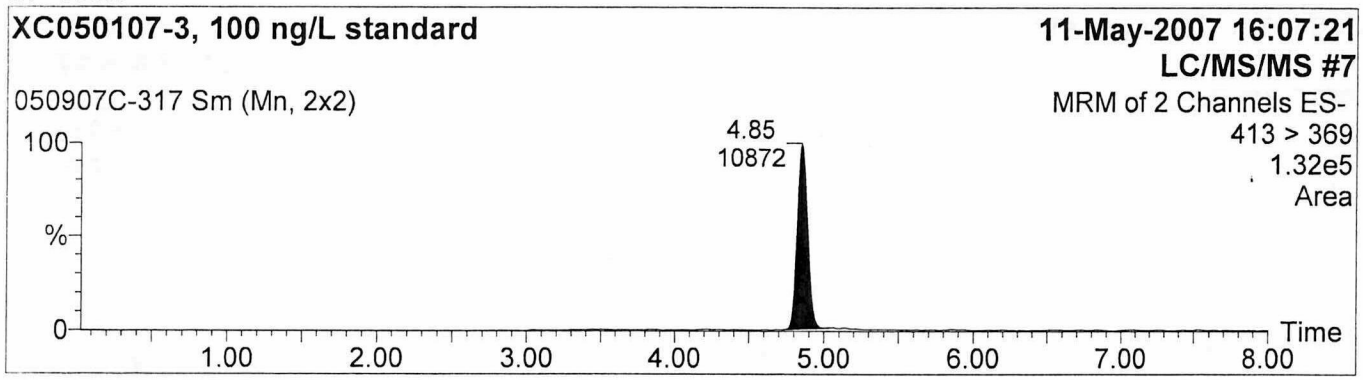
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

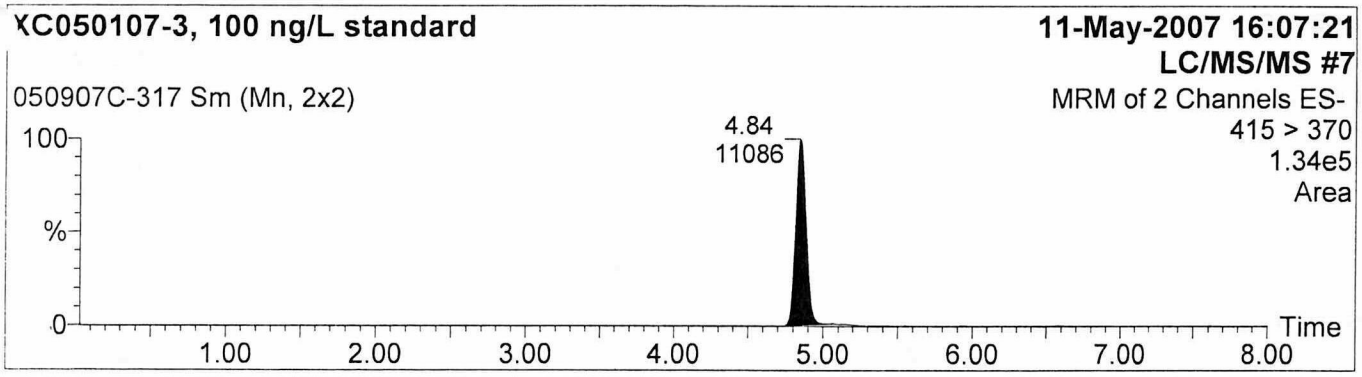
Printed: Tue May 15 10:27:53 2007

Name: 050907C-317
Text:

1: PFOA



2: 13C-PFOA



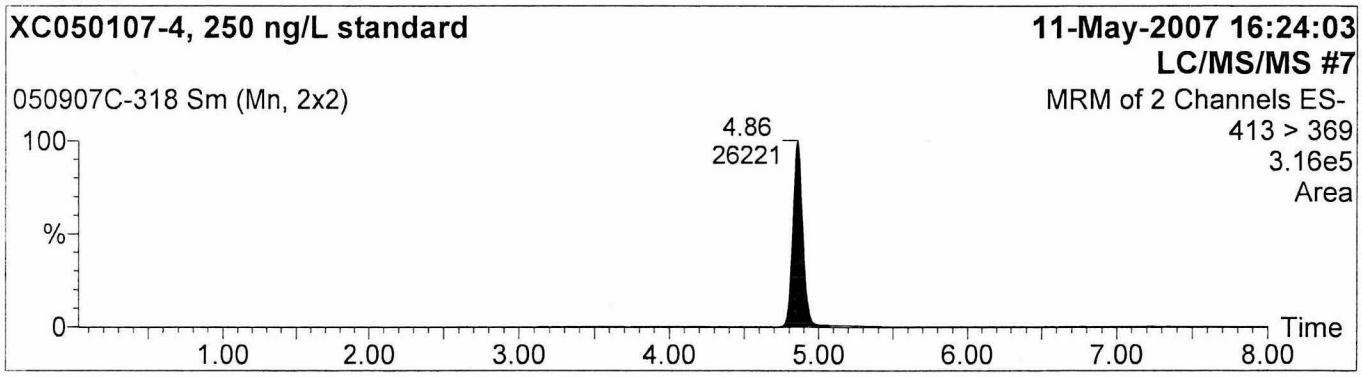
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

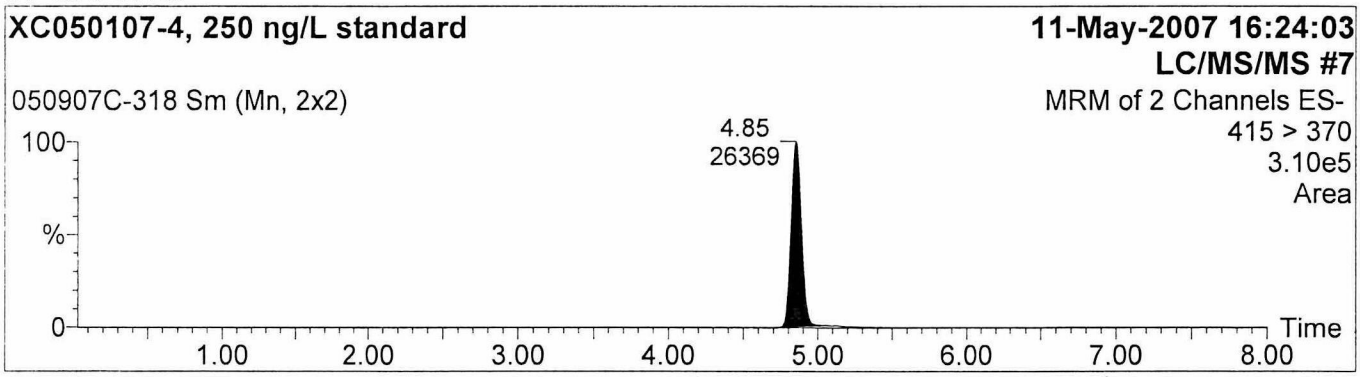
Printed: Tue May 15 10:27:53 2007

Name: 050907C-318
Text:

1: PFOA



2: 13C-PFOA



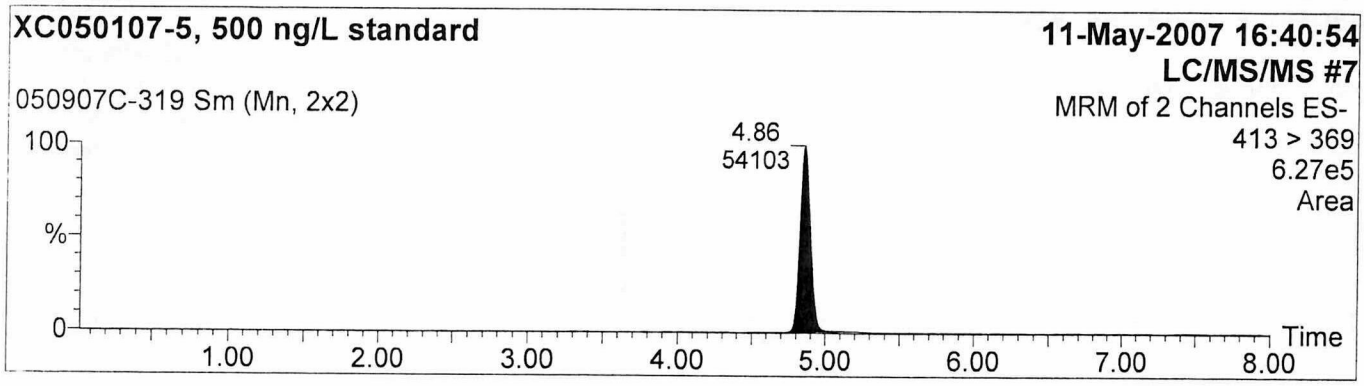
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

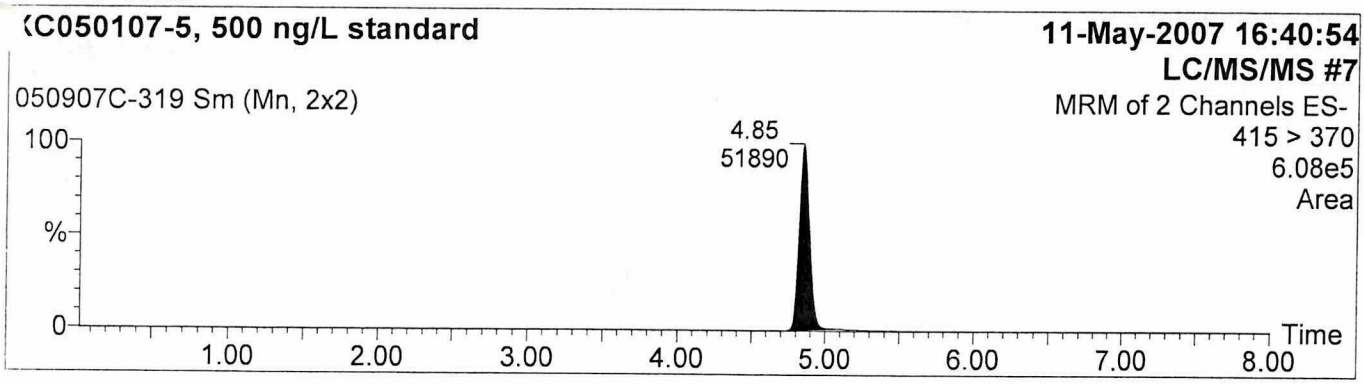
Printed: Tue May 15 10:27:53 2007

Name: 050907C-319
Text:

1: PFOA



2: 13C-PFOA



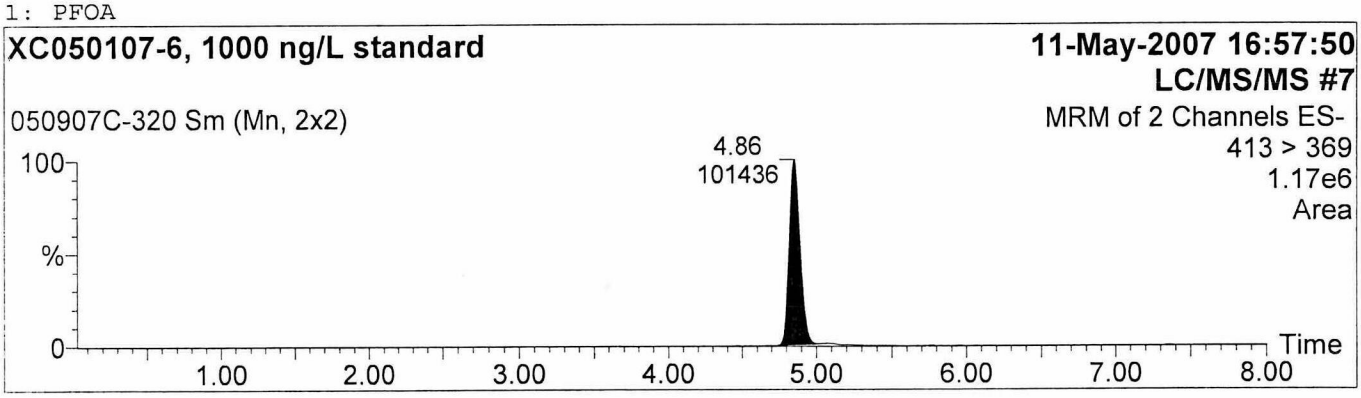
Study No: L11423, Set No: 050907C. Ext.Date: 05/02&09/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050907C Buffer
Last modified: Tue May 15 10:24:03 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

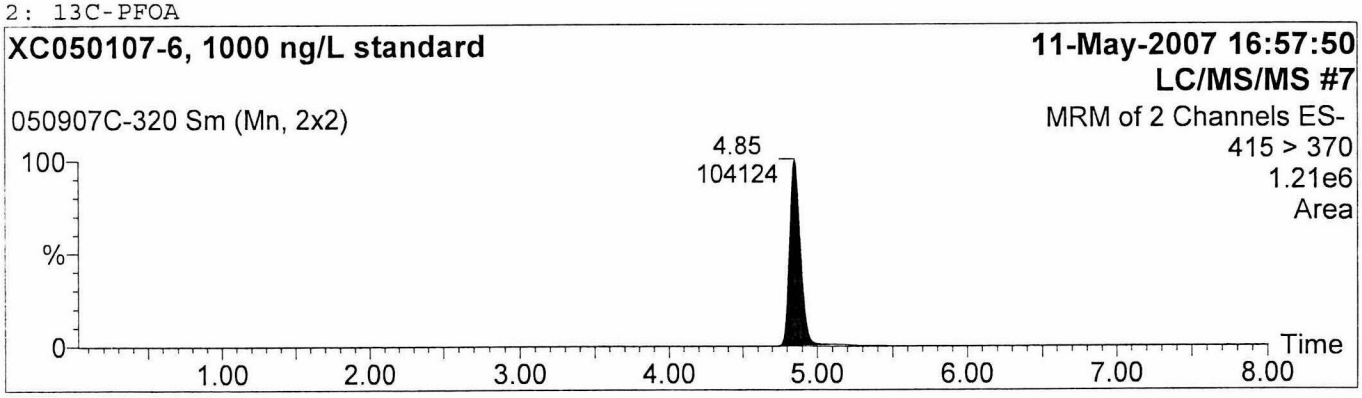
Printed: Tue May 15 10:27:53 2007

Name: 050907C-320
Text:

1: PFOA



2: 13C-PFOA



RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050207B
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	Methanol/Filter Rinse	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	4.0 mL	Extraction Date:	05/02/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/03/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050207B-201	0	-	-	0	-	-	-	-
XC050107-1	-	CS	050207B-202	25	-	-	2755	-	-	-	-
XC050107-2	-	CS	050207B-203	50	-	-	4947	-	-	-	-
XC050107-3	-	CS	050207B-204	100	-	-	10383	-	-	-	-
XC050107-4	-	CS	050207B-205	250	-	-	23678	-	-	-	-
XC050107-5	-	CS	050207B-206	500	-	-	47488	-	-	-	-
XC050107-6	-	CS	050207B-207	1000	-	-	90666	-	-	-	-
Methanol Wash	-	C	050207B-208	-	-	-	262	-	-	-	-
Reagent Control	NA	C	050207B-209	-	-	1	0	ND	ND	-	-
Reagent Spk A	NA	LCS	050207B-210	-	-	1	5503	58.0	60.3	50	116
Reagent Spk B	NA	LCS	050207B-211	-	-	1	42288	450	468	500	90
L11423-20 Spk C	SG-A-020	LF	050207B-212	-	390	10000	35744	^	-	5000000	-
L11423-20 Spk C	SG-A-020	LF	050207B-213	-	390	10	11878364	*	-	5000000	-
Methanol Wash	-	C	050207B-214	-	-	-	0	-	-	-	-
XC050107-1	-	CS	050207B-215	25	-	-	2127	-	-	-	-
XC050107-2	-	CS	050207B-216	50	-	-	3910	-	-	-	-
L11423-4	SG-A-004	S	050207B-217	-	100	10	209	ND	ND	-	-
L11423-8	SG-A-008	S	050207B-218	-	100	10	7963	842	876	-	-
L11423-12	SG-A-012	S	050207B-219	-	310	10	149428	**	-	-	-
L11423-16	SG-A-016	S	050207B-220	-	350	10	115459	**	-	-	-
L11423-20	SG-A-020	S	050207B-221	-	390	10	117439	**	-	-	-
L11423-20 Rep	SG-A-020	S	050207B-222	-	390	10	124077	**	-	-	-
XC050107-3	-	CS	050207B-223	100	-	-	9542	-	-	-	-
XC050107-4	-	CS	050207B-224	250	-	-	23221	-	-	-	-
XC050107-5	-	CS	050207B-225	500	-	-	47684	-	-	-	-
XC050107-6	-	CS	050207B-226	1000	-	-	95895	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 59.4780
Slope = 93.9170
Coef. Of Det. = 0.998604

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.
**Sample requires dilution. See data set 050407D.
^Sample requires a lower spiking level. For re-extraction and analysis see data set 050407D.

Spreadsheet prepared by: MLC / 5/3/07

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050207B
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	^{13}C-PFOA	Matrix:	Methanol/Filter Rinse	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	415 -> 370	Sample Volume Extracted:	4.0 mL	Extraction Date:	05/02/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/03/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	13C-PFOA Found (ng/L)	Amount 13C-PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050207B-201	0	-	-	0	-	-	-
XC050107-1	-	CS	050207B-202	25	-	-	2545	-	-	-
XC050107-2	-	CS	050207B-203	50	-	-	5127	-	-	-
XC050107-3	-	CS	050207B-204	100	-	-	9935	-	-	-
XC050107-4	-	CS	050207B-205	250	-	-	23935	-	-	-
XC050107-5	-	CS	050207B-206	500	-	-	47611	-	-	-
XC050107-6	-	CS	050207B-207	1000	-	-	91126	-	-	-
Methanol Wash	-	C	050207B-208	-	-	-	0	-	-	-
Reagent Control	NA	C	050207B-209	-	-	1	0	ND	-	-
Reagent Spk A	NA	LCS	050207B-210	-	-	1	4661	50.1	50	100
Reagent Spk B	NA	LCS	050207B-211	-	-	1	47035	505	500	101
L11423-20 Spk C	SG-A-020	LF	050207B-212	-	390	10000	67	*	1280	-
L11423-20 Spk C	SG-A-020	LF	050207B-213	-	390	10	16300	1750	1280	137
Methanol Wash	-	C	050207B-214	-	-	-	0	-	-	-
XC050107-1	-	CS	050207B-215	25	-	-	2076	-	-	-
XC050107-2	-	CS	050207B-216	50	-	-	3828	-	-	-
L11423-4	SG-A-004	S	050207B-217	-	100	10	41778	4490	5000	90
L4376-8	SG-A-008	S	050207B-218	-	100	10	39312	4220	5000	84
L11423-12	SG-A-012	S	050207B-219	-	310	10	13912	1490	1610	93
L11423-16	SG-A-016	S	050207B-220	-	350	10	12356	1330	1430	93
L11423-20	SG-A-020	S	050207B-221	-	390	10	11462	1230	1280	96
L11423-20 Rep	SG-A-020	S	050207B-222	-	390	10	11765	1260	1280	98
XC050107-3	-	CS	050207B-223	100	-	-	9244	-	-	-
XC050107-4	-	CS	050207B-224	250	-	-	22904	-	-	-
XC050107-5	-	CS	050207B-225	500	-	-	47755	-	-	-
XC050107-6	-	CS	050207B-226	1000	-	-	92420	-	-	-

13C-PFOA Found (ng/L) = (peak area - intercept) / slope x DF

Recovery (%) = $\frac{\text{13C-PFOA found (ng/L)}}{\text{amount 13C-PFOA added (ng/L)}} \times 100$

Standard Curve : Linear (1/x weighted)
Intercept = -0.662820
Slope = 93.1207
Coef. Of Det. = 0.999252

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.

Spreadsheet prepared by: MLC / 5/3/07

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

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423 Matrix: MeOH Rinse Impinger Sample
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 34
Time 8:25 AM Date 5/02/2007 Initials EDD

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-A-020	L11423-20* Spk C	390 mL	40.0	20000.0
SG-A-004	L11423-4*	100 ml	40.0	-
SG-A-008	L11423-8*	100 mL	40.0	-
SG-A-012	L1142312*	310 mL	40.0	-
SG-A-016	L11423-16*	350 mL	40.0	-
SG-A-020	L11423-20*	390 mL	40.0	-
SG-A-020	L11423-20 Rep*	390 mL	40.0	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
L11423-20* Spk C	SS20794 (100000 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-

All samples were measured:
Time 10:25 AM Date 5/02/2007 Initials EDD

After measuring samples were returned to refrigerator No. 32
Time 3:30 PM Date 5/02/2007 Initials EDD

Comments: *Sample is pre-diluted ten times by taking 4 mL of sample and bringing the volume up to 40 mL with HPLC water

Analysis Summary:
Data Set: 050207B Initials/Date: MLC / 5/2/07
Data Set: 050407D Initials/Date: MLC / 5/4/07
Data Set: - Initials/Date: - / -

Set extraction/analysis data verified by: CEE Date: 05/24/07

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

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007C[$$] PROTOCOL NUMBER: NA

MATRIX: MeOH Rinse Impinger Sample ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	Reagent Control			-		-	-	Methanol	46303
na	Reagent Spk A			-		-	-	C18 SPE	053387081B
na	Reagent Spk B			-		-	-	HPLC Water	46335
SG-A-020	L11423-20* Spk C			(1) 0.1/10, 0.1/1.0		-	-	-	-
SG-A-004	L11423-4*			-		-	-	-	-
SG-A-008	L11423-8*			-		-	-	-	-
SG-A-012	L1142312*			-		0.1/10		-	-
SG-A-016	L11423-16*			-		0.1/10		-	-
SG-A-020	L11423-20*			-		0.1/10		-	-
SG-A-020	L11423-20 Rep*	✓	✓	-	✓	0.1/10	✓	*Initials/Date*	EDD 5/2/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C441404
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date	-	EDD 5/2/07	EDD 5/2/07	MLC 5/2/07	MLC 5/2/07	MLC 5/4/07	MLC 5/4/07	*Initials/Date*	MLC 5/2/07

(1) DF = 1000 MLC 5/2/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

*Sample was diluted ten times prior to extraction.
**Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:

Final extracts stored in refrigerator 32 Initials: EDD Date: 5/2/2007

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

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Exygen STUDY NO. L11423

Protocol No.: None
Method No.: $$

Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA

Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC050107-0	Methanol	46303
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC050107-1	C18 SPE	0053370818
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC050107-2	Water	46335
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC050107-3	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC050107-4	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC050107-5	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC050107-6	-	-
-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/01/07

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) freezer # 34 Time: 9:10 AM — Initials/Date: EDD / 5/01/07

40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD / 5/01/07

After measuring, samples returned to (refrigerator) freezer # 34 Time: 11:10 AM — Initials/Date: EDD / 5/01/07

Samples fortified: — Initials/Date: AMB / 05/01/07

SPE clean-up: — Initials/Date: EDD / 5/01/07

Final volume adjusted to 5 mL: — Initials/Date: EDD / 5/01/07

Extracts placed in refrigerator # 32 — Initials/Date: EDD / 5/01/07

Comments:

STANDARD EXPIRATION DATE: 5/15/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/4/07

Masslynx - Sample List

Exygen STUDY NO. L11423

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050207B MeOH Rinse.SPL
Printed: Wed May 02 16:01:55 2007 MLC 5/2/07

Page Position: (1, 1)

L (RE) MLC 5/2/07

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/mL)	Conc B	Conc C	Test ID	DF
1	71	050207B-201	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---	0	1
2	72	050207B-202	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
3	73	050207B-203	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
4	74	050207B-204	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
5	75	050207B-205	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
6	76	050207B-206	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
7	77	050207B-207	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1
8	92	050207B-208	---	---	Methanol Wash	---	Blank	---	---	---	0	1
9	33	050207B-209	---	---	Reagent Control	---	Blank	---	---	---	0	1
10	34	050207B-210	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---	0	1
11	35	050207B-211	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500 1280	---	0	1
12	36	050207B-212	---	---	L11423-20 Spk C, 5,000,000 ng/L, DF=10,000	---	QC	5000000	(D) ~~1950~~	---	0	10000
13	37	050207B-213	---	---	L11423-20 Spk C, 5,000,000 ng/L, DF=10	---	QC	5000000	(D) ~~1950~~ 1280	---	0	10
14	92	050207B-214	---	---	Methanol Wash	---	Blank	---	---	---	0	1
15	72	050207B-215	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
16	73	050207B-216	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
17	38	050207B-217	---	---	L11423-4, DF=10	---	Analyte	---	5000	---	0	10
18	39	050207B-218	---	---	L11423-8, DF=10	---	Analyte	---	5000	---	0	10
19	40	050207B-219	---	---	L11423-12, DF=10	---	Analyte	---	(D) ~~1550~~ 1610	---	0	10
20	41	050207B-220	---	---	L11423-16, DF=10	---	Analyte	---	(D) ~~1750~~ 1430	---	0	10
21	42	050207B-221	---	---	L11423-20, DF=10	---	Analyte	---	(D) ~~1950~~ 1280	---	0	10
22	43	050207B-222	---	---	L11423-20 Rep, DF=10	---	Analyte	---	(D) ~~1950~~ 1280	---	0	10
23	74	050207B-223	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
24	75	050207B-224	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
25	76	050207B-225	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
26	77	050207B-226	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1

(D) (RE) MLC 5/2/07

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050207B MeOH Rinse.SPL
Printed: Wed May 02 16:01:55 2007 MLC 5/2/07

Exygen STUDY NO. 411423

	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
20	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
21	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
22	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
23	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
24	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
25	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
26	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA

Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/2/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/3/07

RE MLC 5/3/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Wed May 02 16:02:04 2007 MLC 5/2/07

Solvent Delay (mins) : 0.00

Function : 1 **MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Wednesday, May 02, 2007 15:50:12

Printed: Wednesday, May 02, 2007 16:02:09 MLC 5/2/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

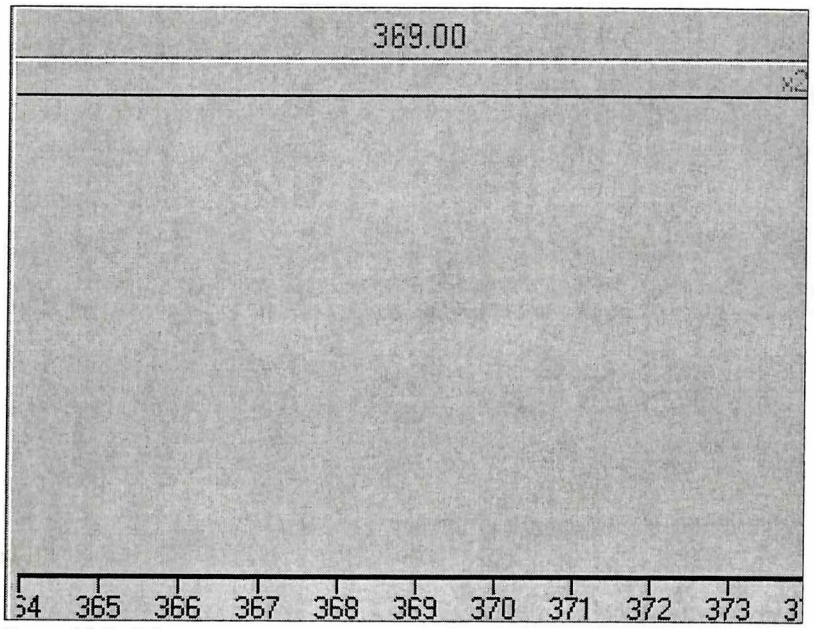
HP1100 Autosampler Initial Conditions

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	92

Tuning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Wed May 02 16:02:27 2007 MLC 5/2/07



Dau 413.00

SOURCE (ESP-)	**Set**	**Rdbk**
Capillary	3.00	-2.94
Cone	10	-10
Hexapole 1	0.0	
Aperture 1	0.0	
Hexapole 2	0.0	
Source Block Temp.	100	102
Desolvation Temp.	300	300

Analyser	**Set**	**Rdbk**
LM Res 1	14.0	
HM Res 1	14.0	
IEnergy 1	1.0	
Entrance	-2	8
Collision	10	9
Exit	2	11
LM Res 2	14.0	
HM Res 2	14.0	
IEnergy 2	2.0	
Multiplier	700	-695

Pressures	**Rdbk**
Analyser Vacuum	OFF
Gas Cell	2.3e-3

Gas Flows	**Rdbk**
Cone Gas	89.7
Desolvation	681.0

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050207B
Extraction Date: 05/02/07
Analyzed on: 05/03/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2755
XC050107-2	50	4947
XC050107-3	100	10383
XC050107-4	250	23678
XC050107-5	500	47488
XC050107-6	1000	90666

Coefficient of Determination: 0.999498311

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999749124
R Square	0.999498311
Adjusted R Square	0.999372888
Standard Error	851.5032075
Observations	6

MLC
5/4/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	5778029528	5778029528	7969.061538	9.44004E-08
Residual	4	2900230.849	725057.7123		
Total	5	5780929759			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	1002.966014	475.6614851	2.108571002	0.102655743	-317.6819876	2323.614016	-317.6819876	2323.614016
X Variable 1	90.33724879	1.011959821	89.2696003	9.44004E-08	87.52759789	93.14689968	87.52759789	93.14689968

Initial Calibration Curve for ^{13}C-PFOA

Exygen Study No.: L11423
Set No: 050207B
Extraction Date: 05/02/07
Analyzed on: 05/03/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2545
XC050107-2	50	5127
XC050107-3	100	9935
XC050107-4	250	23935
XC050107-5	500	47611
XC050107-6	1000	91126

Coefficient of Determination: 0.999550158

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999775054
R Square	0.999550158
Adjusted R Square	0.999437697
Standard Error	811.6858448
Observations	6

MLC
5/4/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	5855718492	5855718492	8888.004088	7.58957E-08
Residual	4	2635335.643	658833.9107		
Total	5	5858353828			

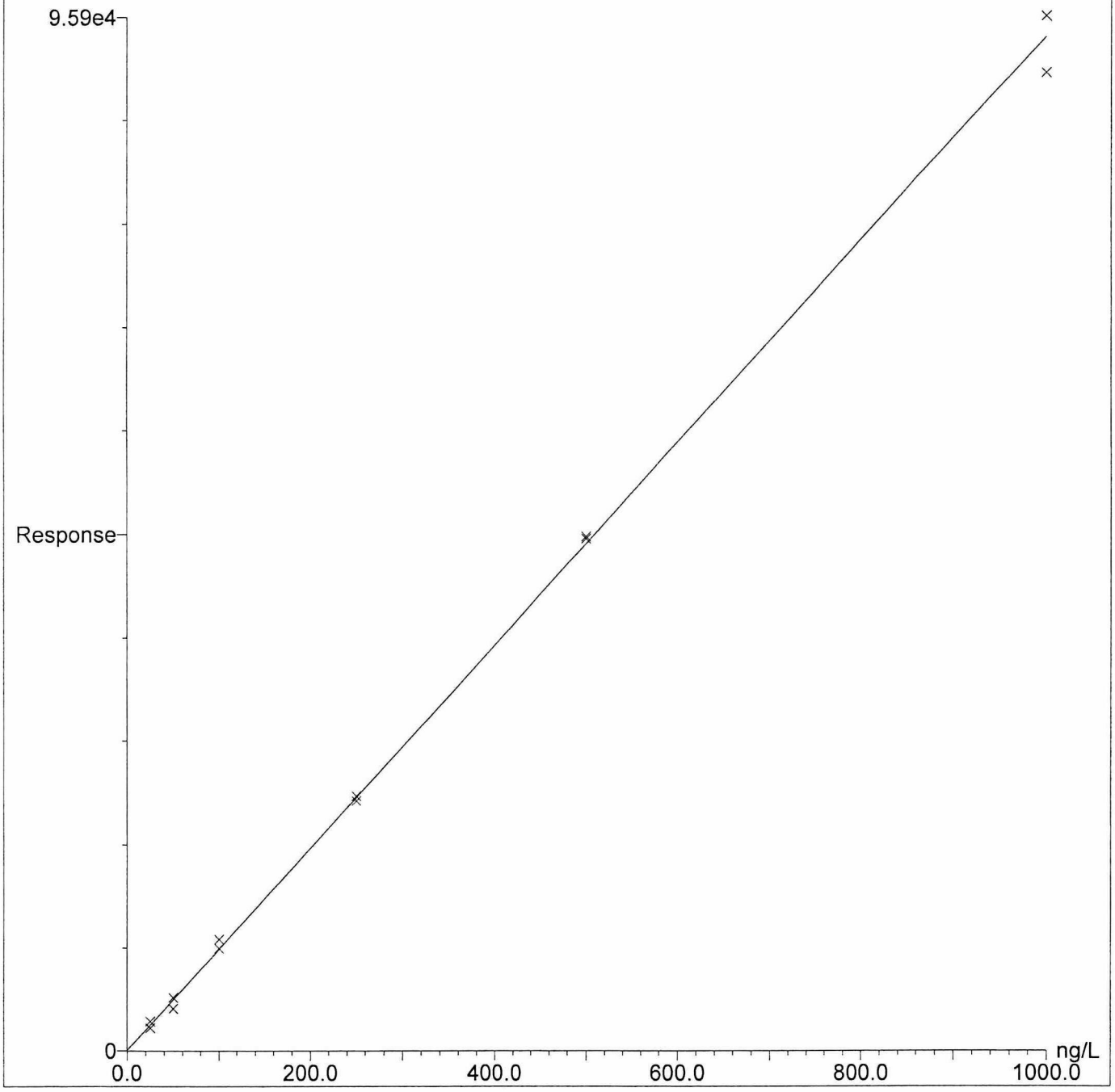
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	869.1019567	453.4189549	1.916774646	0.127745045	-389.7908811	2127.994795	-389.7908811	2127.994795
X Variable 1	90.94253936	0.964639305	94.27621168	7.58957E-08	88.26427128	93.62080743	88.26427128	93.62080743

Quantify Calibration Report
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050207B MeOH Rinse Impinger
Last modified: Thu May 03 15:37:17 2007
Printed: Thu May 03 15:38:53 2007

MLC 5/3/07

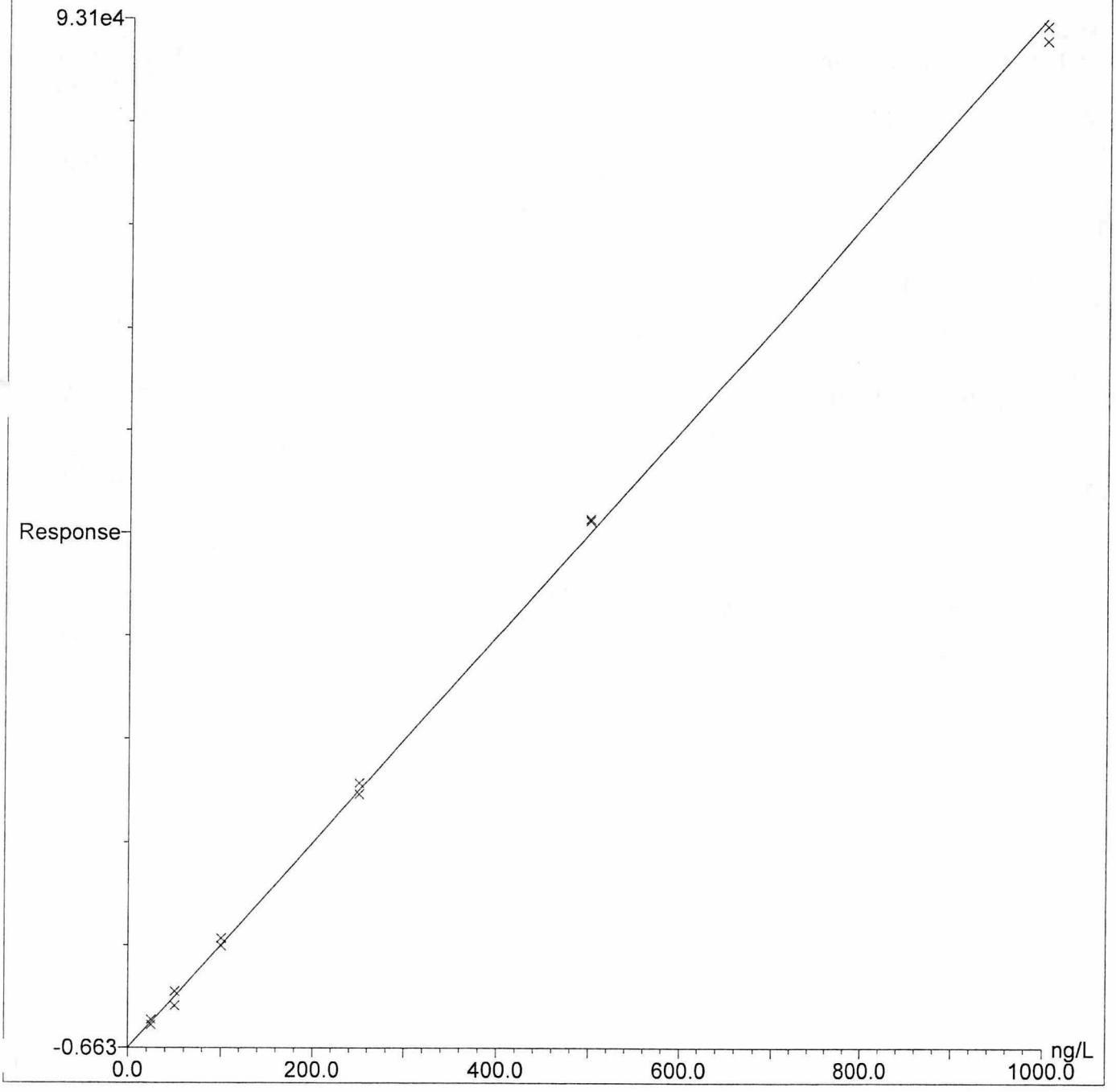
Compound 1 name: PFOA
Coefficient of Determination: 0.998604
Calibration curve: 93.9170 * x + 59.4780
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050207B MeOH Rinse Impinger
Last modified: Thu May 03 15:37:17 2007
Printed: Thu May 03 15:38:53 2007 MLC 5/3/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.999252
Calibration curve: 93.1207 * x + -0.662820
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

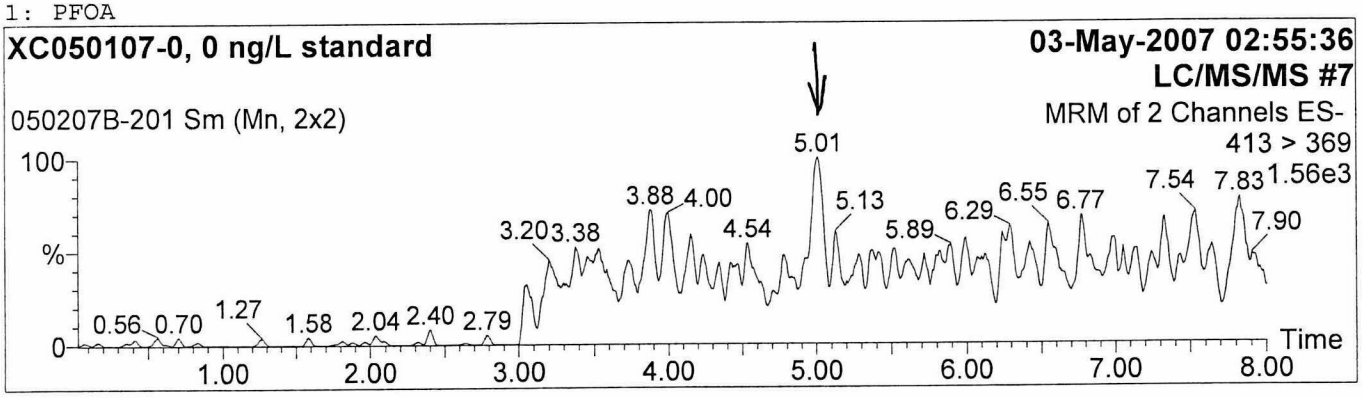
Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

Printed: Thu May 03 15:38:55 2007

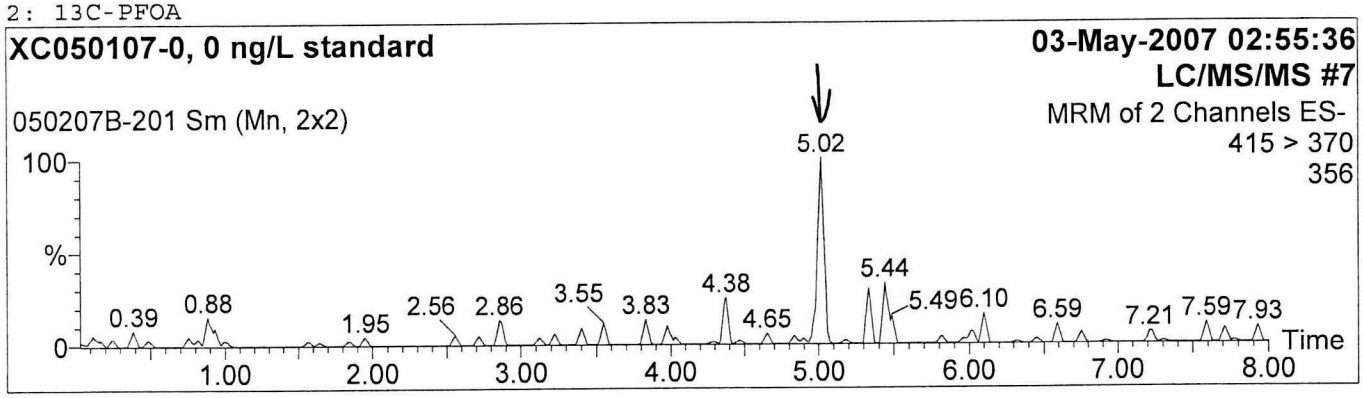
Initials MCC
Date 5/3/07
Run# 050207B-201 To 050207B-226

Name: 050207B-201
Text:

1: PFOA



2: 13C-PFOA



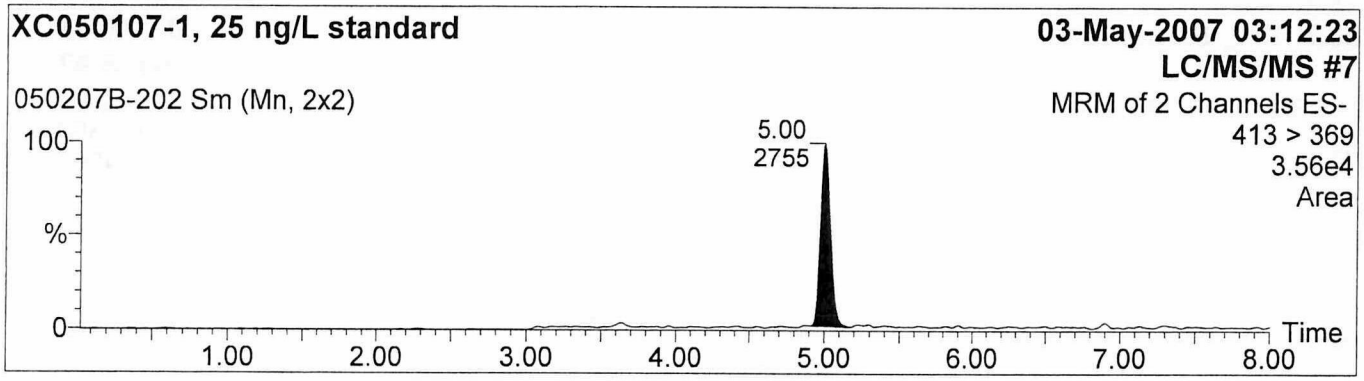
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

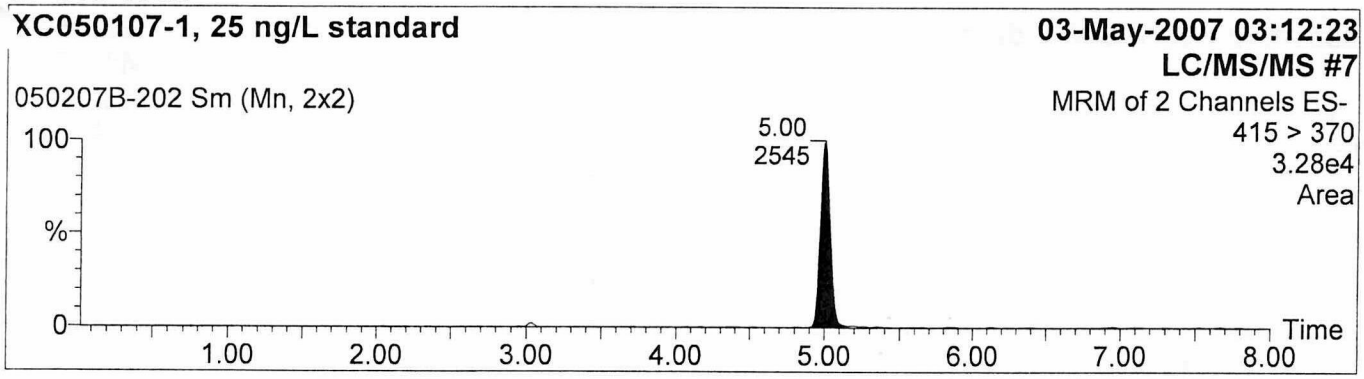
Printed: Thu May 03 15:38:55 2007

Name: 050207B-202
Text:

1: PFOA



2: 13C-PFOA



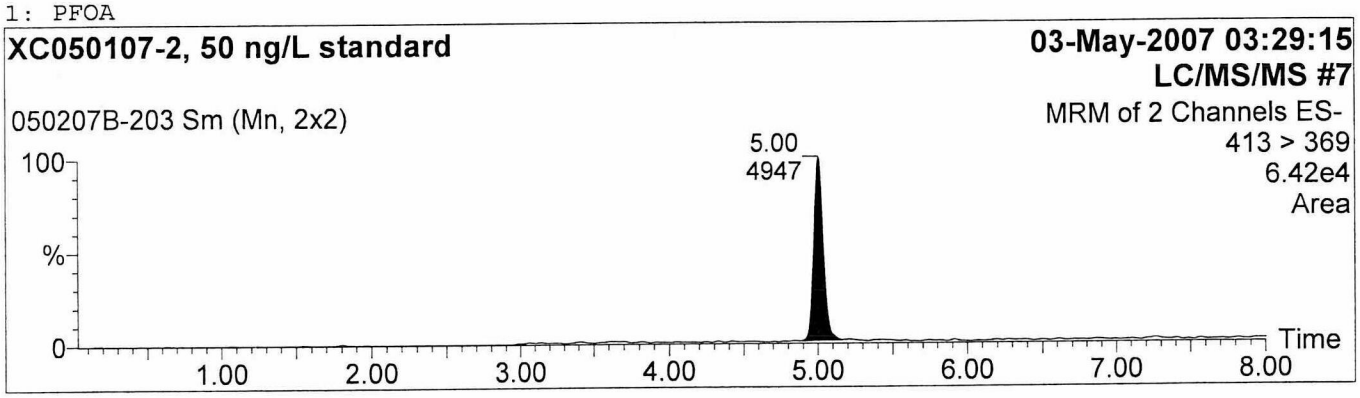
Quantify Sample Report
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

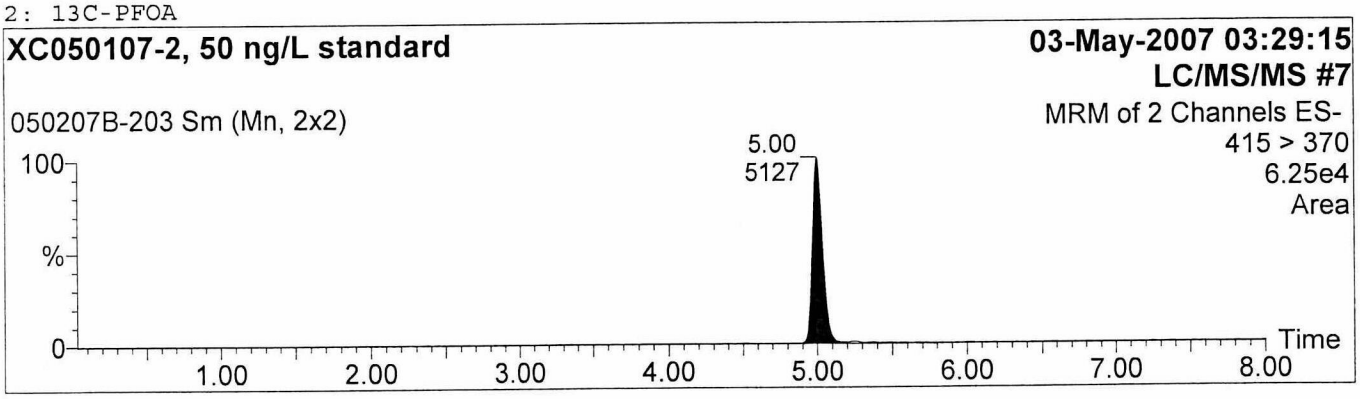
Printed: Thu May 03 15:38:55 2007

Name: 050207B-203
Text:

1: PFOA



2: 13C-PFOA



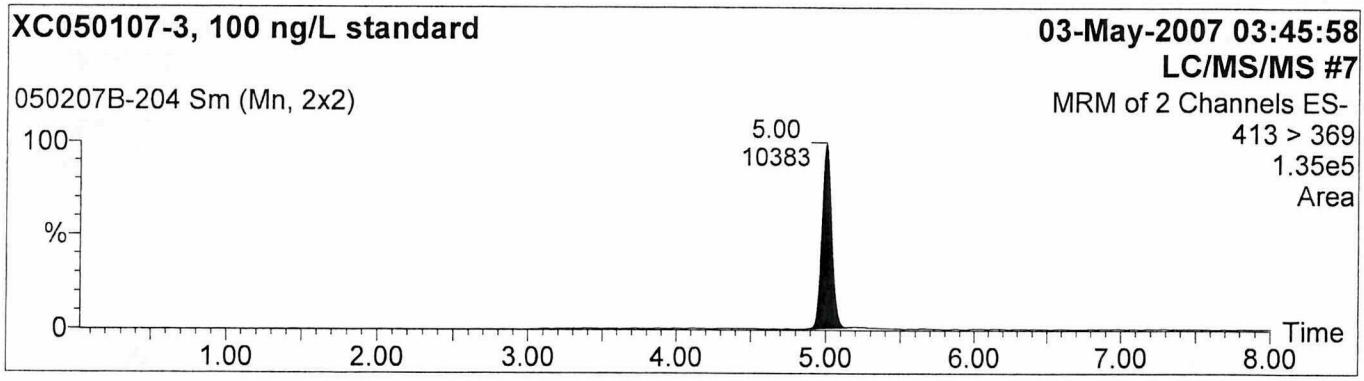
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

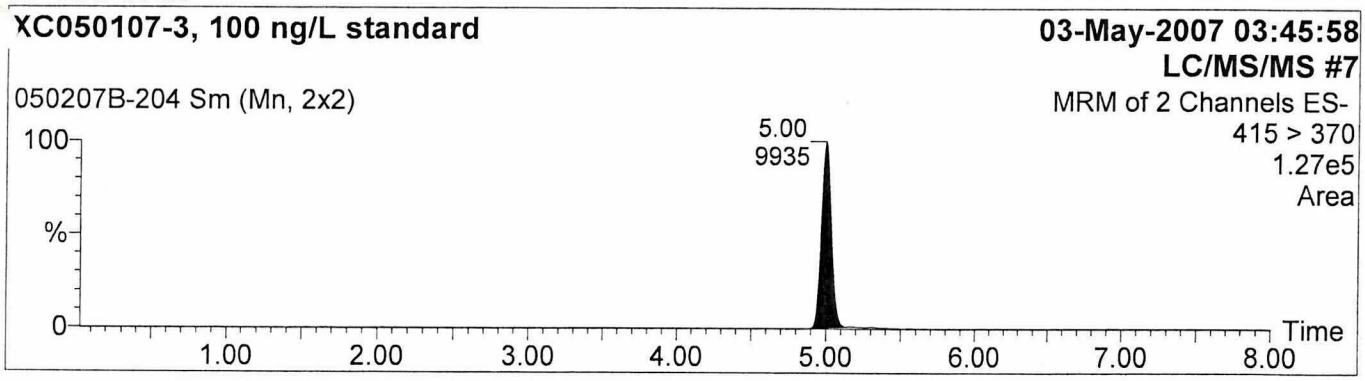
Printed: Thu May 03 15:38:55 2007

Name: 050207B-204
Text:

1: PFOA



2: 13C-PFOA



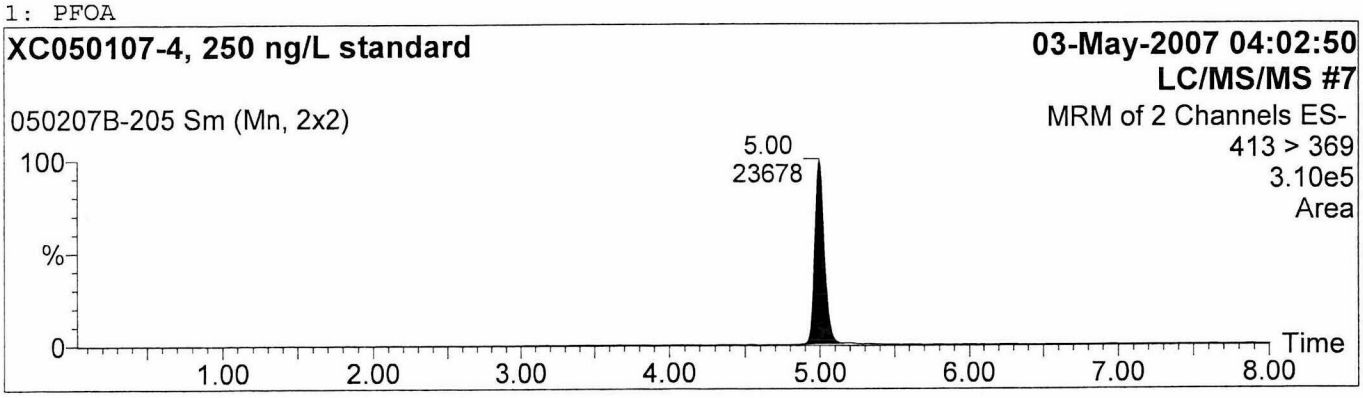
Quantify Sample Report
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

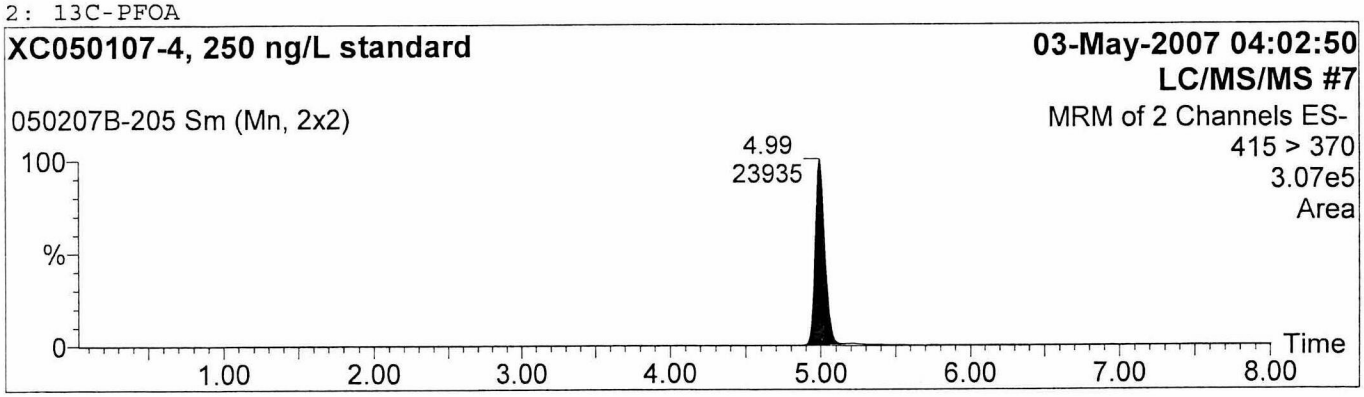
Printed: Thu May 03 15:38:55 2007

Name: 050207B-205
Text:

1: PFOA



2: 13C-PFOA



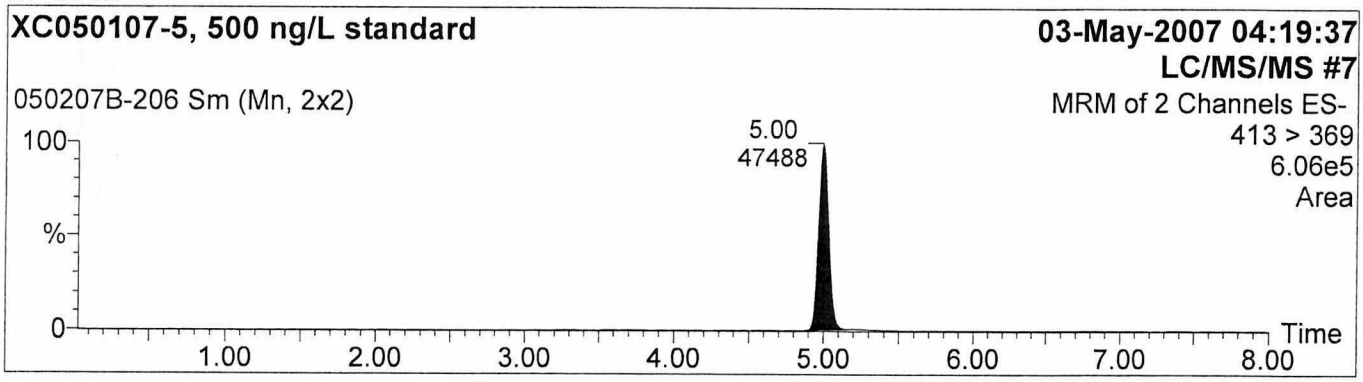
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

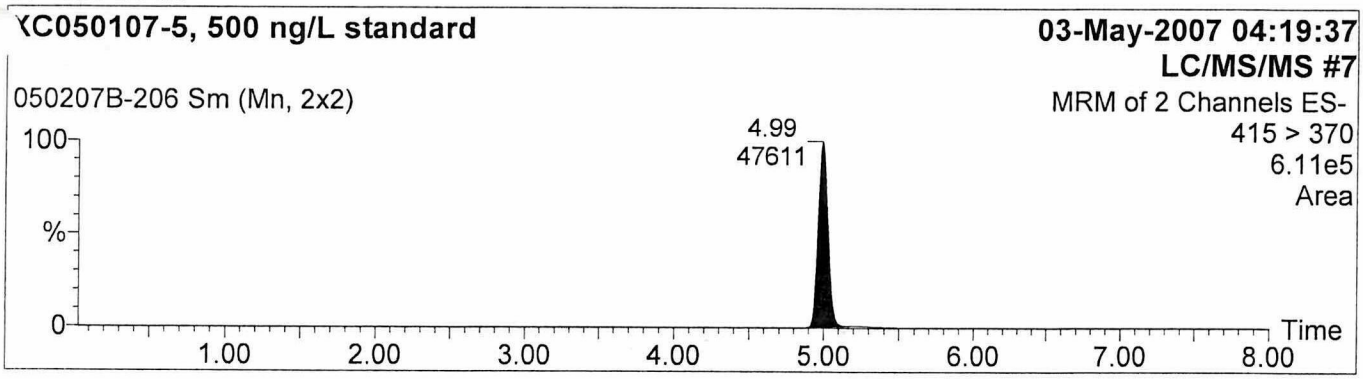
Printed: Thu May 03 15:38:55 2007

Name: 050207B-206
Text:

1: PFOA



2: 13C-PFOA



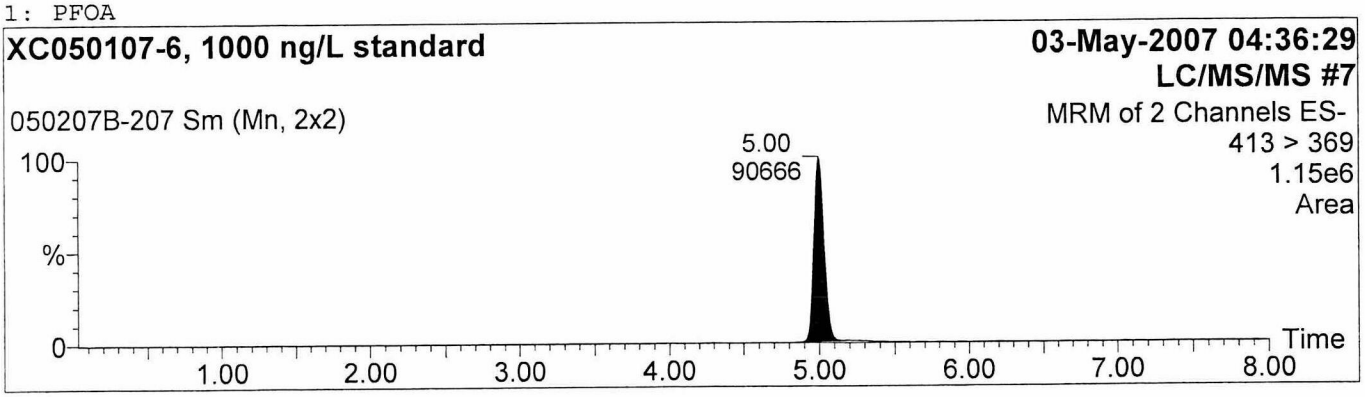
Quantify Sample Report
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

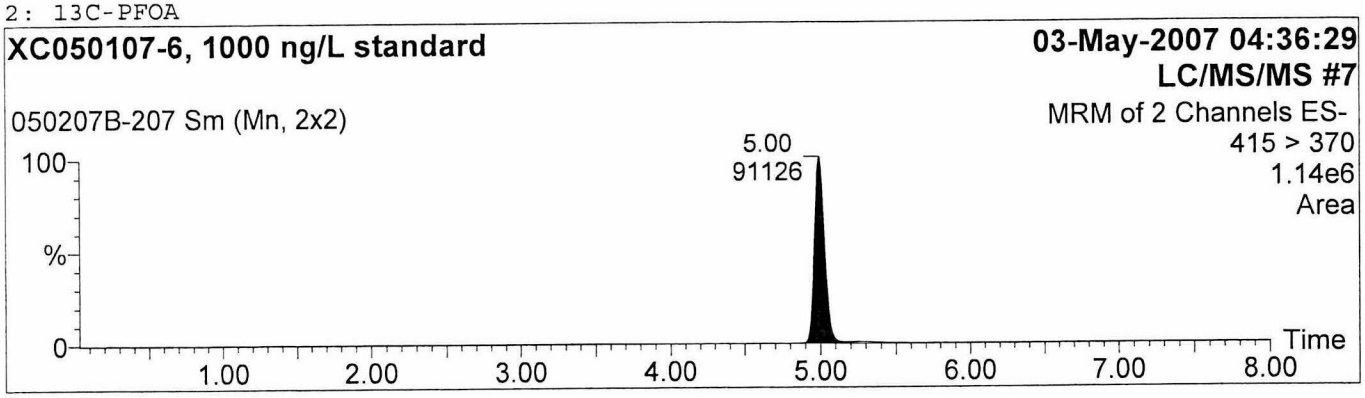
Printed: Thu May 03 15:38:55 2007

Name: 050207B-207
Text:

1: PFOA



2: 13C-PFOA



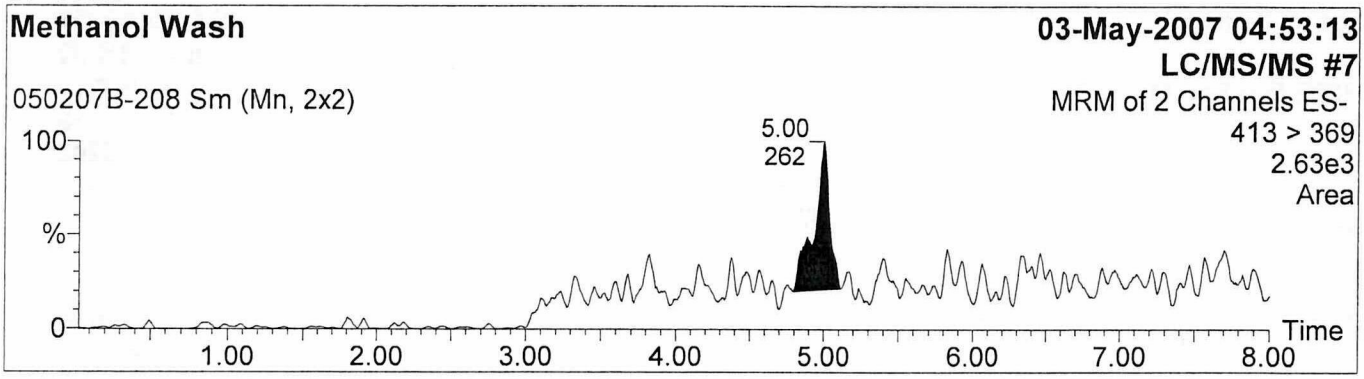
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

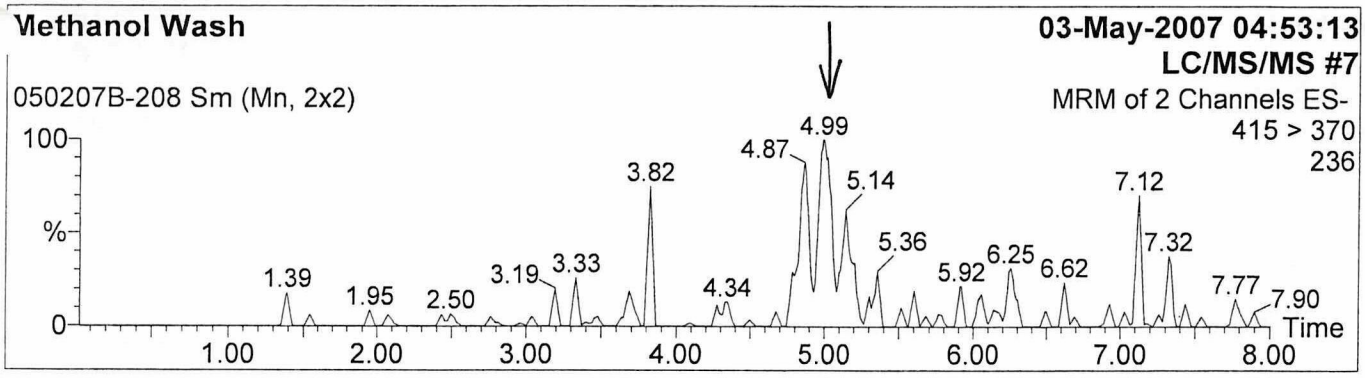
Printed: Thu May 03 15:38:55 2007

Name: 050207B-208
Text:

1: PFOA



2: 13C-PFOA



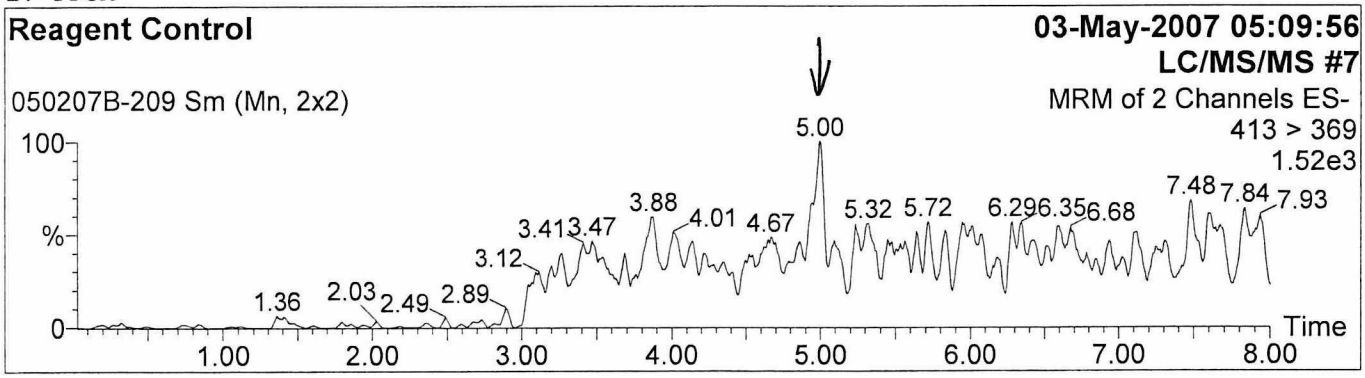
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

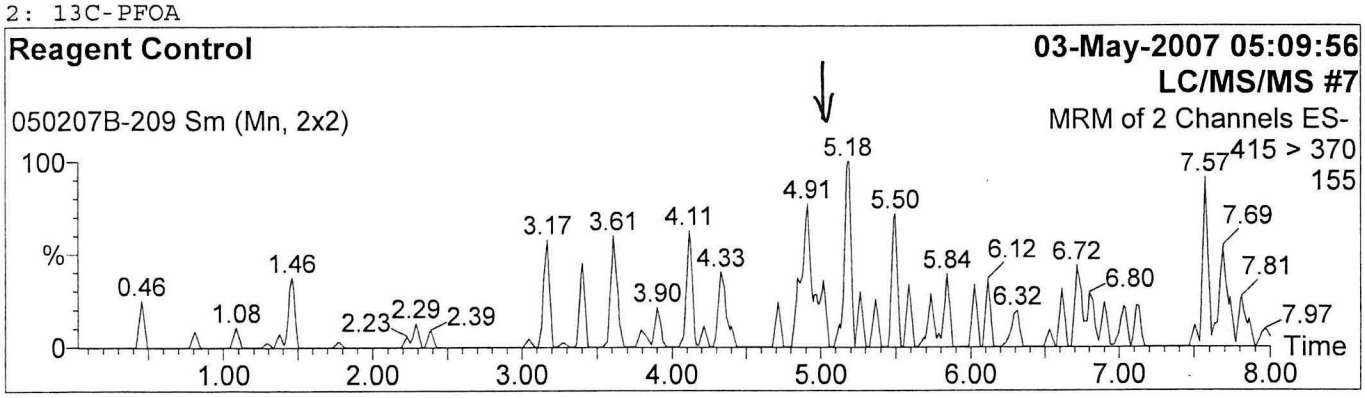
Printed: Thu May 03 15:38:55 2007

Name: 050207B-209
Text:

1: PFOA



2: 13C-PFOA



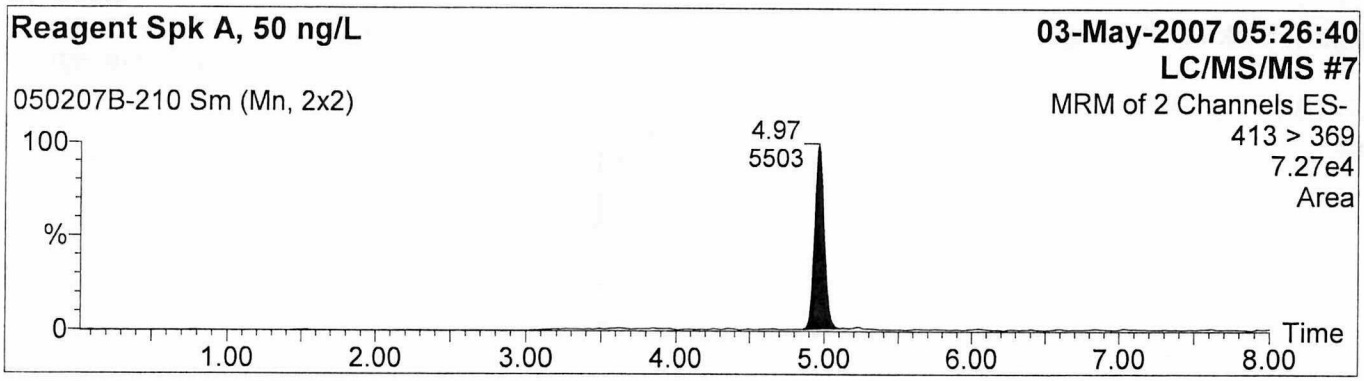
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

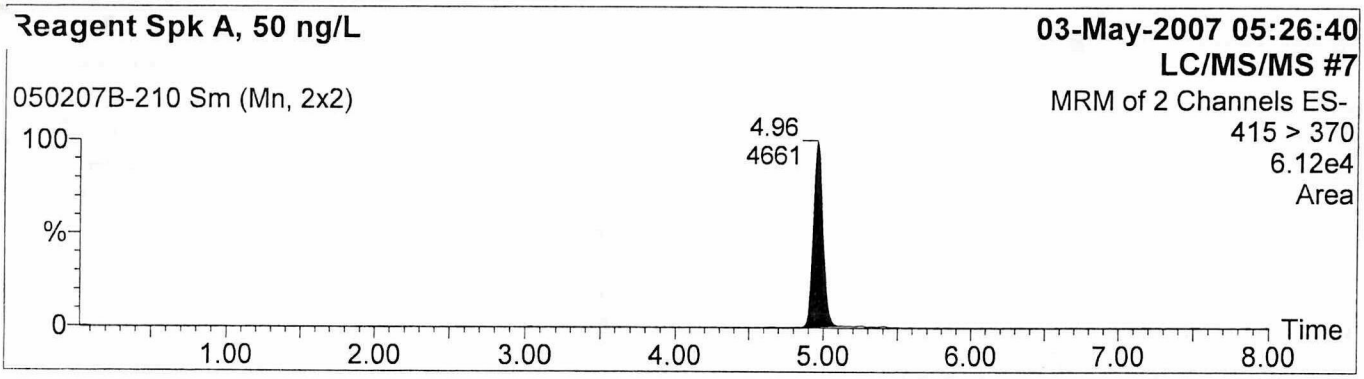
Printed: Thu May 03 15:38:55 2007

Name: 050207B-210
Text:

1: PFOA



2: 13C-PFOA



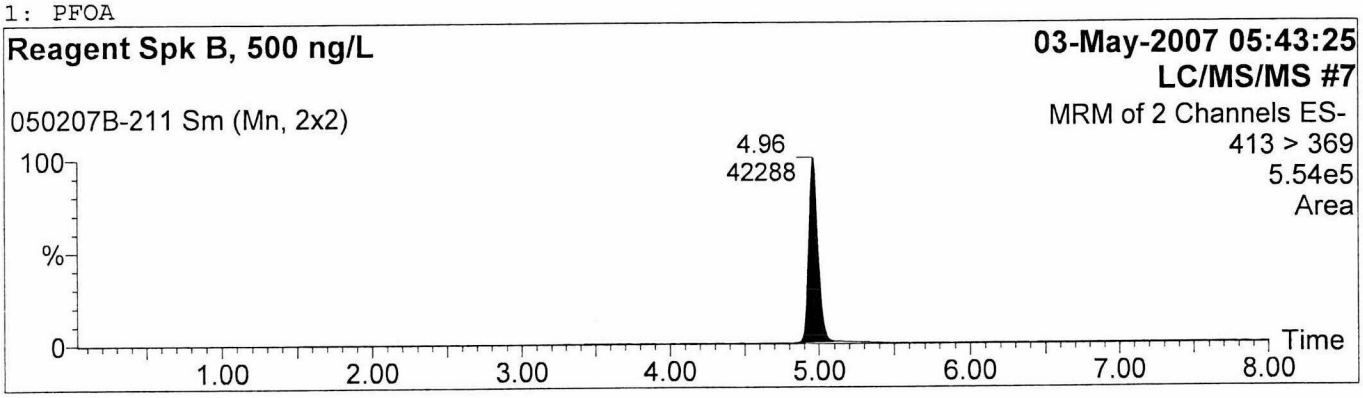
Quantify Sample Report
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

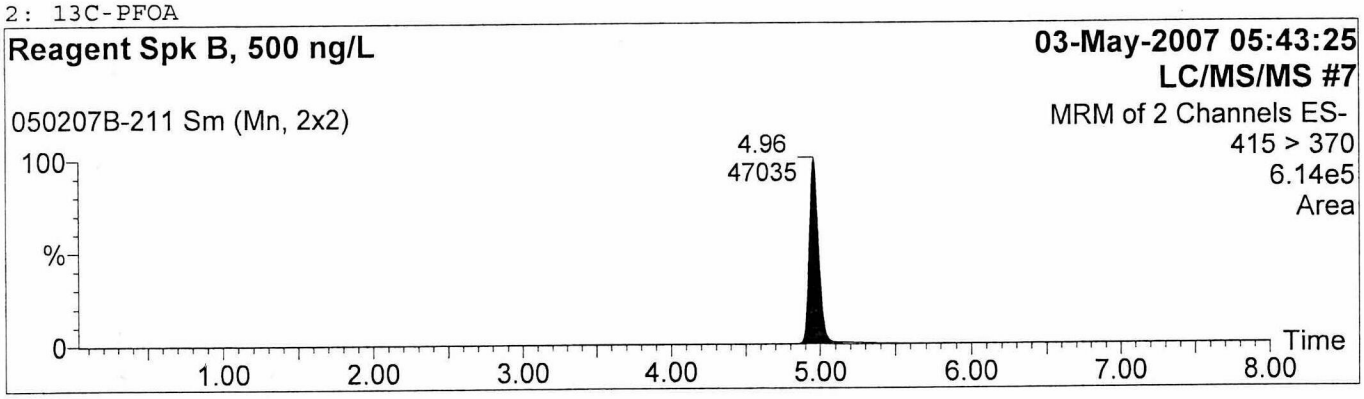
Printed: Thu May 03 15:38:55 2007

Name: 050207B-211
Text:

1: PFOA



2: 13C-PFOA



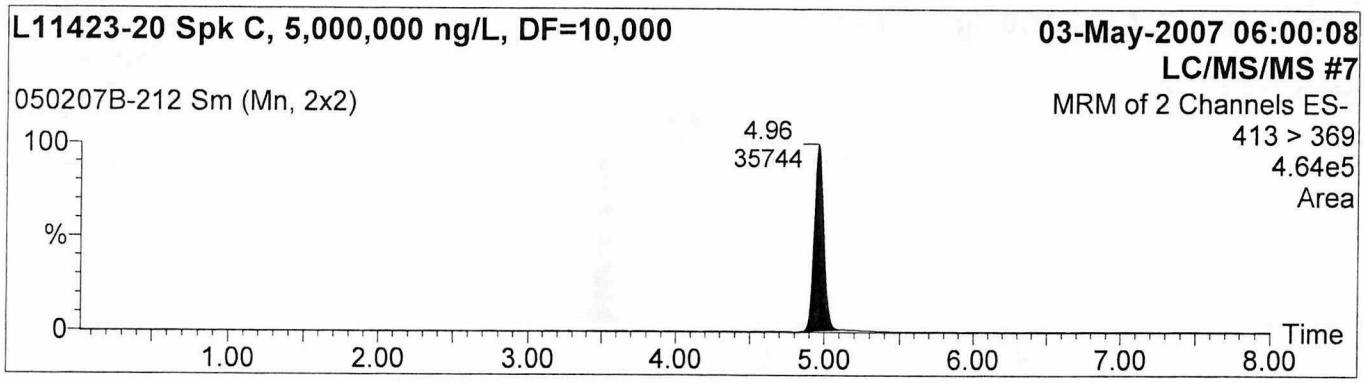
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

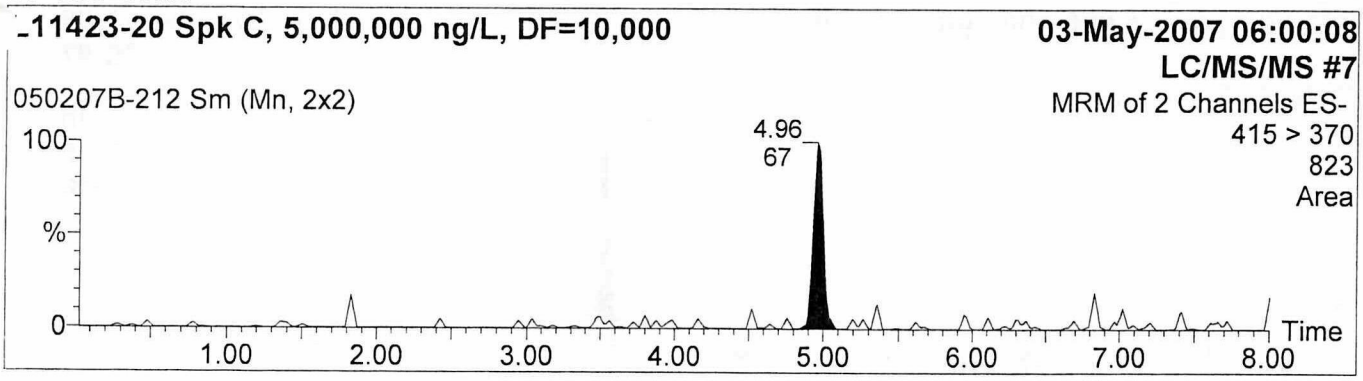
Printed: Thu May 03 15:38:55 2007

Name: 050207B-212
Text:

1: PFOA



2: 13C-PFOA



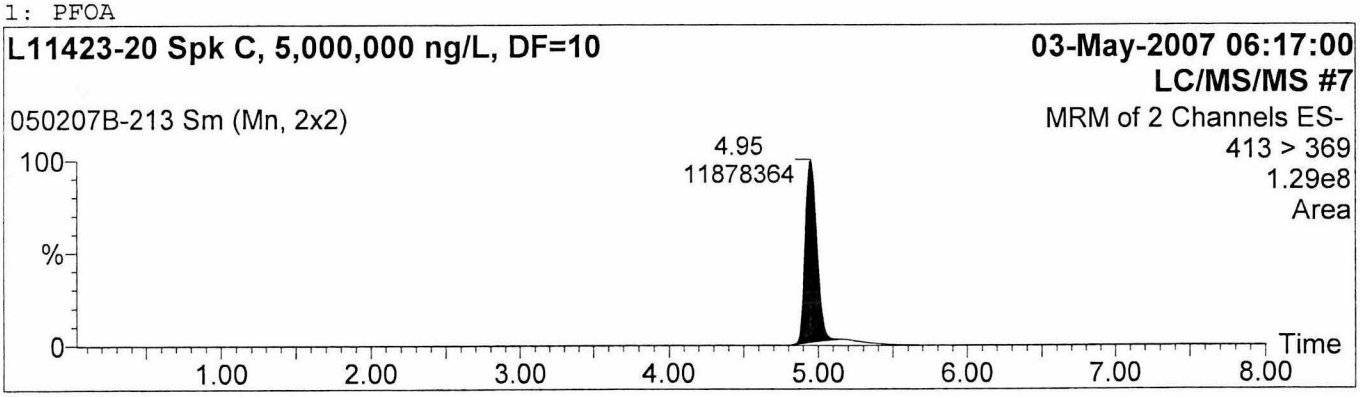
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

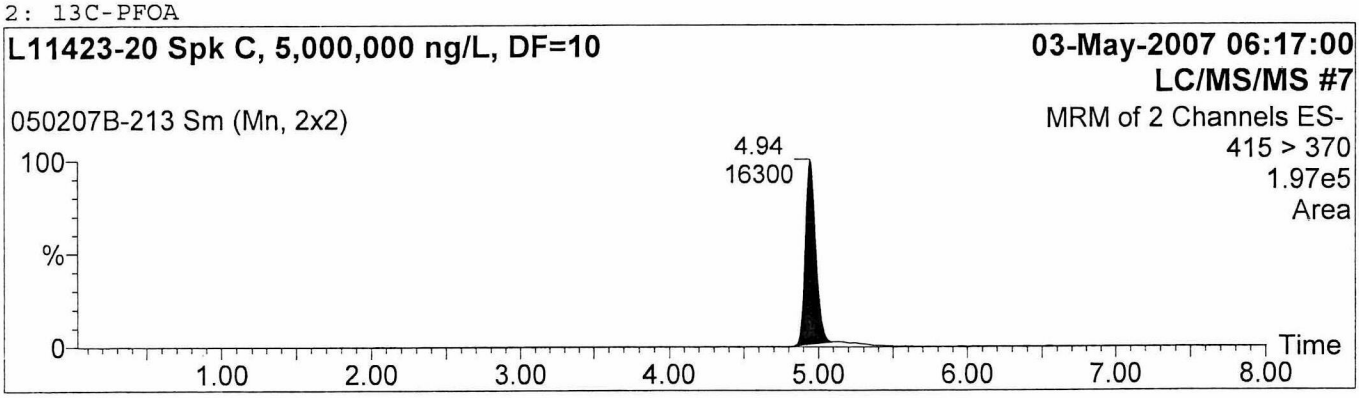
Printed: Thu May 03 15:38:55 2007

Name: 050207B-213
Text:

1: PFOA



2: 13C-PFOA



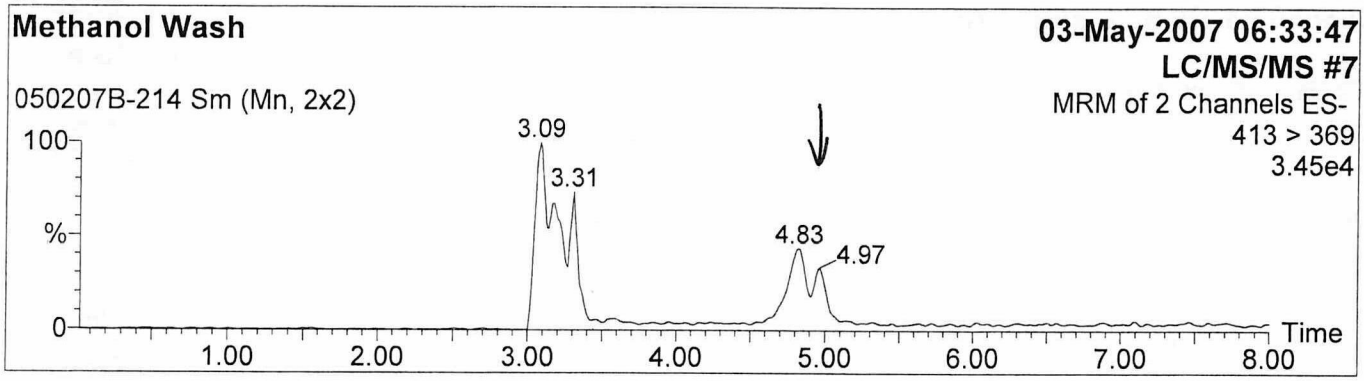
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

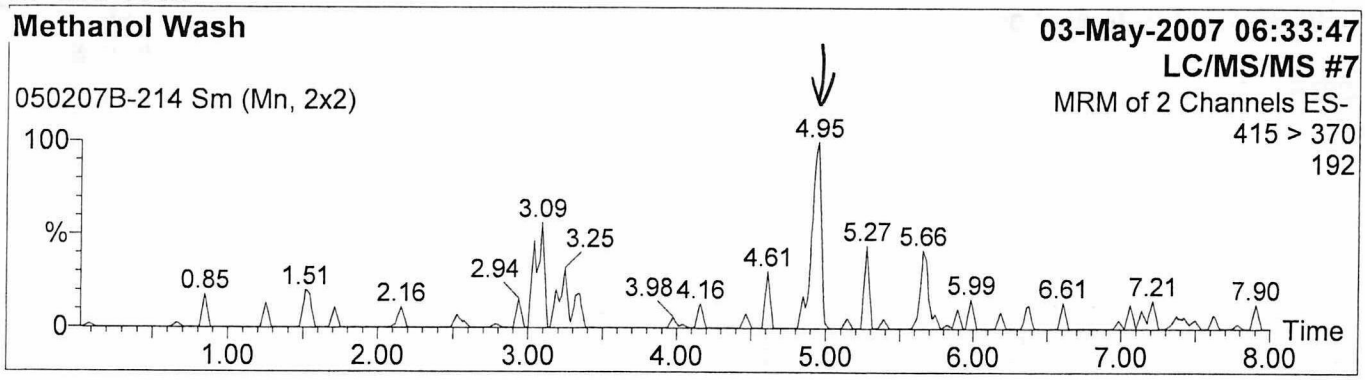
Printed: Thu May 03 15:38:55 2007

Name: 050207B-214
Text:

1: PFOA



2: 13C-PFOA

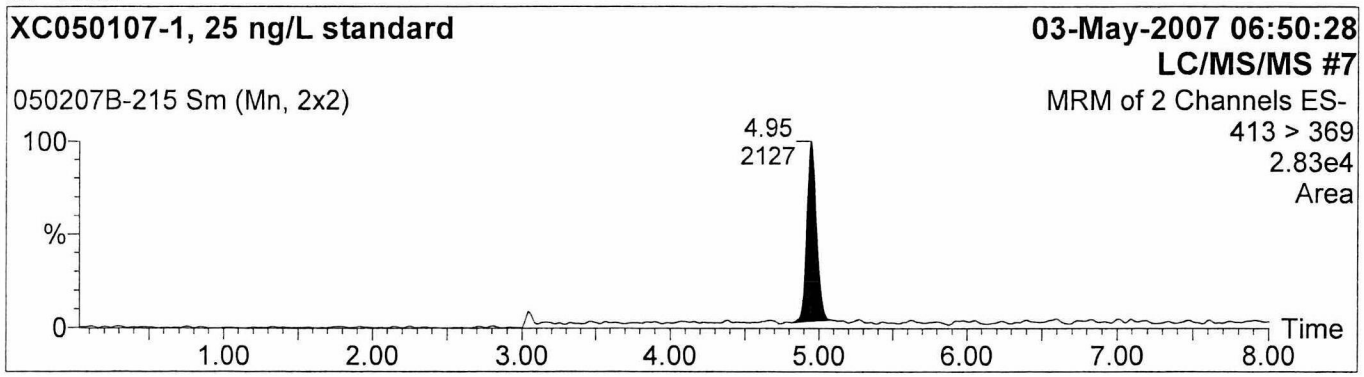


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

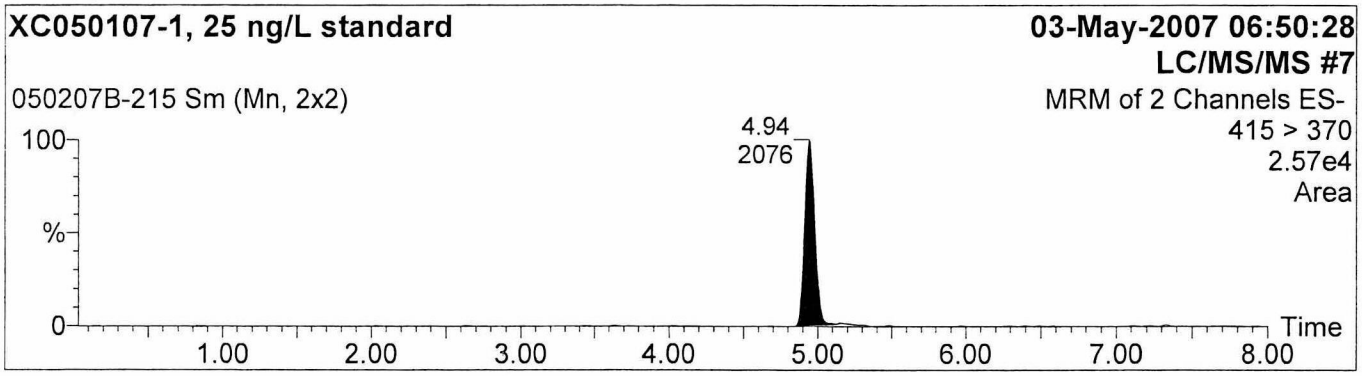
Printed: Thu May 03 15:38:55 2007

Name: 050207B-215
Text:

1: PFOA



2: 13C-PFOA



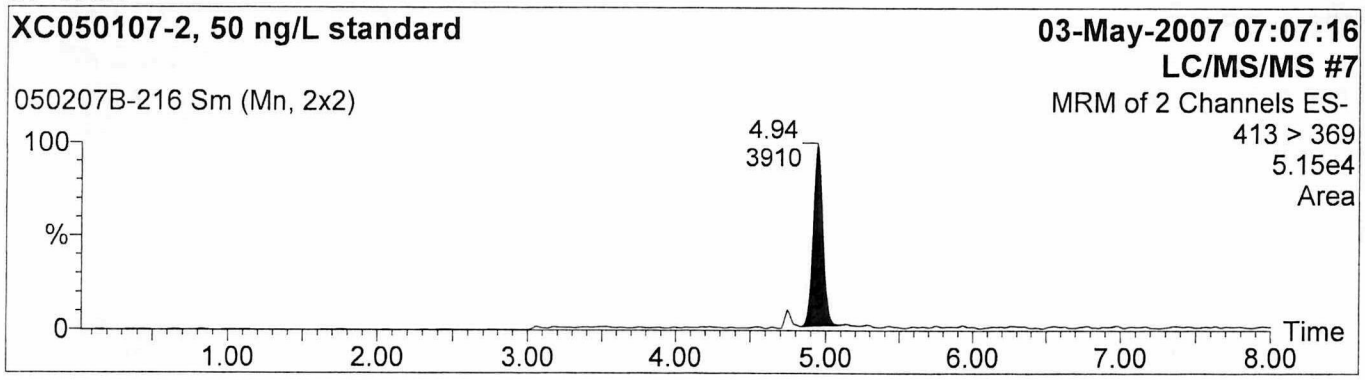
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

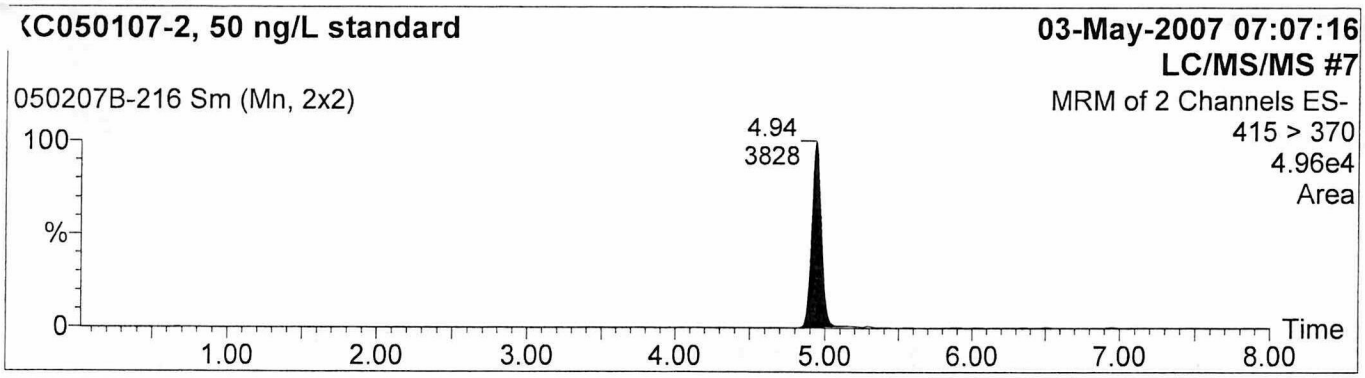
Printed: Thu May 03 15:38:55 2007

Name: 050207B-216
Text:

1: PFOA



2: 13C-PFOA



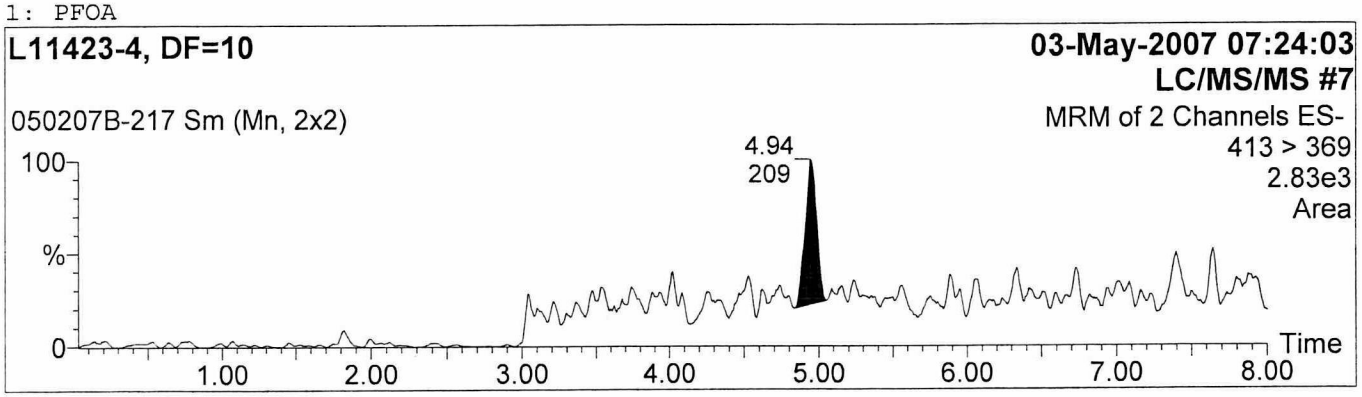
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

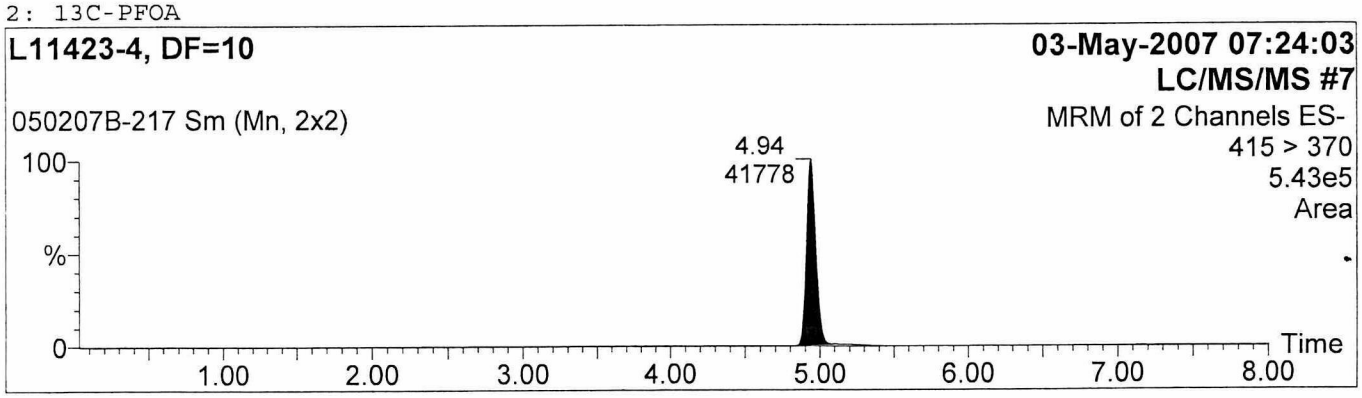
Printed: Thu May 03 15:38:55 2007

Name: 050207B-217
Text:

1: PFOA



2: 13C-PFOA



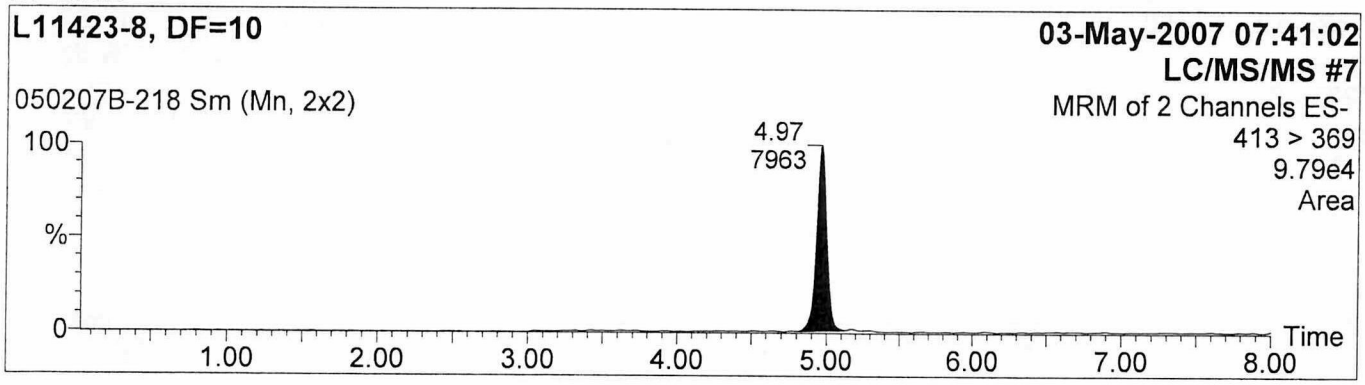
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

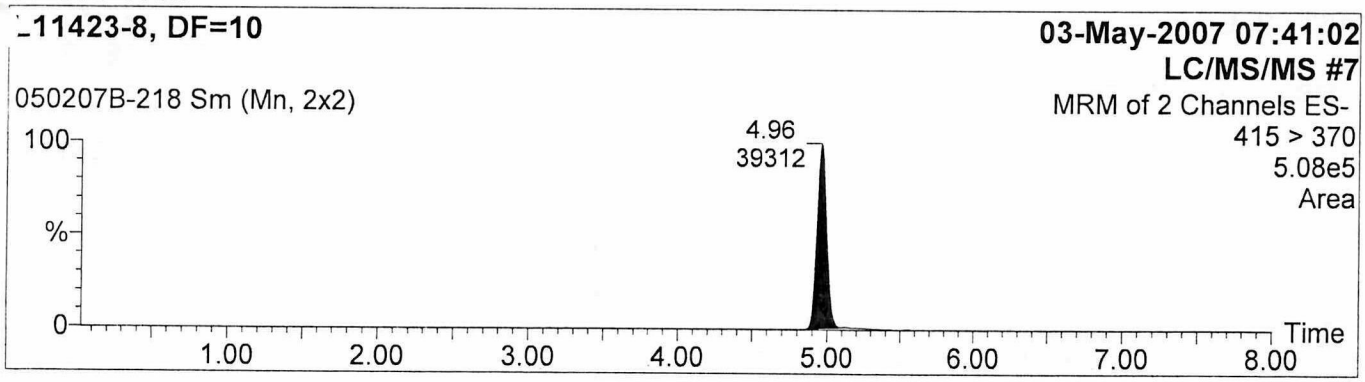
Printed: Thu May 03 15:38:55 2007

Name: 050207B-218
Text:

1: PFOA



2: 13C-PFOA



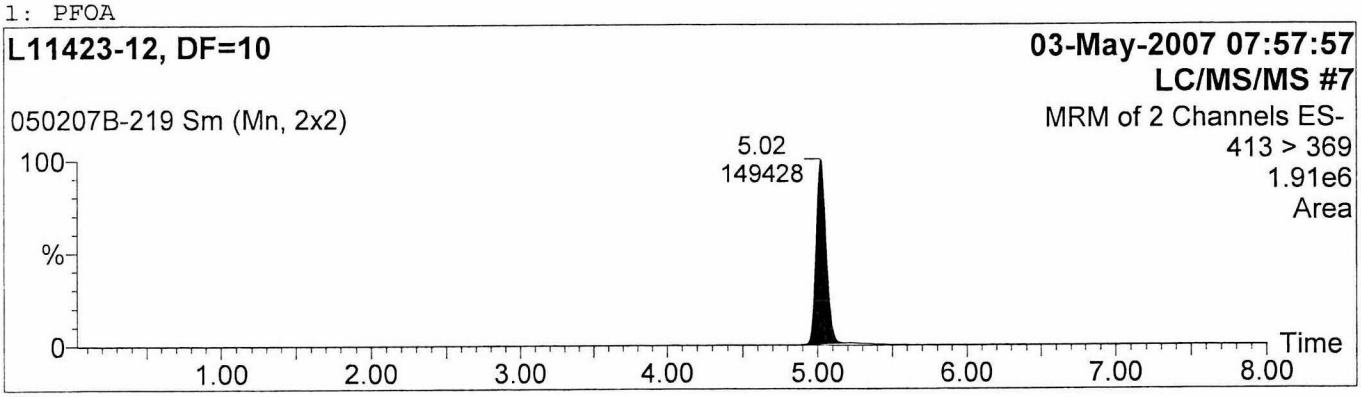
Quantify Sample Report
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

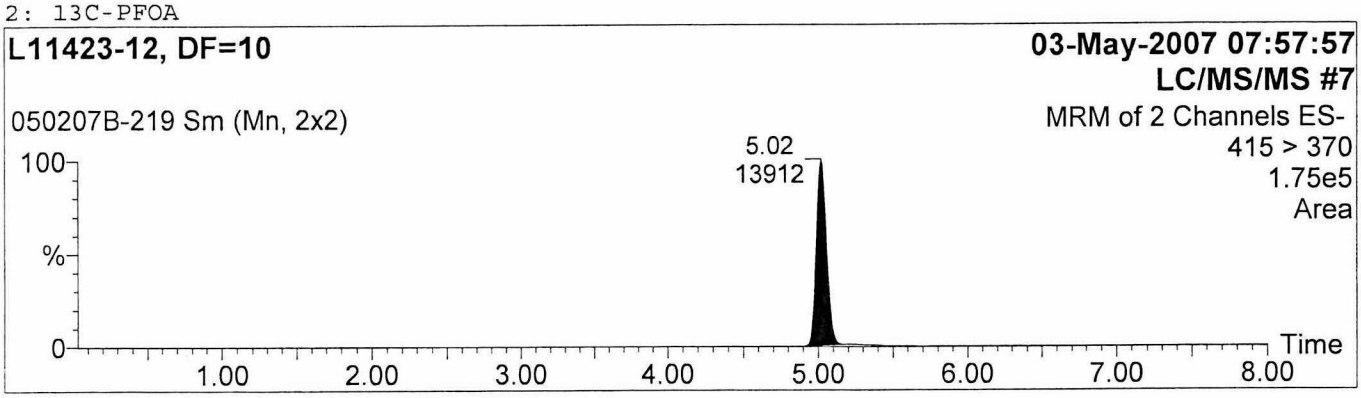
Printed: Thu May 03 15:38:55 2007

Name: 050207B-219
Text:

1: PFOA



2: 13C-PFOA



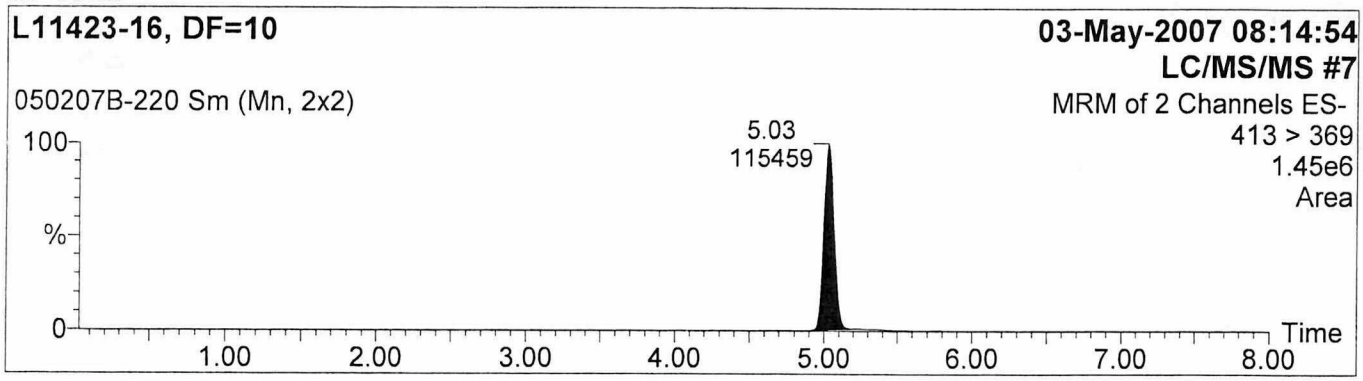
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

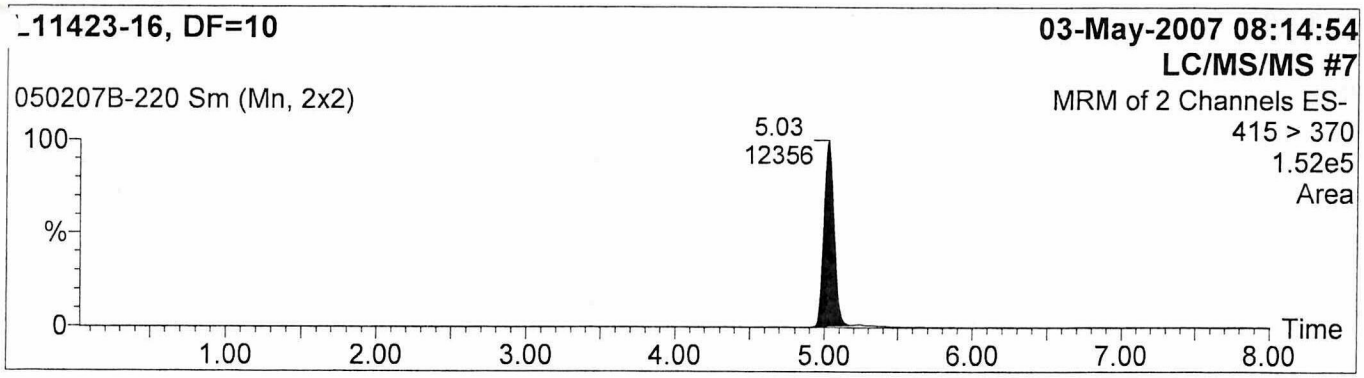
Printed: Thu May 03 15:38:55 2007

Name: 050207B-220
Text:

1: PFOA



2: 13C-PFOA



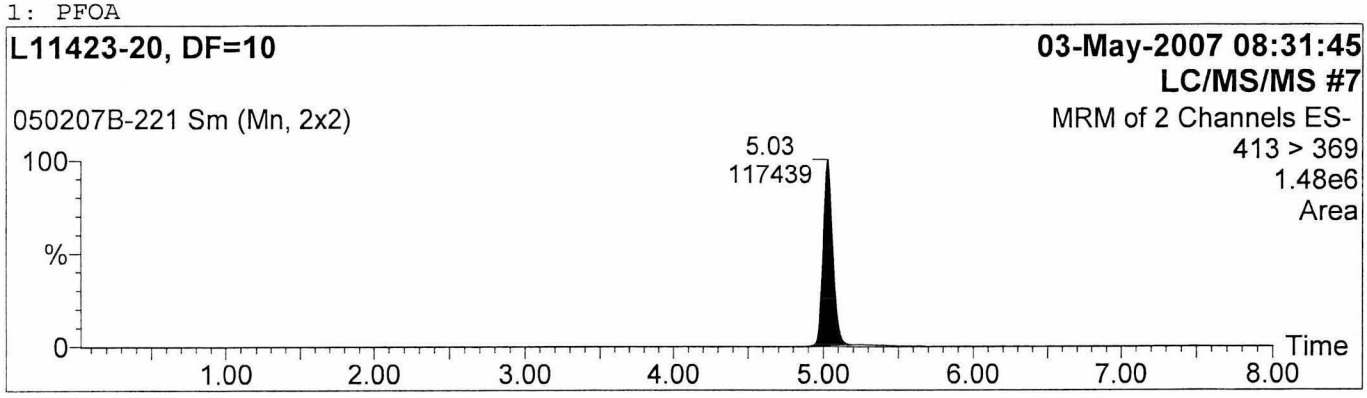
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

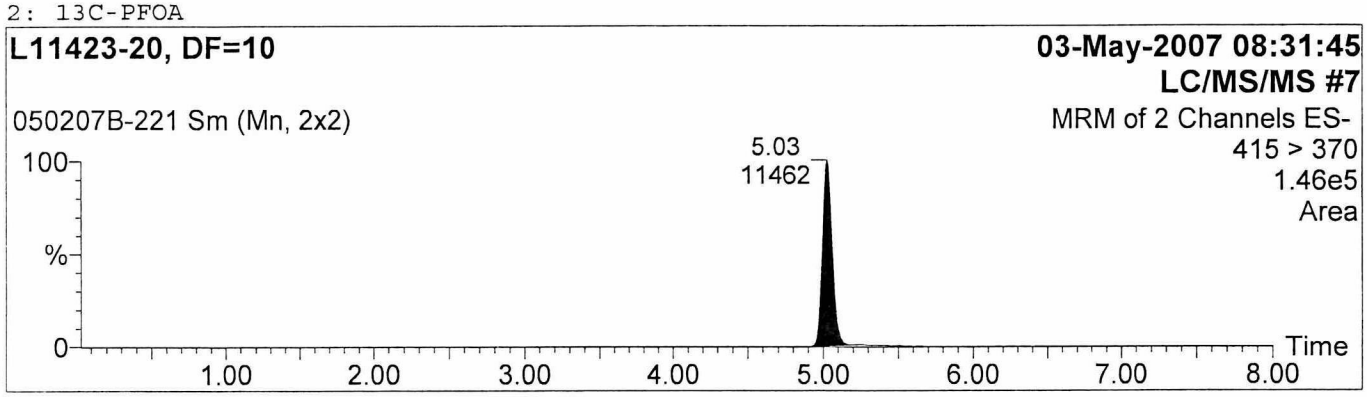
Printed: Thu May 03 15:38:55 2007

Name: 050207B-221
Text:

1: PFOA



2: 13C-PFOA



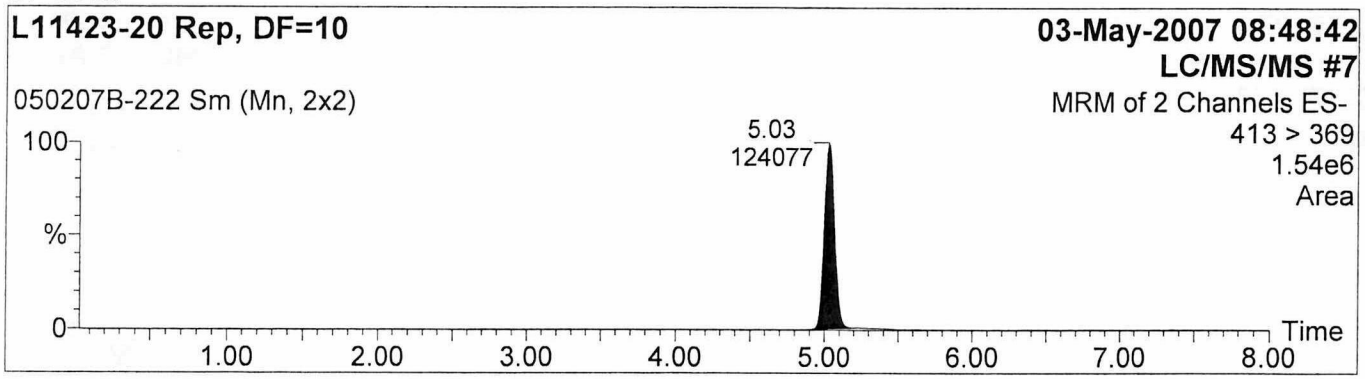
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

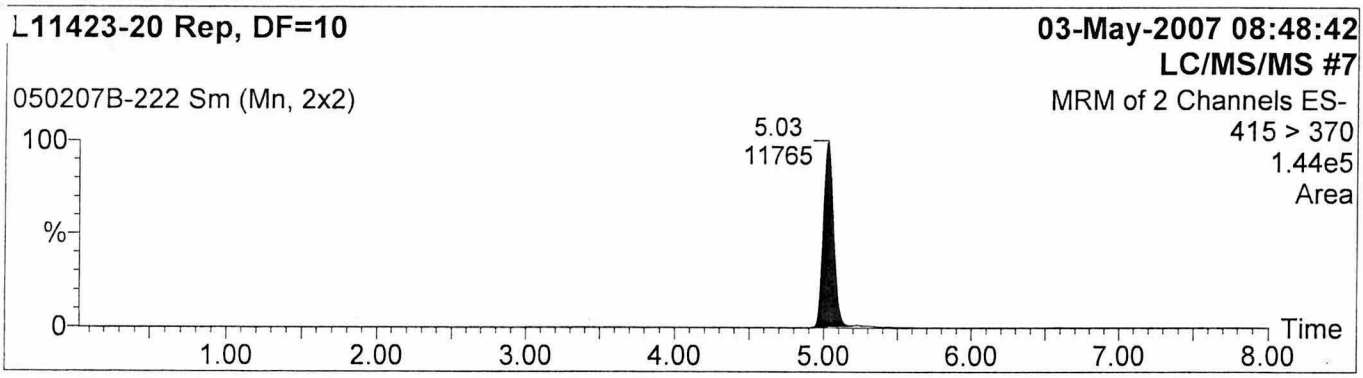
Printed: Thu May 03 15:38:55 2007

Name: 050207B-222
Text:

1: PFOA



2: 13C-PFOA



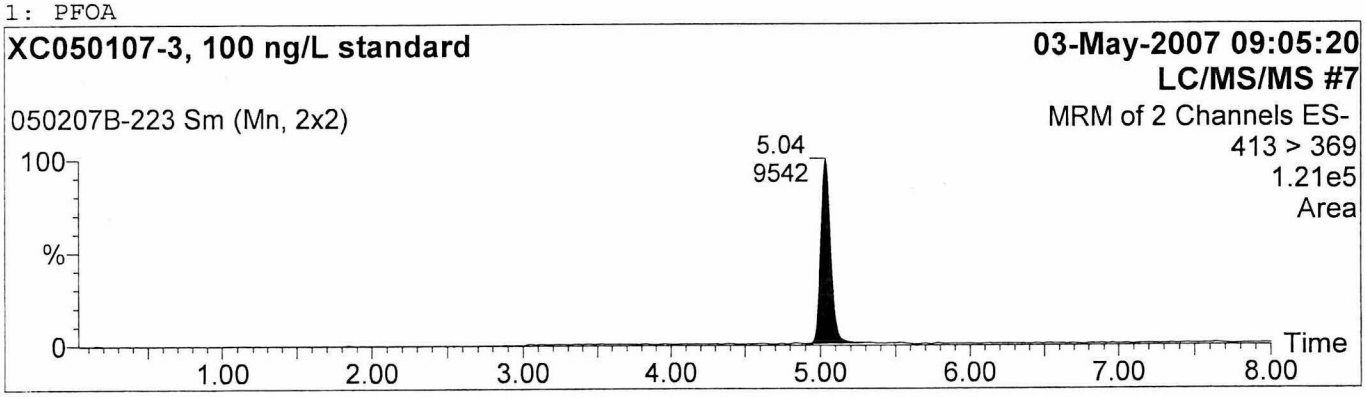
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

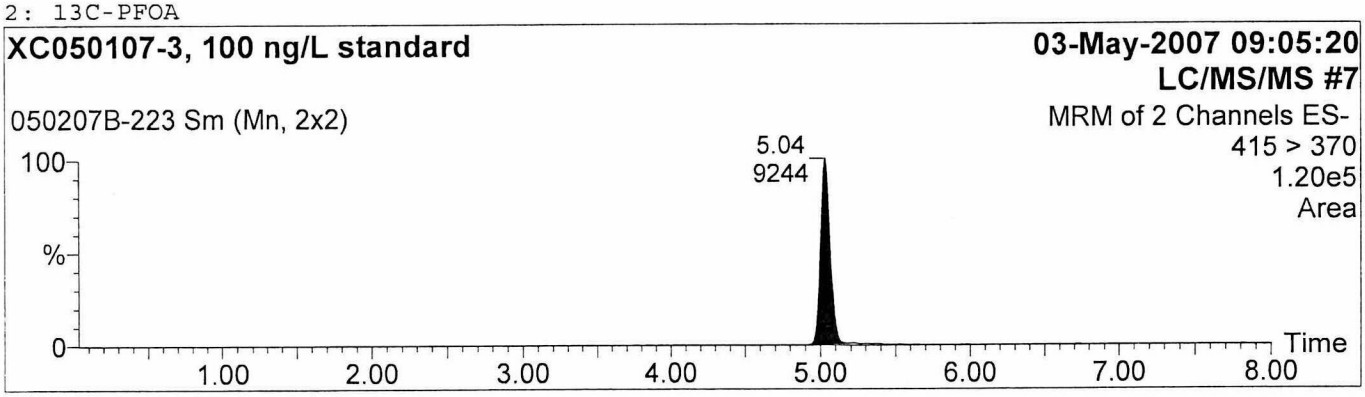
Printed: Thu May 03 15:38:55 2007

Name: 050207B-223
Text:

1: PFOA



2: 13C-PFOA



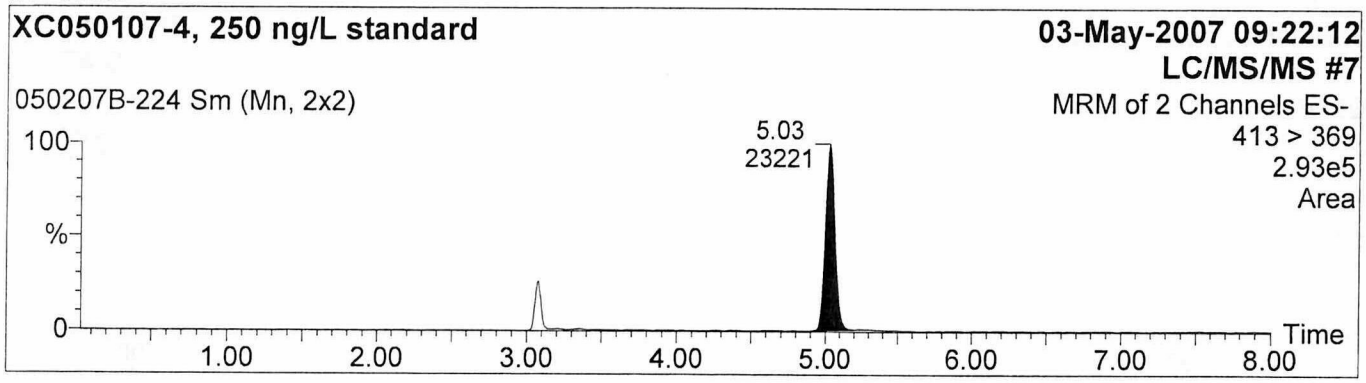
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

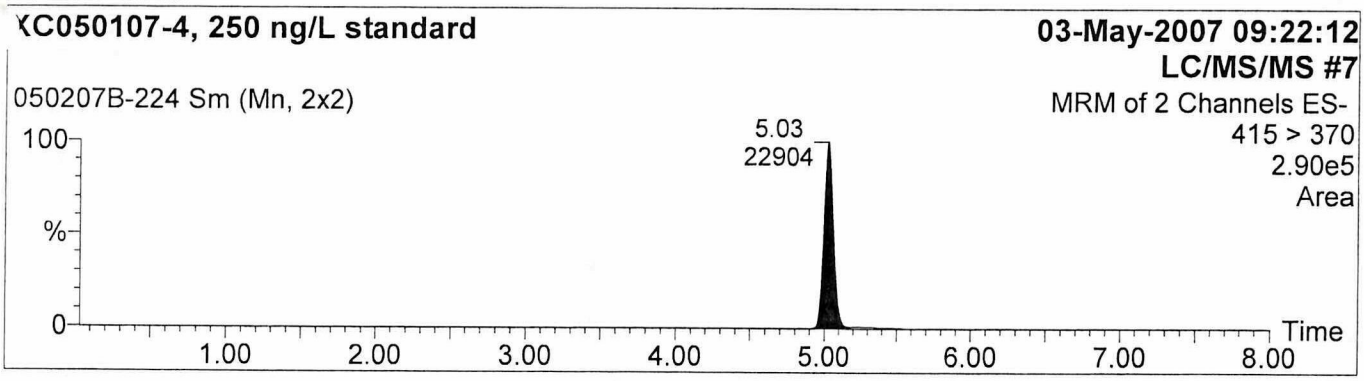
Printed: Thu May 03 15:38:55 2007

Name: 050207B-224
Text:

1: PFOA



2: 13C-PFOA



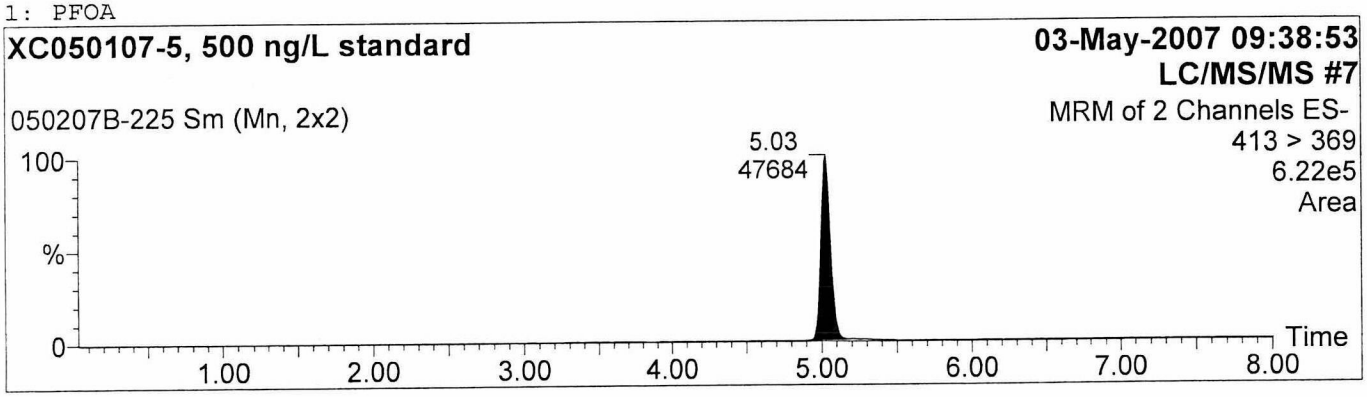
Quantify Sample Report
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

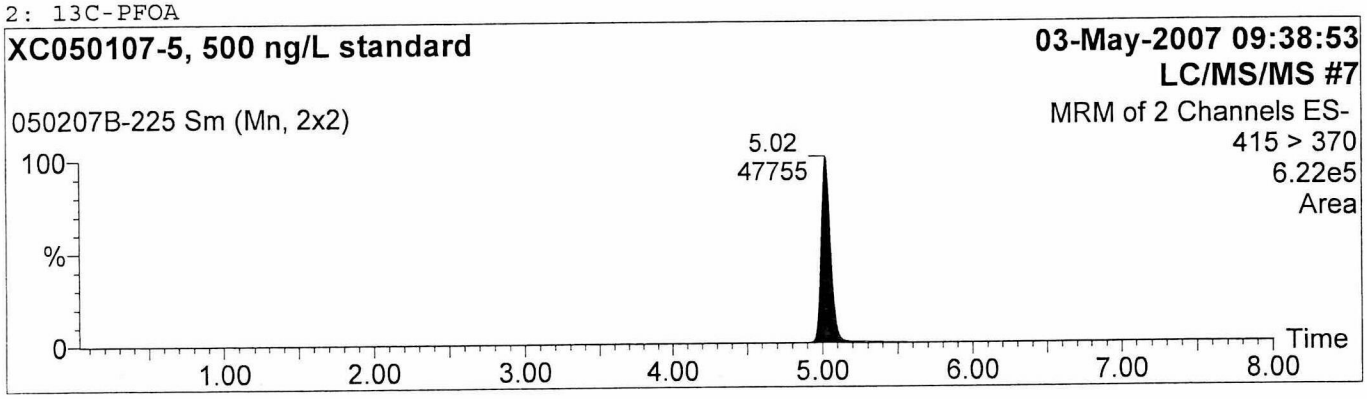
Printed: Thu May 03 15:38:55 2007

Name: 050207B-225
Text:

1: PFOA



2: 13C-PFOA



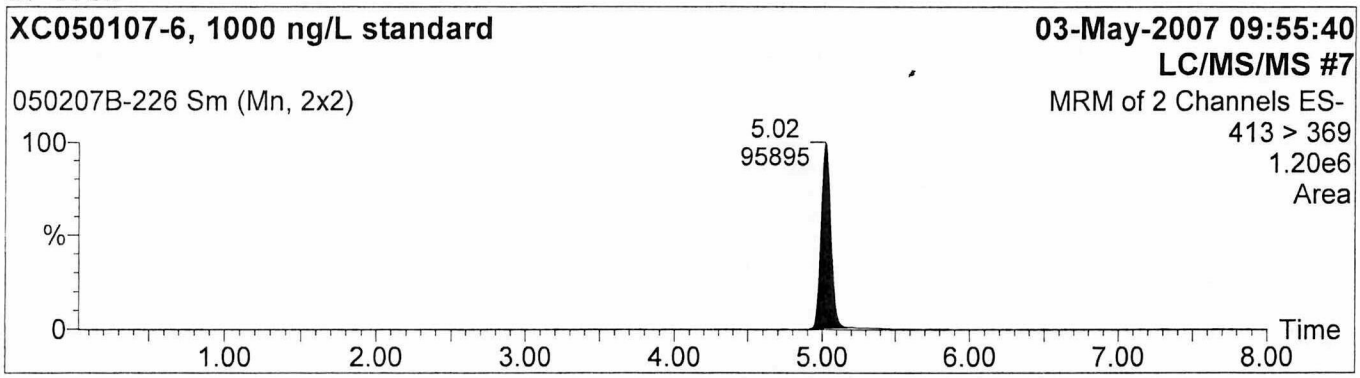
Study No: L11423, Set No: 050207B. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207B MeOH Rinse
Last modified: Thu May 03 15:32:15 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

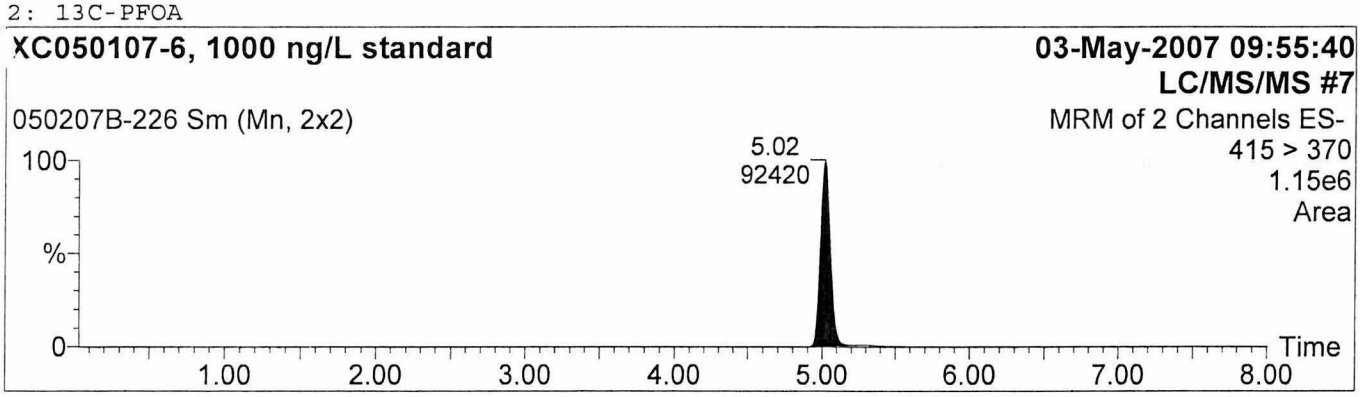
Printed: Thu May 03 15:38:55 2007

Name: 050207B-226
Text:

1: PFOA



2: 13C-PFOA



RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	500 ng/L	Set No:	050407D
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	Methanol/Filter Rinse	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	4.0 mL	Extraction Date:	5/02 & 04/2007
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/06/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050407D-401	0	-	-	0	-	-	-	-
XC050107-1	-	CS	050407D-402	25	-	-	2817	-	-	-	-
XC050107-2	-	CS	050407D-403	50	-	-	5572	-	-	-	-
XC050107-3	-	CS	050407D-404	100	-	-	10772	-	-	-	-
XC050107-4	-	CS	050407D-405	250	-	-	24691	-	-	-	-
XC050107-5	-	CS	050407D-406	500	-	-	51947	-	-	-	-
XC050107-6	-	CS	050407D-407	1000	-	-	100152	-	-	-	-
Methanol Wash	-	C	050407D-408	-	-	-	1141	-	-	-	-
Reagent Control	NA	C	050407D-409	-	-	1	0	ND	ND	-	-
Reagent Spk A	NA	LCS	050407D-410	-	-	1	6197	59.0	61.4	50	118
Reagent Spk B	NA	LCS	050407D-411	-	-	1	50903	527	548	500	105
L11423-20 Spk C	SG-A-020	LF	050407D-412	-	390	1000	6733	64600	67300	50000	103
L11423-20 Spk C	SG-A-020	LF	050407D-413	-	390	10	577953	*	-	50000	-
Methanol Wash	-	C	050407D-414	-	-	-	1333	-	-	-	-
XC050107-1	-	CS	050407D-415	25	-	-	2883	-	-	-	-
XC050107-2	-	CS	050407D-416	50	-	-	5255	-	-	-	-
L11423-12	SG-A-012	S	050407D-417	-	310	100	18501	18800	19600	-	-
L11423-16	SG-A-016	S	050407D-418	-	350	100	13912	14000	14600	-	-
L11423-20	SG-A-020	S	050407D-419	-	390	100	13049	13100	13600	-	-
L11423-20 Rep	SG-A-020	S	050407D-420	-	390	100	13269	13300	13800	-	-
XC050107-3	-	CS	050407D-421	100	-	-	9878	-	-	-	-
XC050107-4	-	CS	050407D-422	250	-	-	24107	-	-	-	-
XC050107-5	-	CS	050407D-423	500	-	-	46114	-	-	-	-
XC050107-6	-	CS	050407D-424	1000	-	-	90413	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 560.259
Slope = 95.5524
Coef. Of Det. = 0.994731

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 250 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 500 ng/L (LOQ)

*Sample was analyzed with several dilution factors in this set. The appropriate result is reported.

Spreadsheet prepared by: MLC / 5/10/07

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

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423 Matrix: MeOH Rinse Impinger Sample
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 34
Time 8:10 AM Date 5/4/2007 Initials EDD

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-A-020	L11423-20* Spk C	390 mL	40.0	200.0
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
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-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
L11423-20* Spk C	SS20796 (1000 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/4/07
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-

All samples were measured:
Time 10:10 AM Date 5/4/2007 Initials EDD

After measuring samples were returned to refrigerator No. 34
Time 3:00 PM Date 5/4/2007 Initials EDD

Comments: *Sample is pre-diluted ten times by taking 4 mL of sample and bringing the volume up to 40 mL with HPLC water

Analysis Summary:
Data Set: 050407D Initials/Date: MLC / 5/4/07
Data Set: - Initials/Date: - / -
Data Set: - Initials/Date: - / -

Set extraction/analysis data verified by: CEE Date: 05/24/07

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

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007C[$$] PROTOCOL NUMBER: NA

MATRIX: MeOH Rinse Impinger Sample ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	Reagent Control			-		-	-	Methanol	46314
na	Reagent Spk A			-		-	-	C18 SPE	605837081B
na	Reagent Spk B			-		-	-	HPLC Water	46335
SG-A-020	L11423-20* Spk C	↓	↓	0.1/10	↓	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	*Initials/Date*	EDD 5/4/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C44404
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date	-	EDD 5/4/07	EDD 5/4/07	MLC 5/4/07	MLC 5/4/07	-	-	*Initials/Date*	MLC 5/4/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

*Sample was diluted ten times prior to extraction.
**Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:

Final extracts stored in refrigerator 32 Initials: EDD Date: 5/4/2007

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

Exygen STUDY NO. L11423

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Protocol No.: None
Method No.: $$
Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA
Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC050107-0	Methanol	46303
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC050107-1	C18 SPE	0053370818
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC050107-2	Water	46335
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC050107-3	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC050107-4	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC050107-5	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC050107-6	-	-
-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/01/07

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) / freezer # 34 Time: 9:10 AM — Initials/Date: EDD 1 5/01/07

40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD 1 5/01/07

After measuring, samples returned to (refrigerator) / freezer # 34 Time: 11:10 AM — Initials/Date: EDD 1 5/01/07

Samples fortified: Initials/Date: AMB 1 05/01/07

SPE clean-up: Initials/Date: EDD 1 5/01/07

Final volume adjusted to 5 mL: Initials/Date: EDD 1 5/01/07

Extracts placed in refrigerator # 32 Initials/Date: EDD 1 5/01/07

Comments:

STANDARD EXPIRATION DATE: 5/15/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/10/07

Masslynx - Sample List

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050407D MeOH Rinse.SPL
Printed: Fri May 04 15:14:41 2007 MLC 5/4/07

Exygen STUDY NO. L11423

Page Position: (1, 1)

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/mL)	Conc B	Conc C	Test ID	DF
1	94	050407D-401	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---	0	1
2	95	050407D-402	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
3	96	050407D-403	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
4	97	050407D-404	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
5	98	050407D-405	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
6	99	050407D-406	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
7	100	050407D-407	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1
8	92	050407D-408	---	---	Methanol Wash	---	Blank	---	---	---	0	1
9	64	050407D-409	---	---	Reagent Control	---	Blank	---	---	---	0	1
10	65	050407D-410	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---	0	1
11	66	050407D-411	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500	---	0	1
12	67	050407D-412	---	---	L11423-20 Spk C, 50,000 ng/L, DF=1,000	---	QC	50000	1282	---	0	1000
13	68	050407D-413	---	---	L11423-20 Spk C, 50,000 ng/L, DF=10	---	QC	50000	1282	---	0	10
14	92	050407D-414	---	---	Methanol Wash	---	Blank	---	---	---	0	1
15	95	050407D-415	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---	0	1
16	96	050407D-416	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---	0	1
17	69	050407D-417	---	---	L11423-12, DF=100	---	Analyte	---	1613	---	0	100
18	70	050407D-418	---	---	L11423-16, DF=100	---	Analyte	---	1429	---	0	100
19	71	050407D-419	---	---	L11423-20, DF=100	---	Analyte	---	1282	---	0	100
20	72	050407D-420	---	---	L11423-20 Rep, DF=100	---	Analyte	---	1282	---	0	100
21	97	050407D-421	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---	0	1
22	98	050407D-422	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---	0	1
23	99	050407D-423	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---	0	1
24	100	050407D-424	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---	0	1

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050407D MeOH Rinse.SPL
Printed: Fri May 04 15:14:41 2007 MLC 5/4/07

Exygen STUDY NO. L 11423

	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
20	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
21	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
22	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
23	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
24	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA

Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/4/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/10/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Fri May 04 15:14:49 2007 MLC 5/4/07

Solvent Delay (mins) : 0.00

Function : 1 MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Friday, May 04, 2007 14:57:57

Printed: Friday, May 04, 2007 15:14:54 MLC 5/4/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

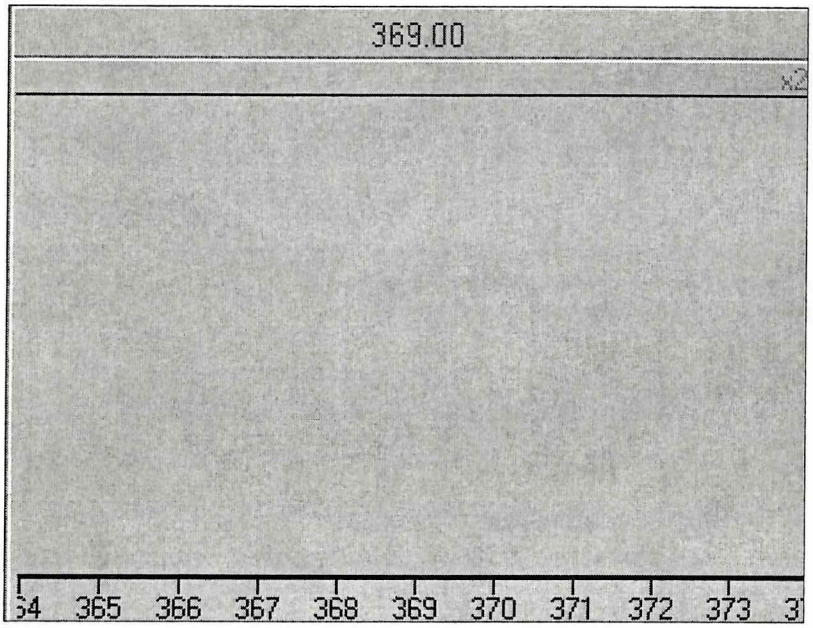
HP1100 Autosampler Initial Conditions

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	77

Tuning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Fri May 04 15:15:04 2007 MLC 5/4/07



Dau 413.00

SOURCE (ESP-)	Set	Rdbk	Analyser	Set	Rdbk
Capillary	3.00	-2.92	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	102	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	90.0
Gas Cell	2.3e-3	Desolvation	682.4

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050407D
Extraction Date: 5/02 & 04/2007
Analyzed on: 05/06/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2817
XC050107-2	50	5572
XC050107-3	100	10772
XC050107-4	250	24691
XC050107-5	500	51947
XC050107-6	1000	100152

Coefficient of Determination: 0.99958502

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999792489
R Square	0.99958502
Adjusted R Square	0.999481275
Standard Error	857.891689
Observations	6

MLC 5/10/07

ANOVA

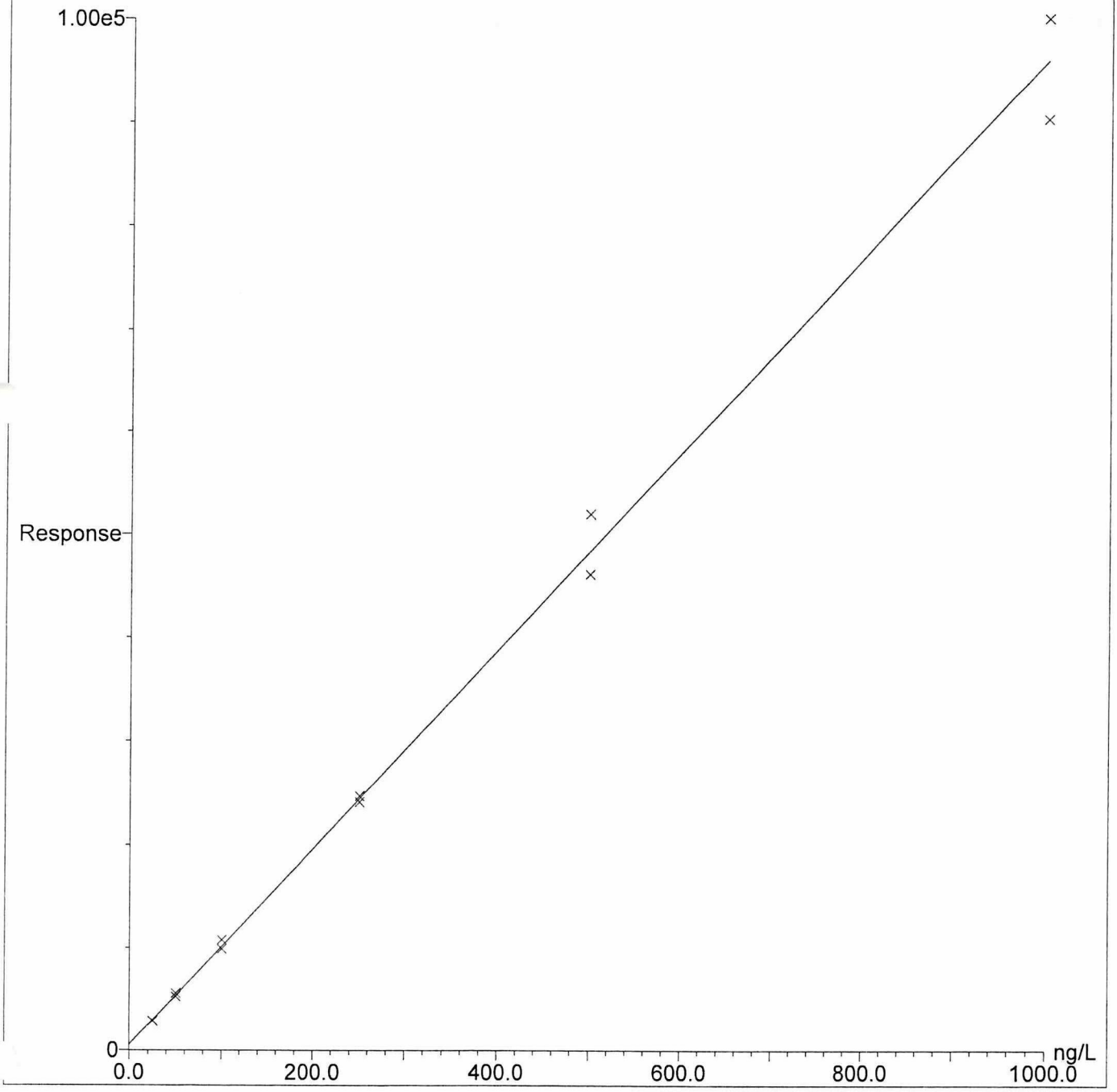
	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	7091168405	7091168405	9635.025719	6.4587E-08
Residual	4	2943912.601	735978.1501		
Total	5	7094112318			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	550.3439753	479.2301794	1.148391731	0.314812833	-780.2123106	1880.900261	-780.2123106	1880.900261
X Variable 1	100.0773694	1.019552144	98.15816685	6.4587E-08	97.24663887	102.9081	97.24663887	102.9081

Quantify Calibration Report
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050407D MeOH
Last modified: Thu May 10 10:54:27 2007
Printed: Thu May 10 10:56:26 2007 MLC 5/10/07

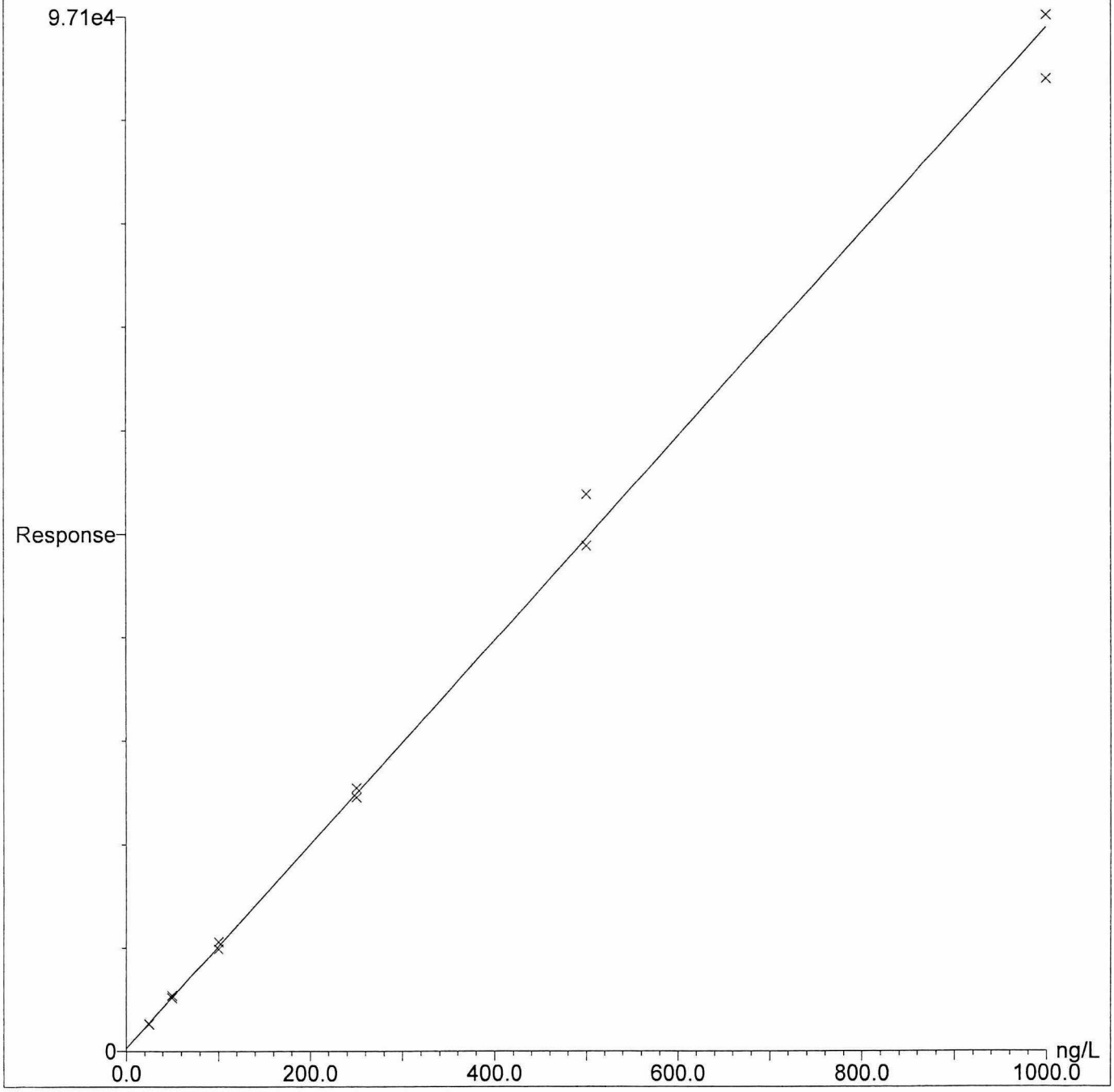
Compound 1 name: PFOA
Coefficient of Determination: 0.994731
Calibration curve: 95.5524 * x + 560.259
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050407D MeOH
Last modified: Thu May 10 10:54:27 2007
Printed: Thu May 10 10:56:26 2007 MLC 5/10/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.996629
Calibration curve: 95.6679 * x + 270.398
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

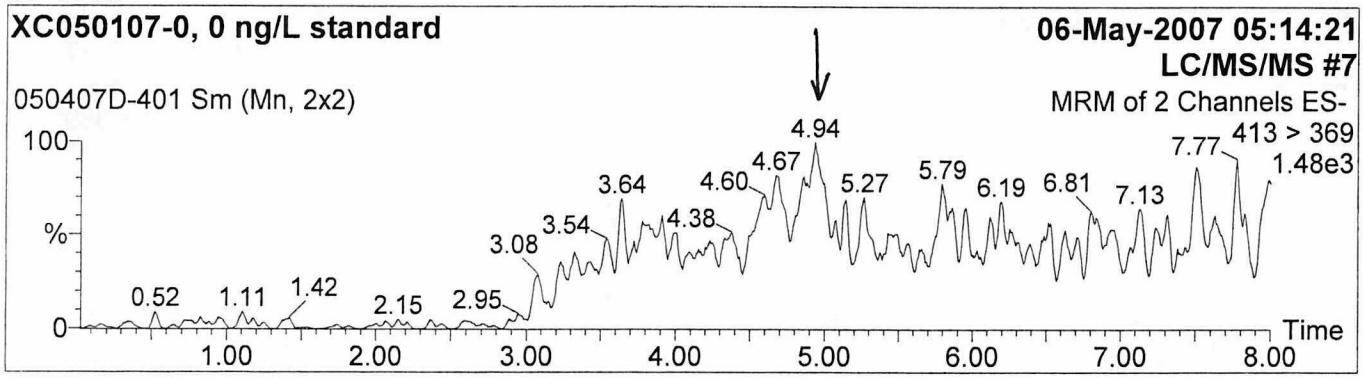
Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

Printed: Thu May 10 10:56:27 2007

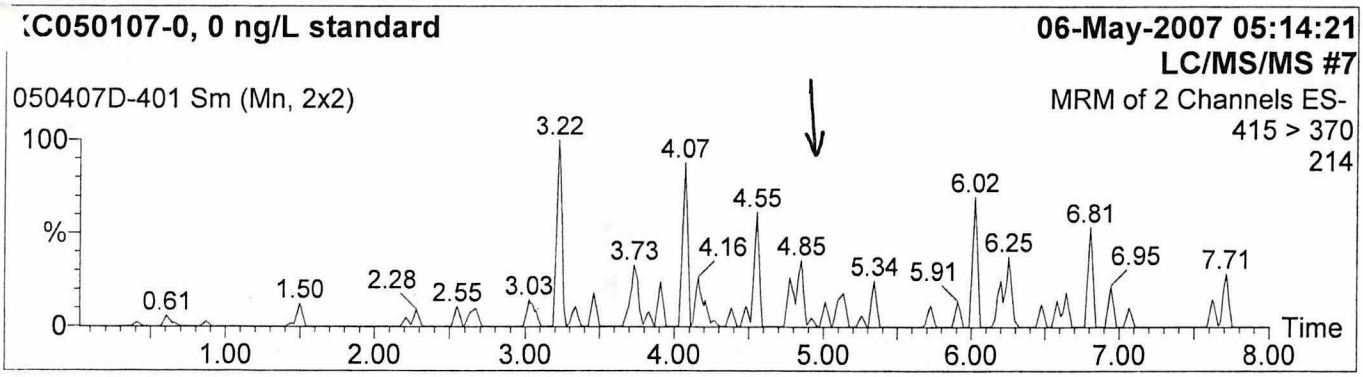
Initials MLC
Date 5/10/07
Run# 050407D-401 To 050407D-424

Name: 050407D-401
Text:

1: PFOA



2: 13C-PFOA



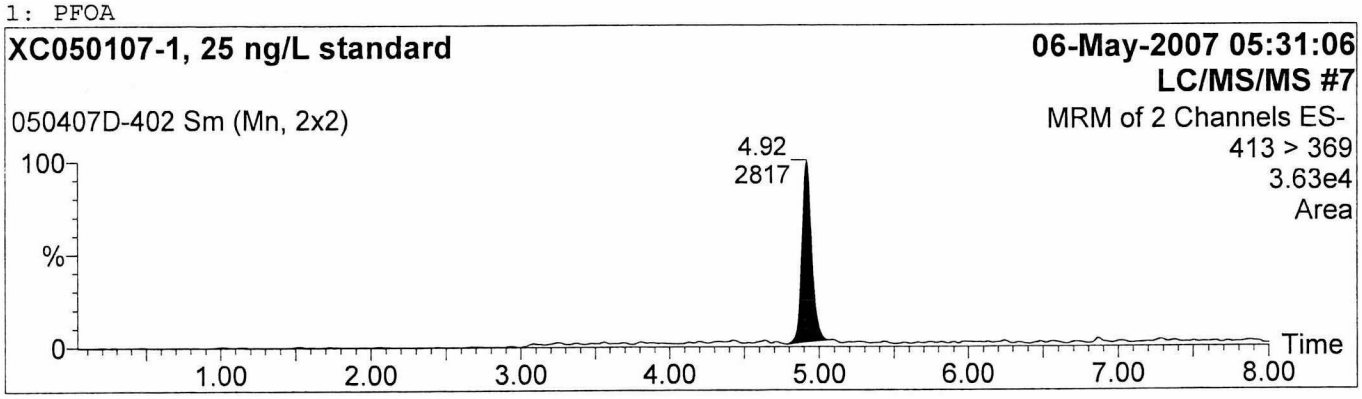
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

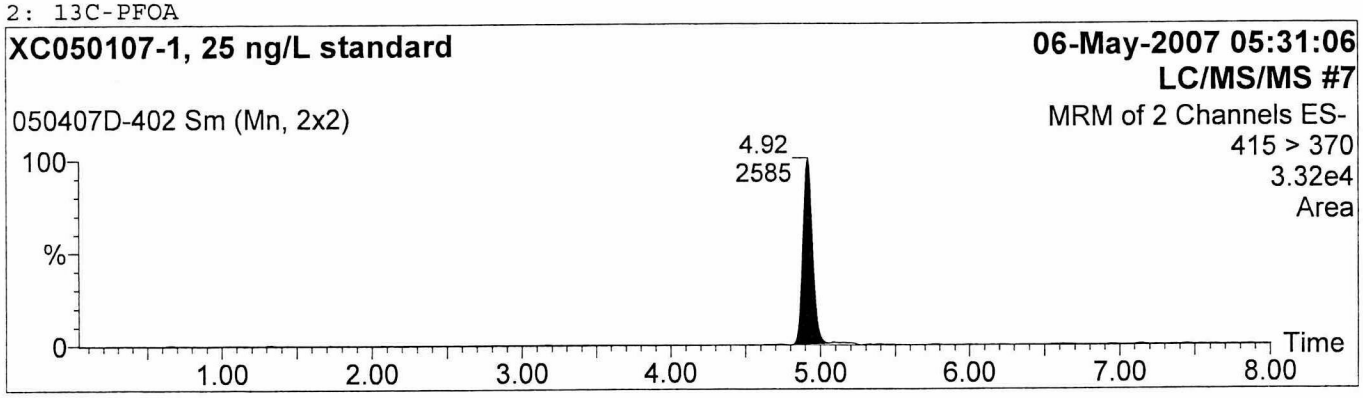
Printed: Thu May 10 10:56:27 2007

Name: 050407D-402
Text:

1: PFOA



2: 13C-PFOA



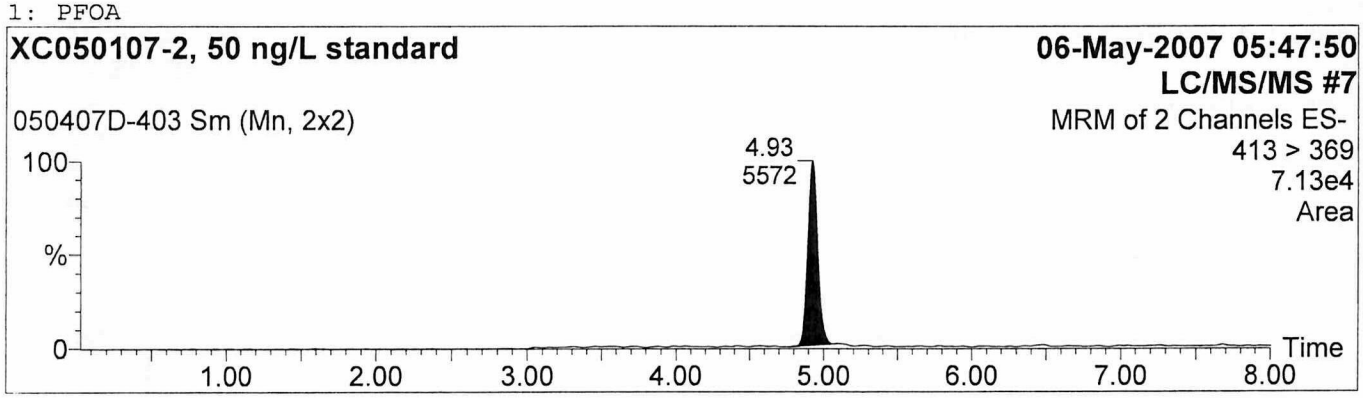
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

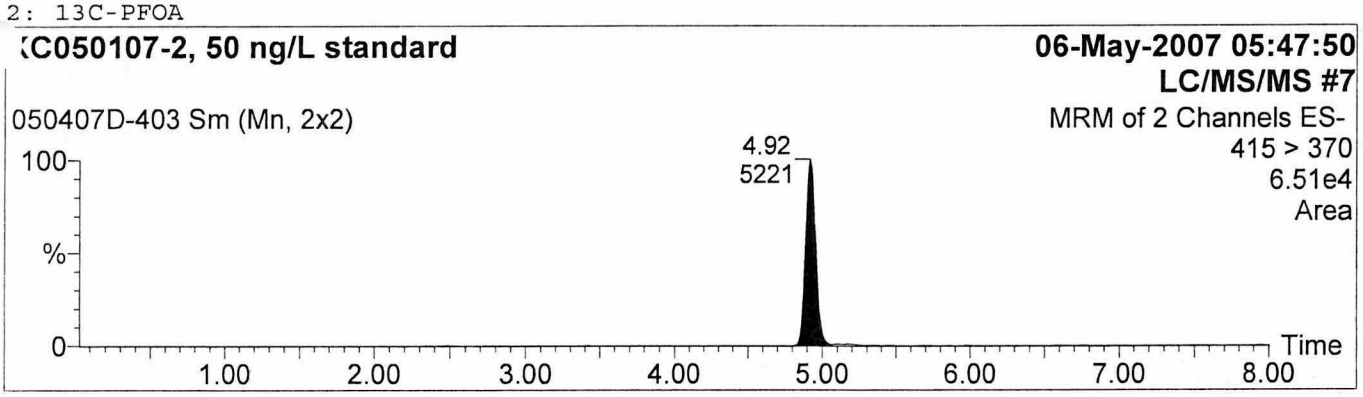
Printed: Thu May 10 10:56:27 2007

Name: 050407D-403
Text:

1: PFOA



2: 13C-PFOA



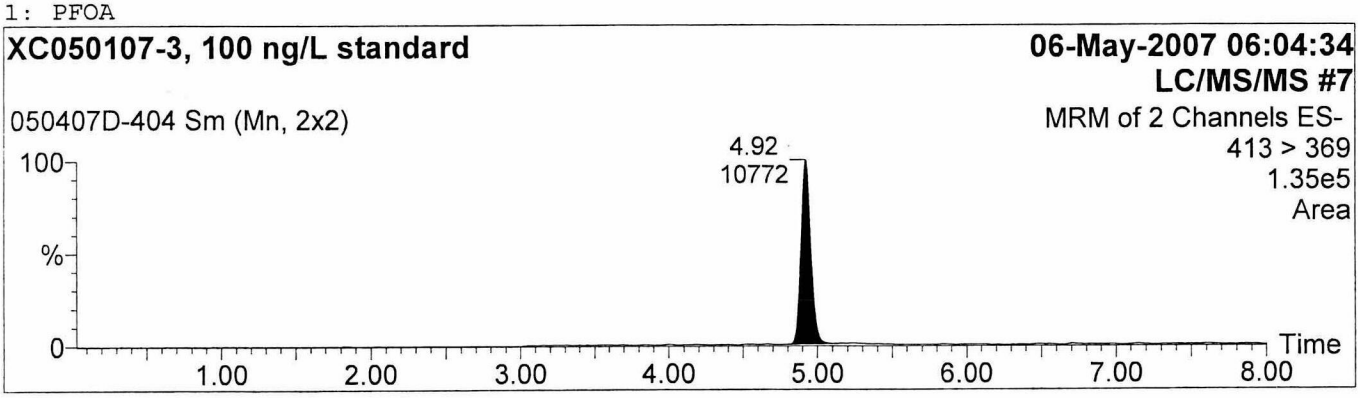
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

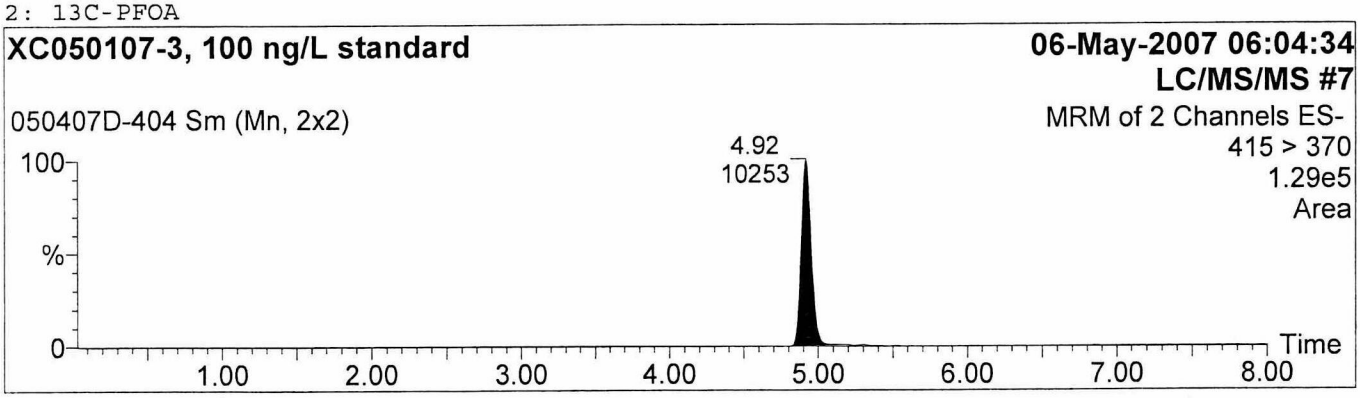
Printed: Thu May 10 10:56:27 2007

Name: 050407D-404
Text:

1: PFOA



2: 13C-PFOA



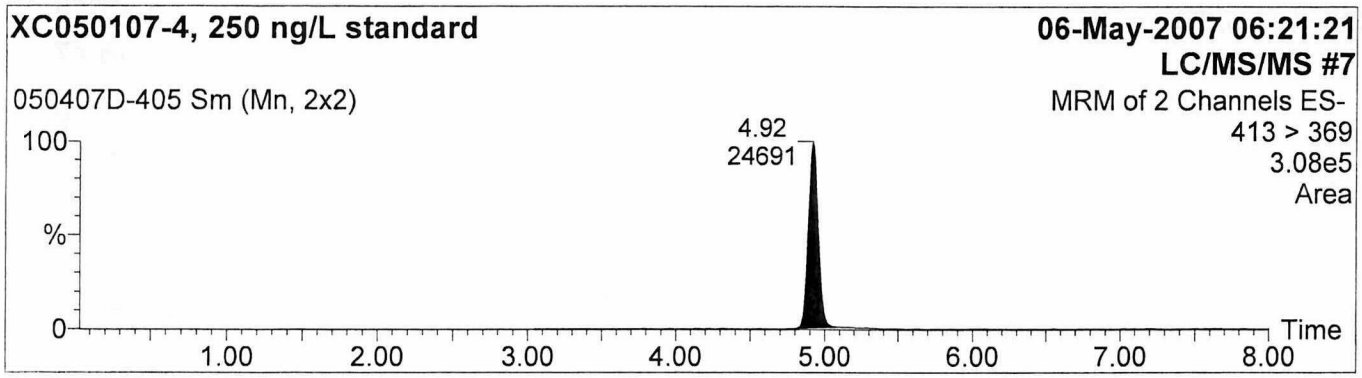
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

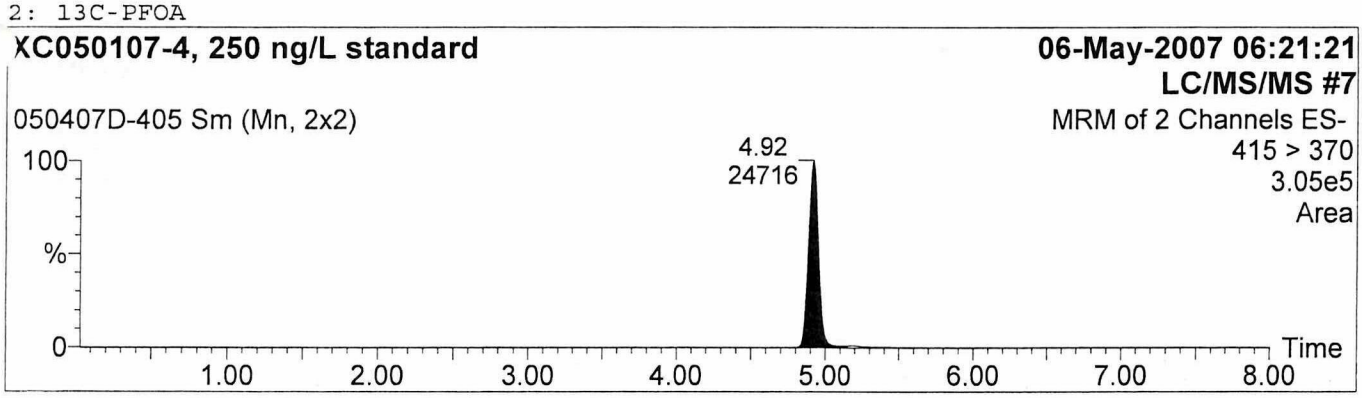
Printed: Thu May 10 10:56:27 2007

Name: 050407D-405
Text:

1: PFOA



2: 13C-PFOA



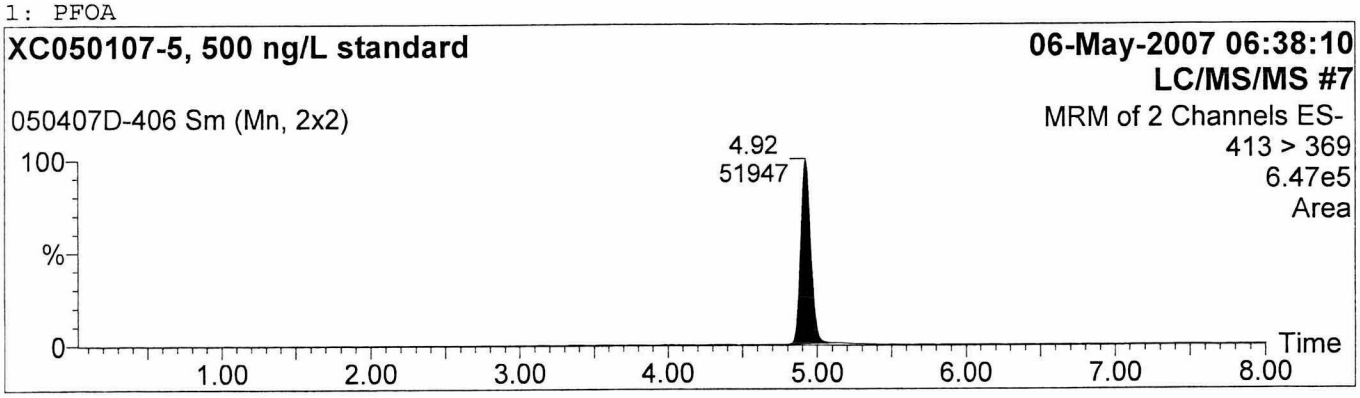
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

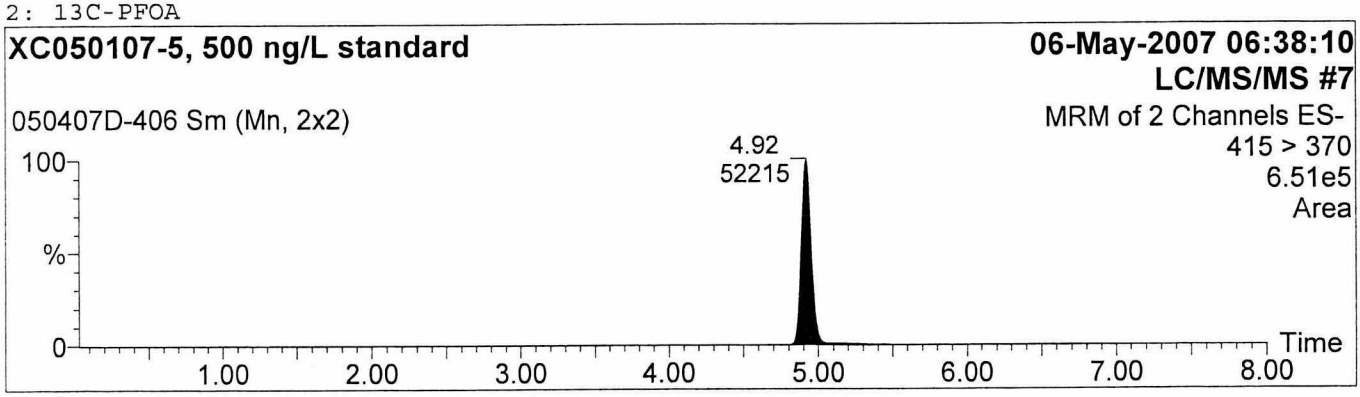
Printed: Thu May 10 10:56:27 2007

Name: 050407D-406
Text:

1: PFOA



2: 13C-PFOA



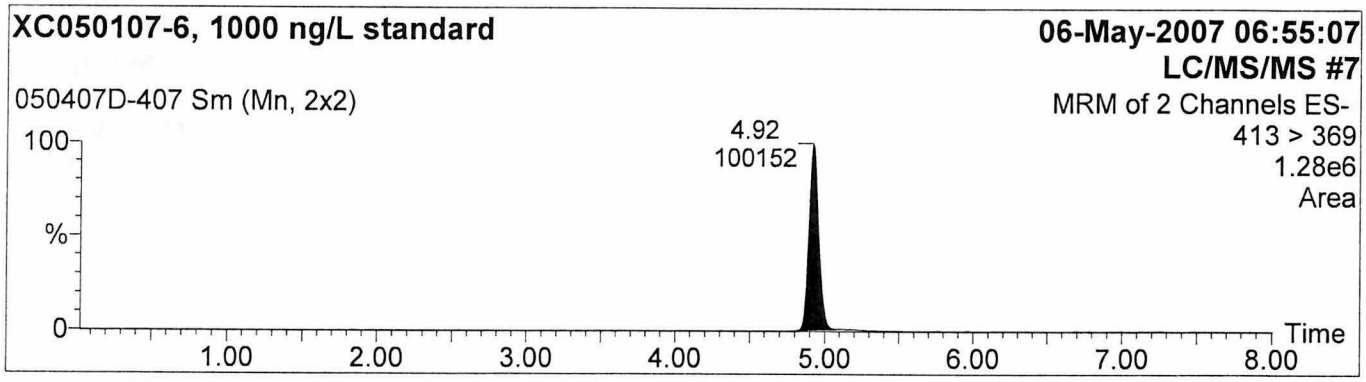
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

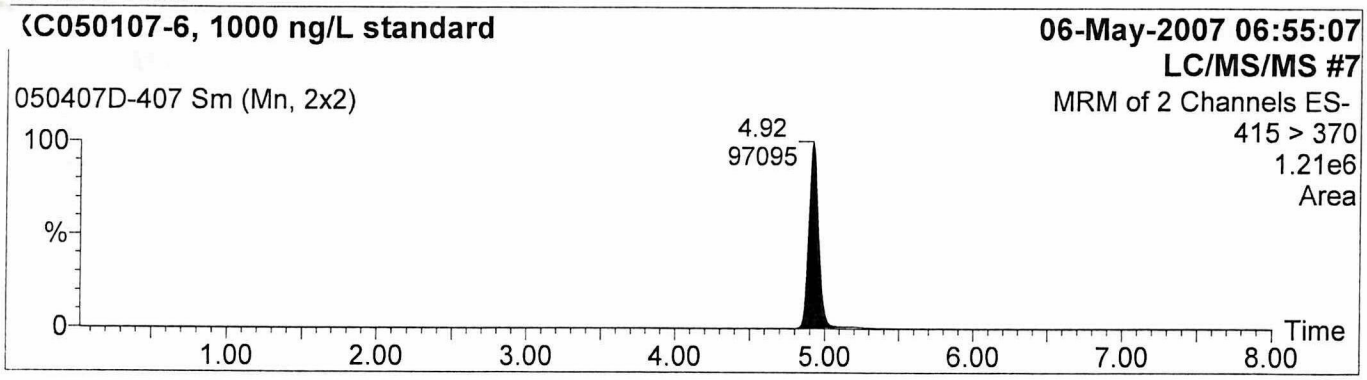
Printed: Thu May 10 10:56:27 2007

Name: 050407D-407
Text:

1: PFOA



2: 13C-PFOA



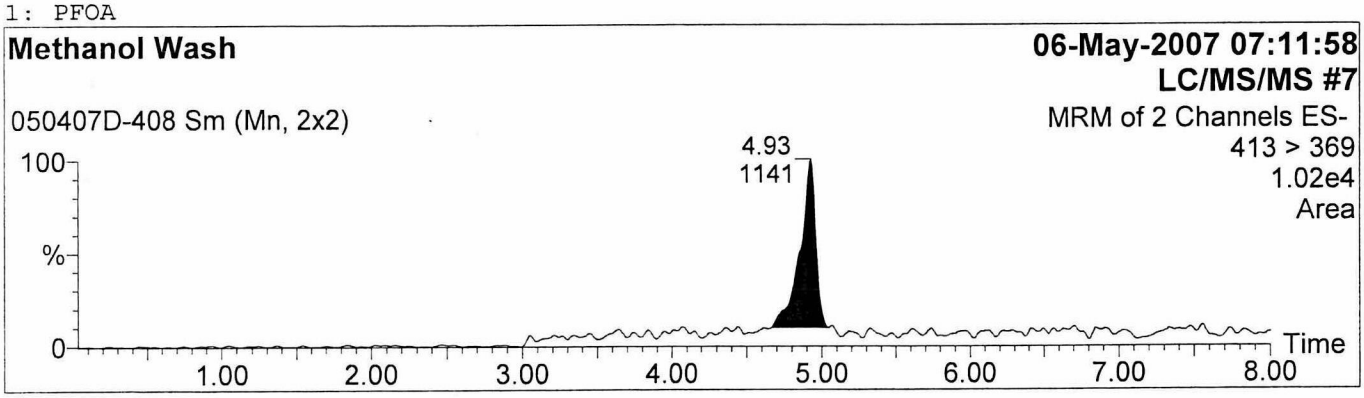
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

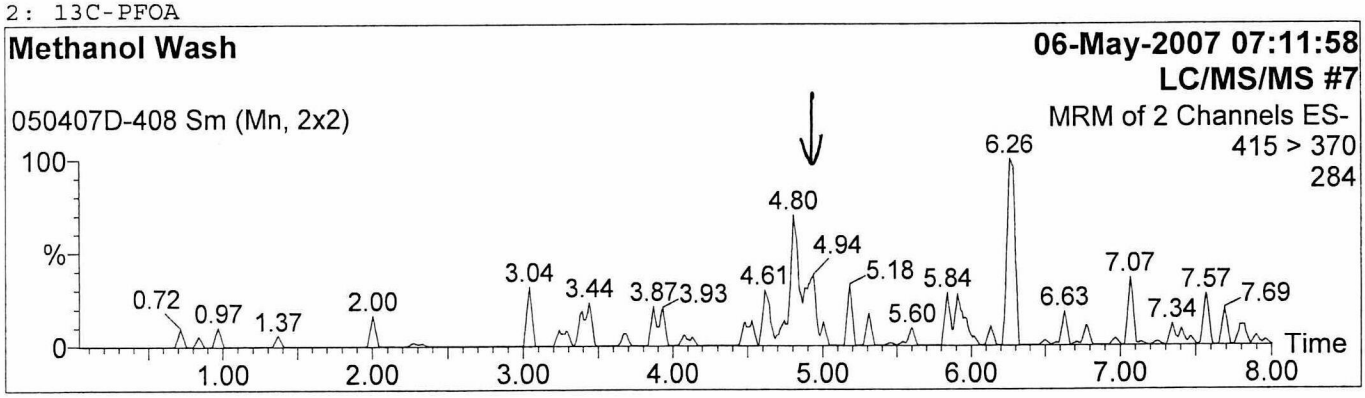
Printed: Thu May 10 10:56:27 2007

Name: 050407D-408
Text:

1: PFOA



2: 13C-PFOA



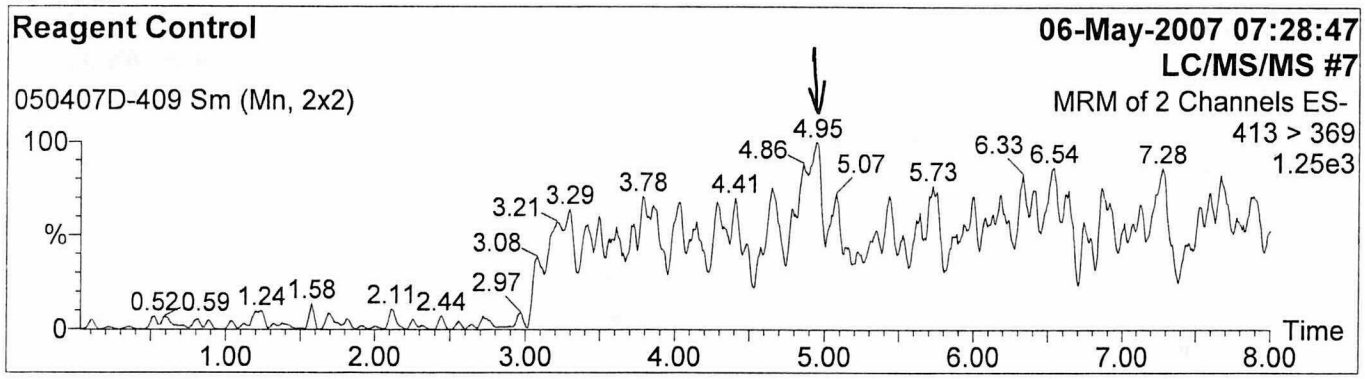
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

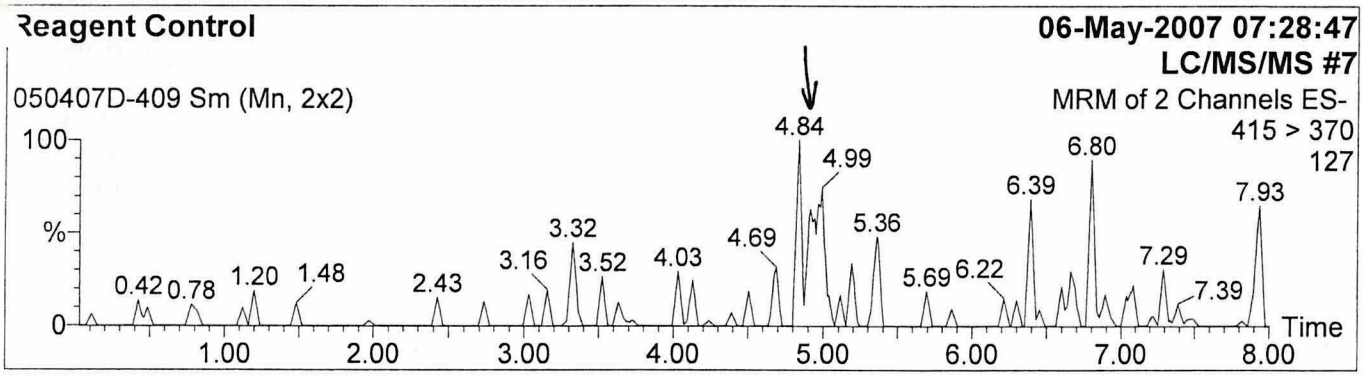
Printed: Thu May 10 10:56:27 2007

Name: 050407D-409
Text:

1: PFOA



2: 13C-PFOA

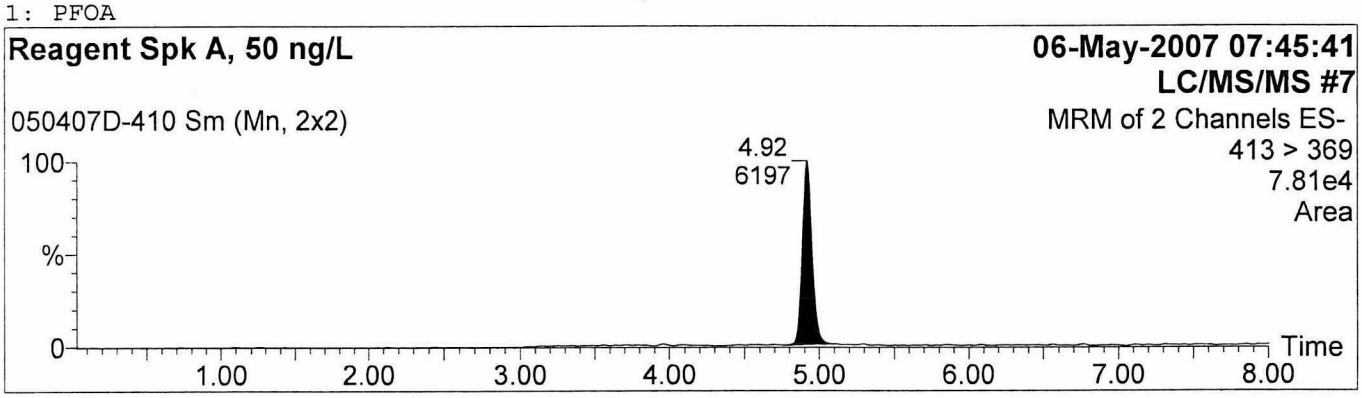


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

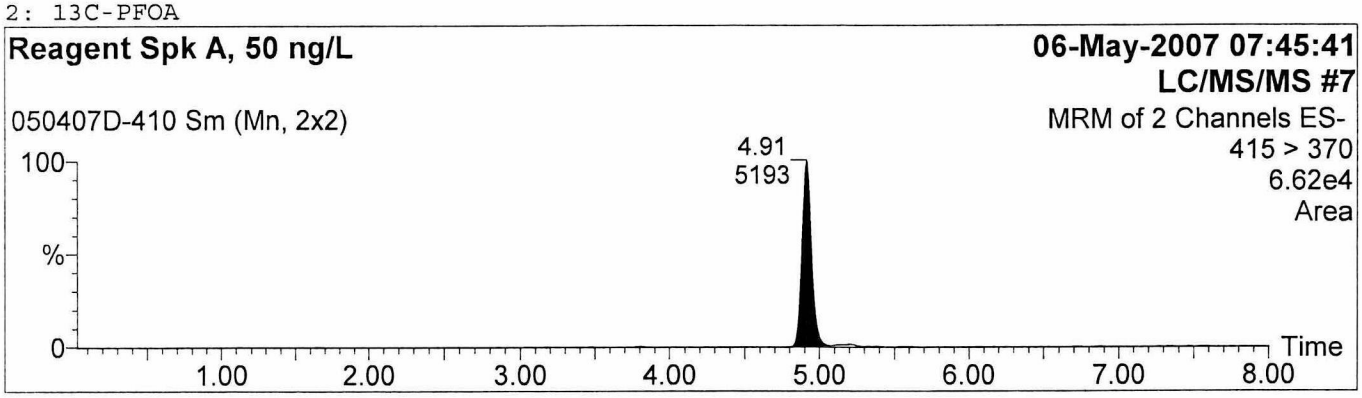
Printed: Thu May 10 10:56:27 2007

Name: 050407D-410
Text:

1: PFOA



2: 13C-PFOA



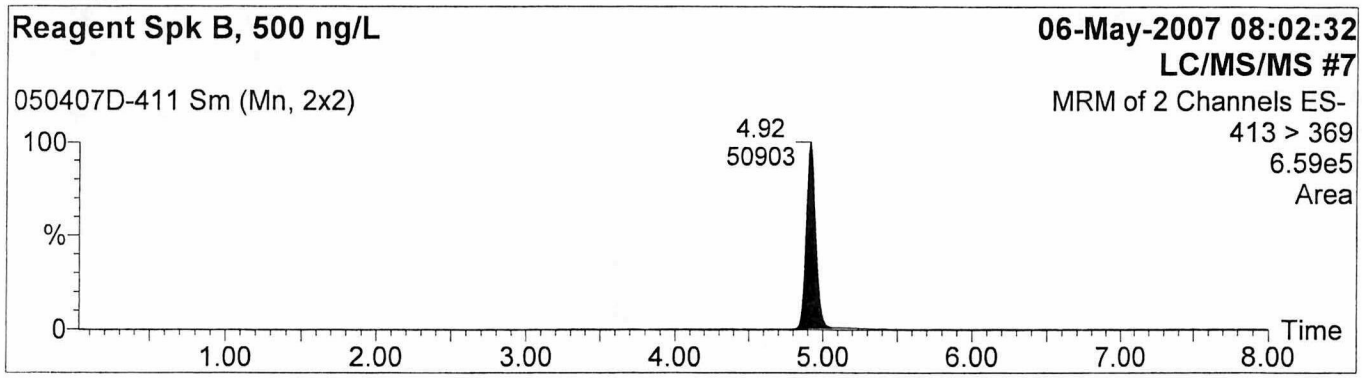
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

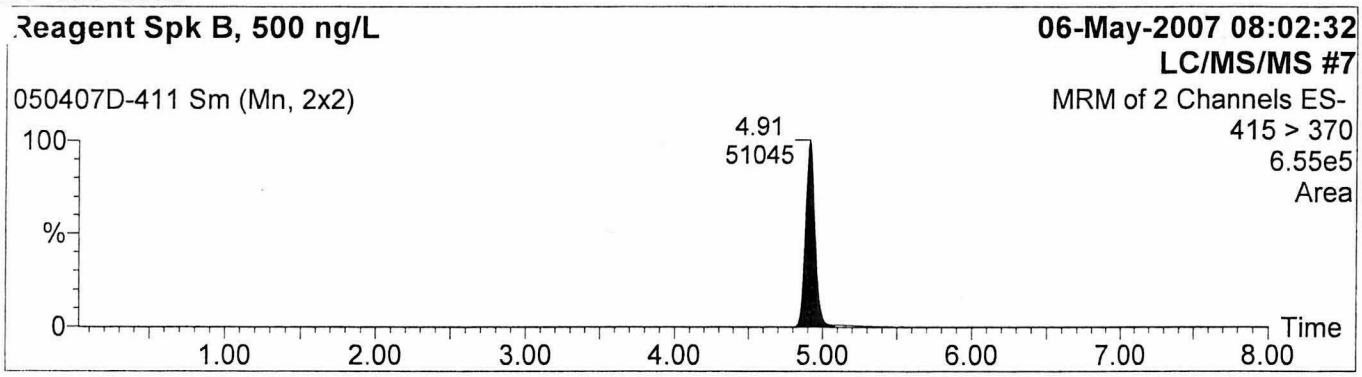
Printed: Thu May 10 10:56:27 2007

Name: 050407D-411
Text:

1: PFOA



2: 13C-PFOA



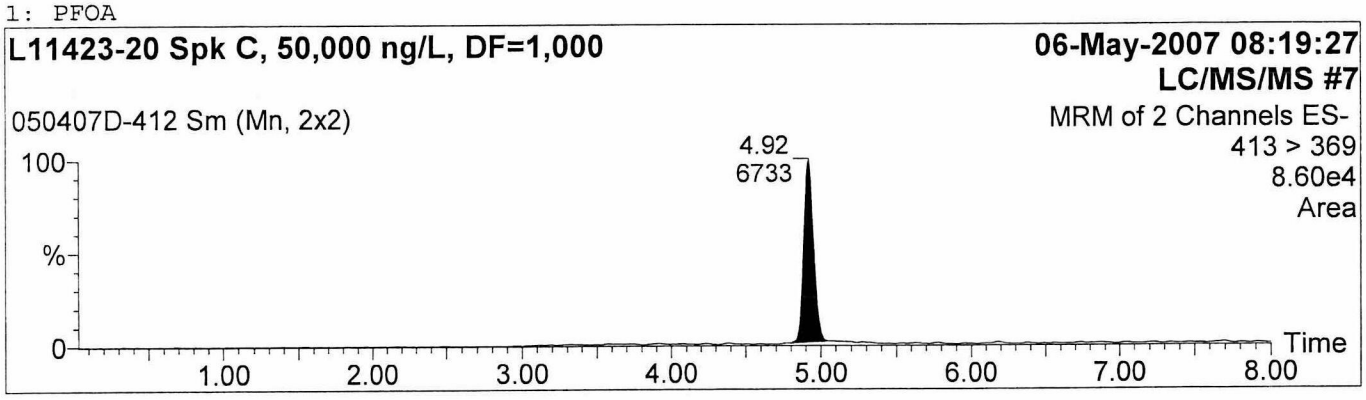
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

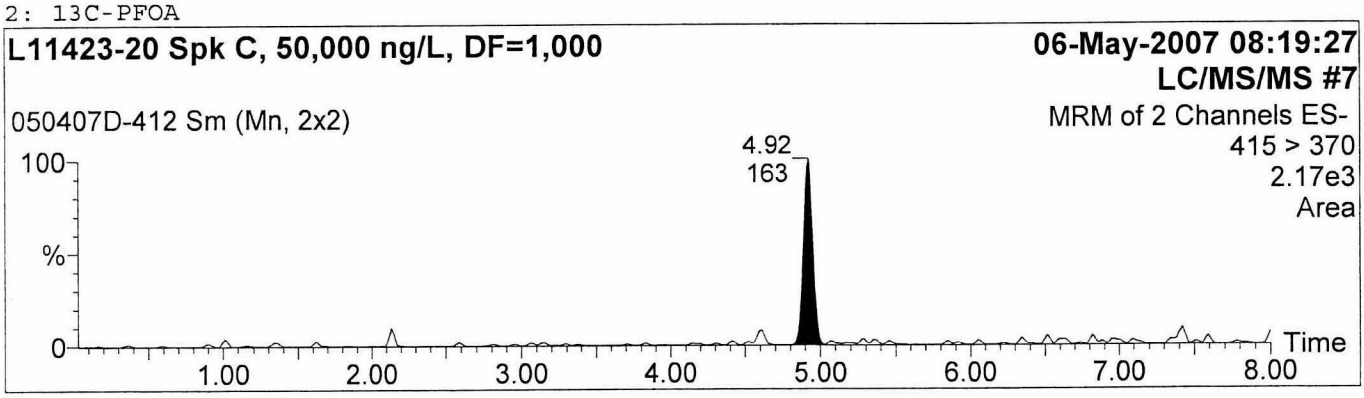
Printed: Thu May 10 10:56:27 2007

Name: 050407D-412
Text:

1: PFOA



2: 13C-PFOA



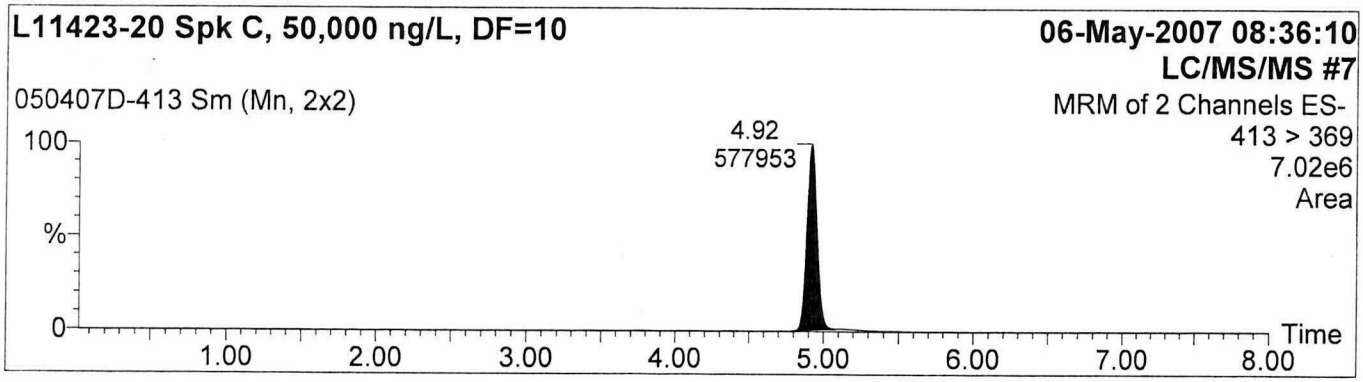
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

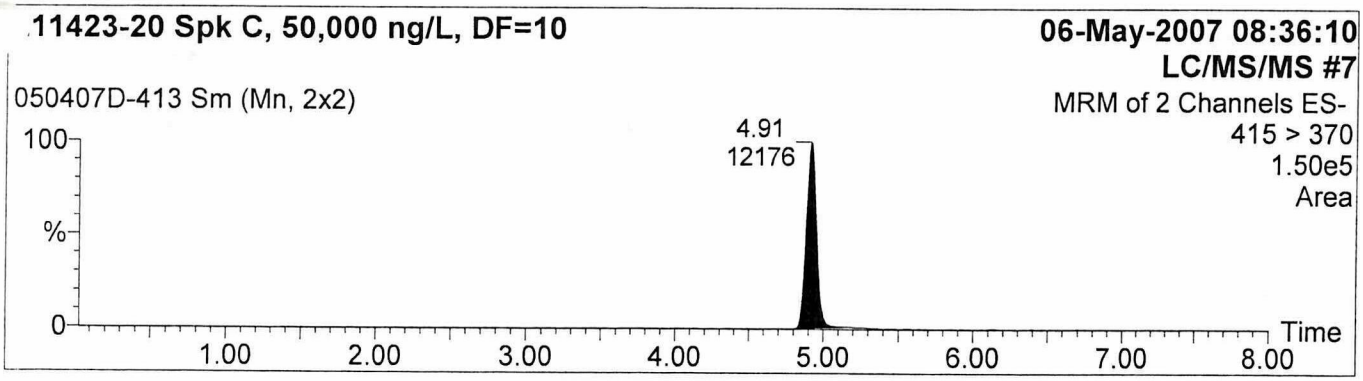
Printed: Thu May 10 10:56:27 2007

Name: 050407D-413
Text:

1: PFOA



2: 13C-PFOA



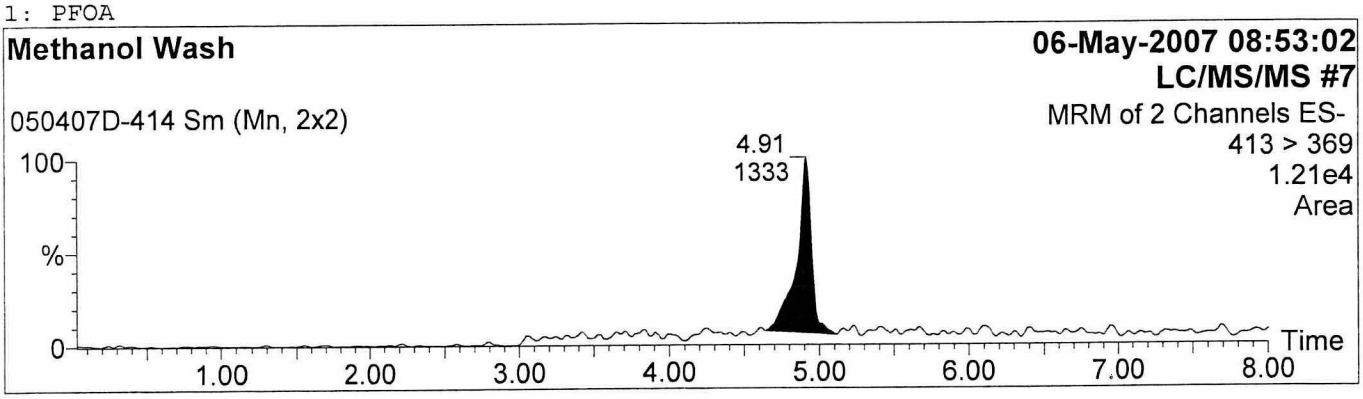
Quantify Sample Report
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

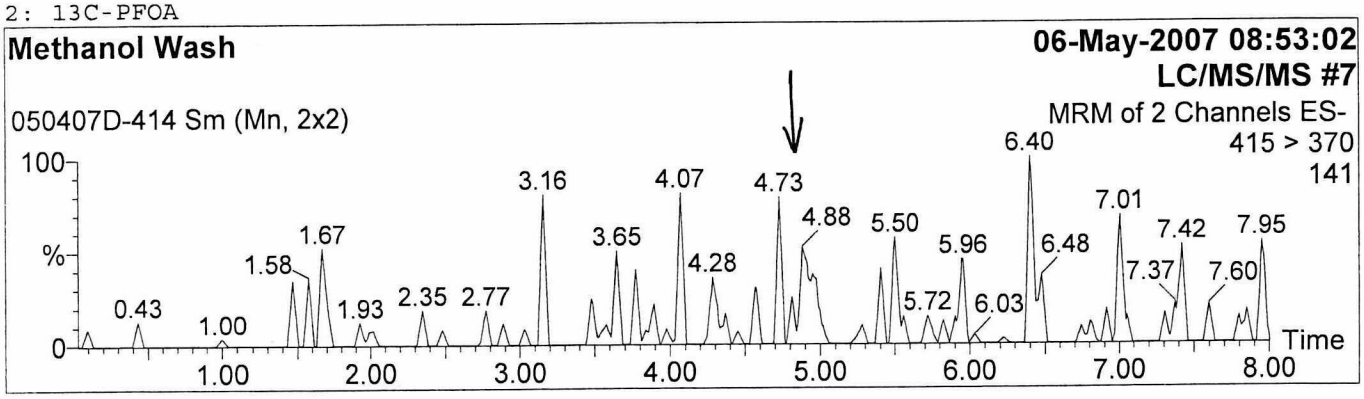
Printed: Thu May 10 10:56:27 2007

Name: 050407D-414
Text:

1: PFOA



2: 13C-PFOA



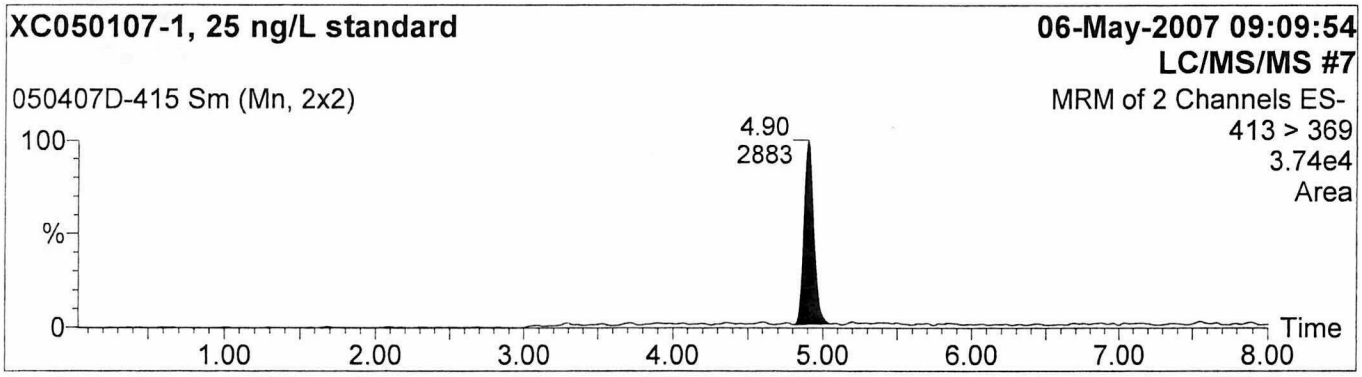
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

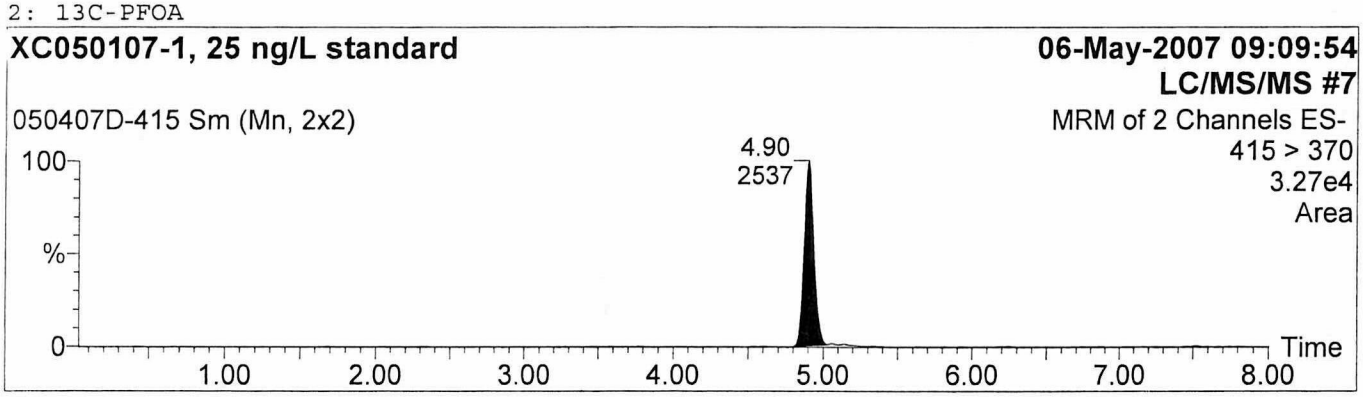
Printed: Thu May 10 10:56:27 2007

Name: 050407D-415
Text:

1: PFOA



2: 13C-PFOA



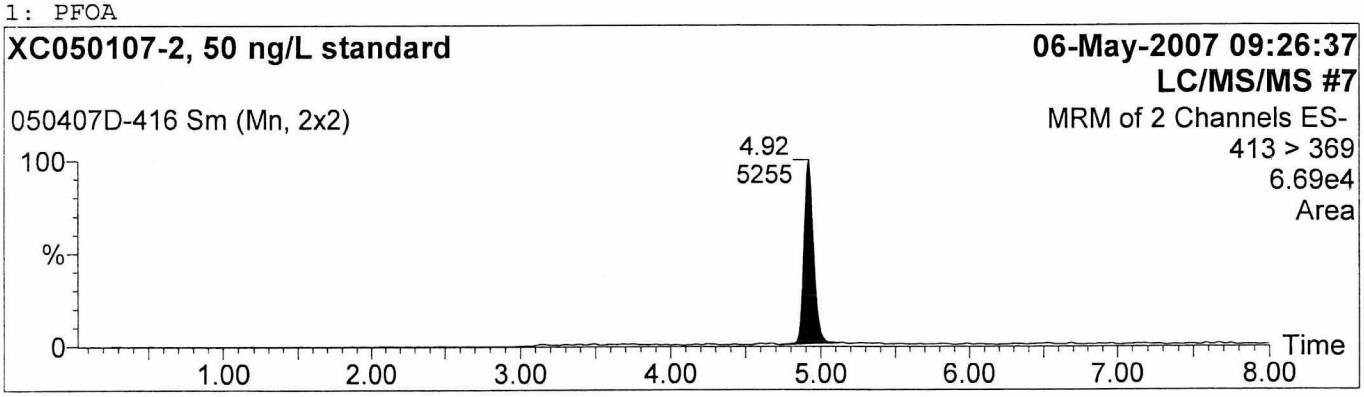
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

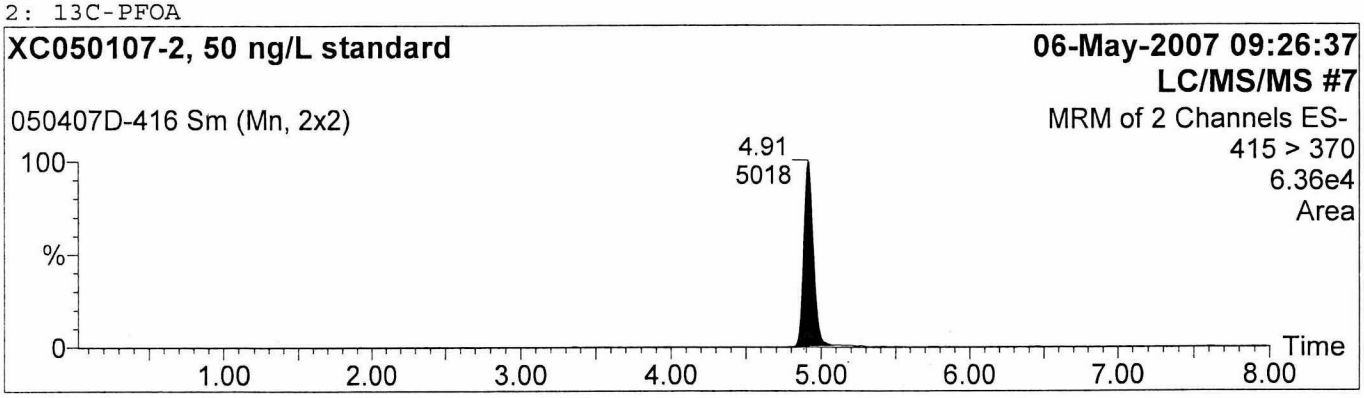
Printed: Thu May 10 10:56:27 2007

Name: 050407D-416
Text:

1: PFOA



2: 13C-PFOA



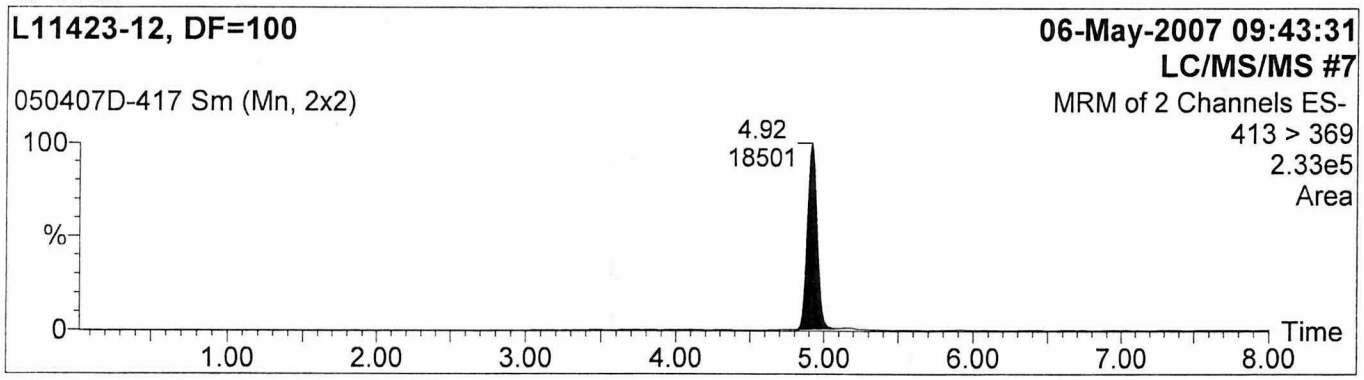
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

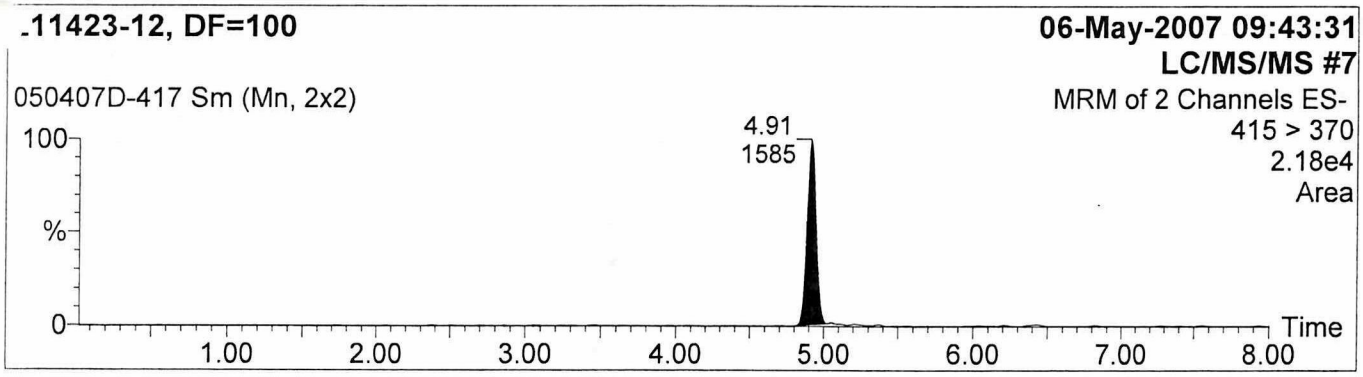
Printed: Thu May 10 10:56:27 2007

Name: 050407D-417
Text:

1: PFOA



2: 13C-PFOA



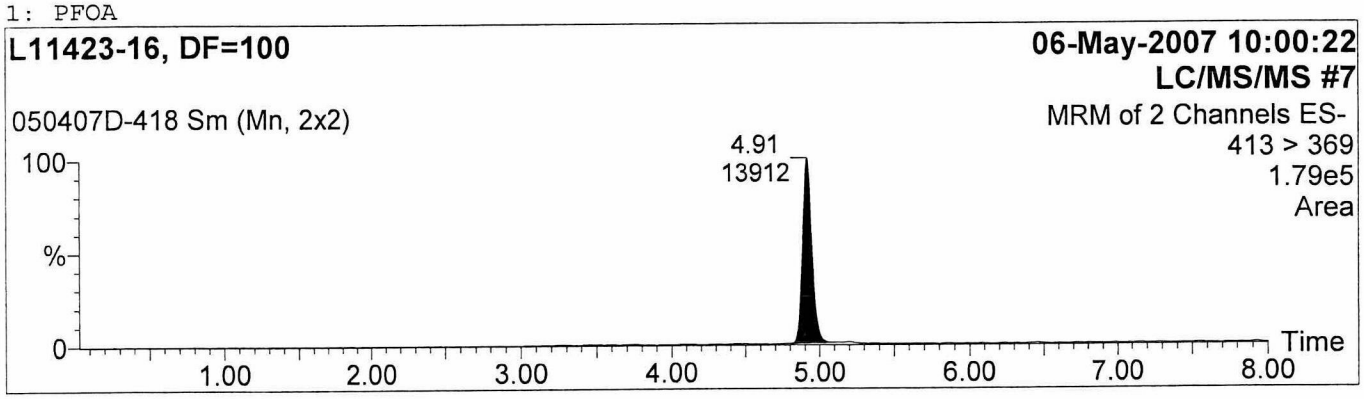
Quantify Sample Report
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

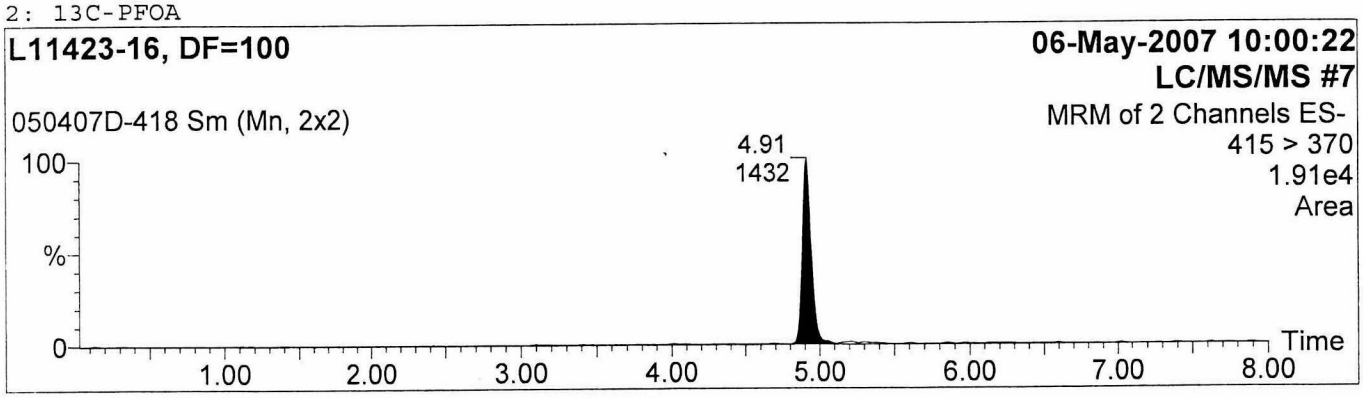
Printed: Thu May 10 10:56:27 2007

Name: 050407D-418
Text:

1: PFOA



2: 13C-PFOA



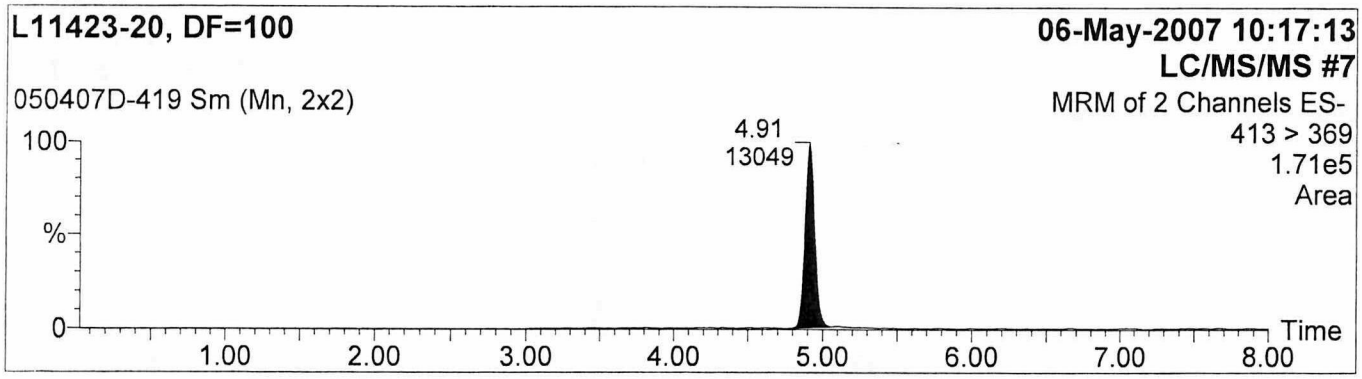
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

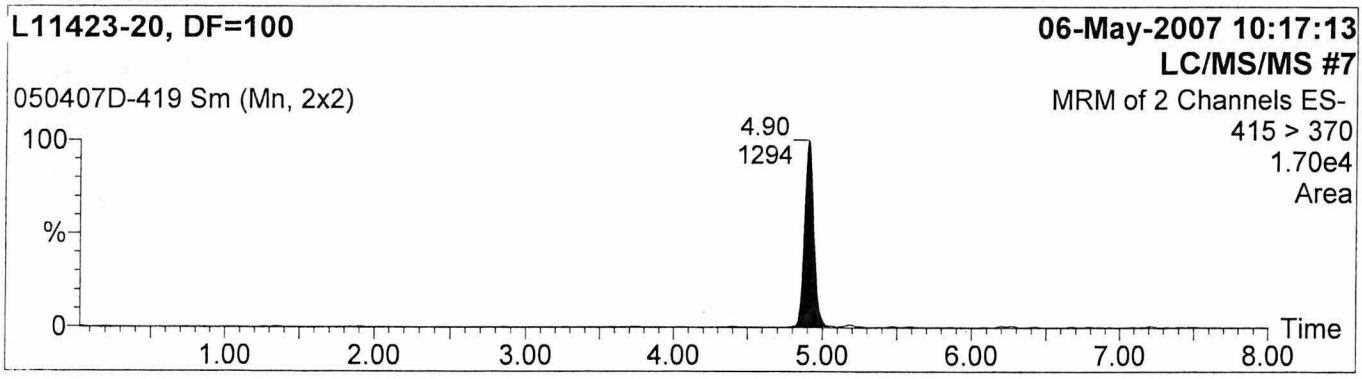
Printed: Thu May 10 10:56:27 2007

Name: 050407D-419
Text:

1: PFOA



2: 13C-PFOA



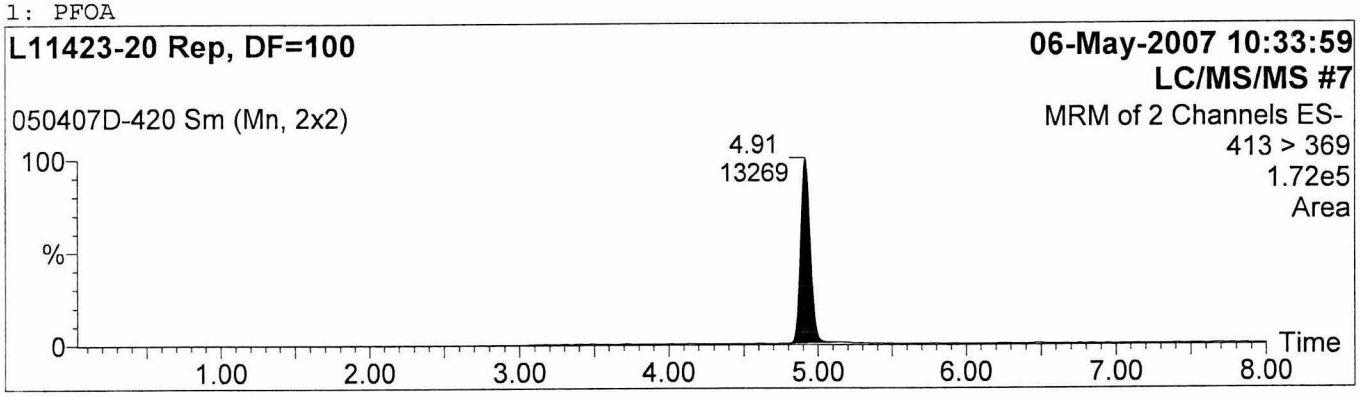
Quantify Sample Report
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

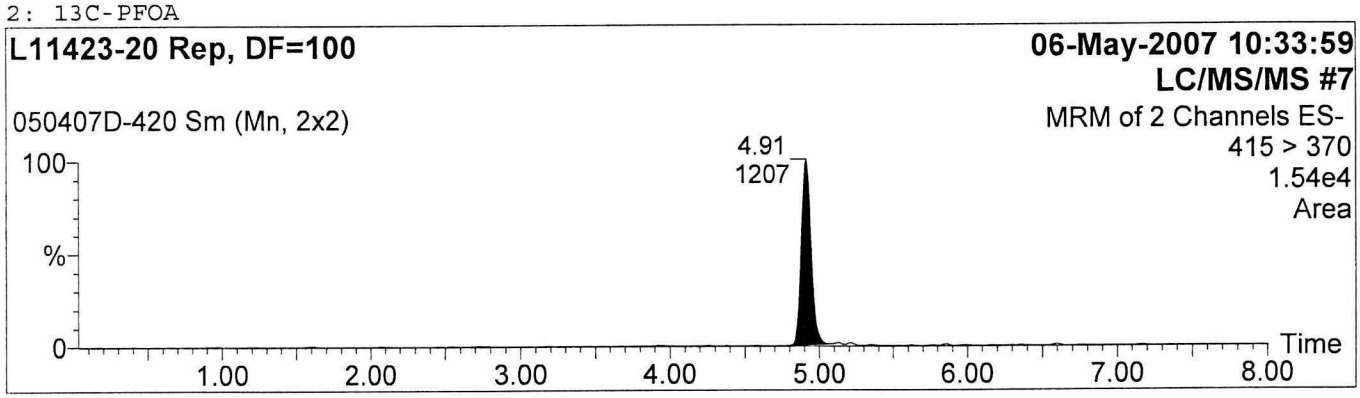
Printed: Thu May 10 10:56:27 2007

Name: 050407D-420
Text:

1: PFOA



2: 13C-PFOA



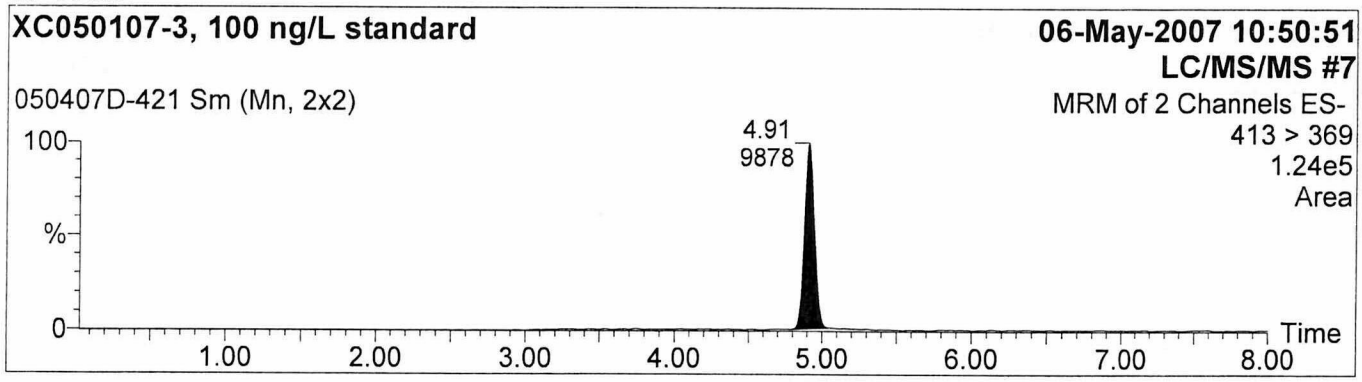
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

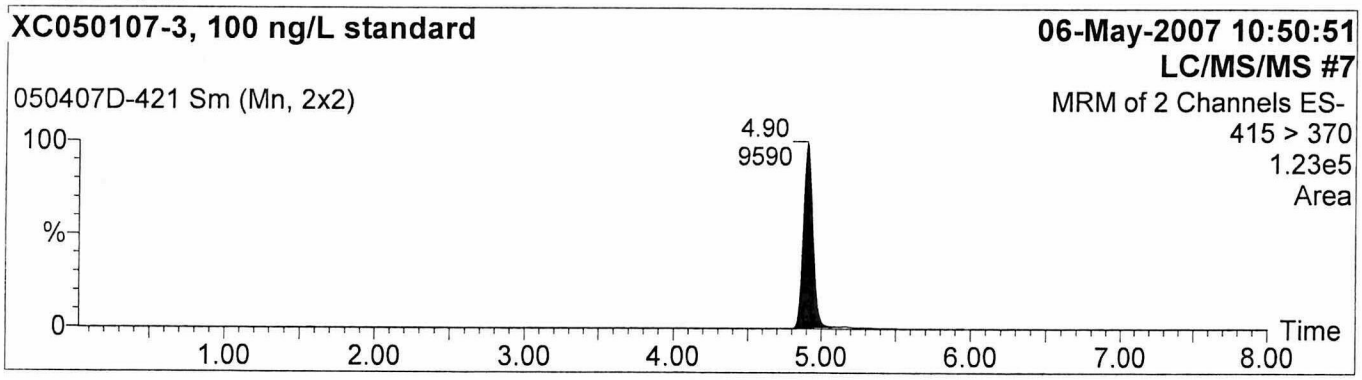
Printed: Thu May 10 10:56:27 2007

Name: 050407D-421
Text:

1: PFOA



2: 13C-PFOA



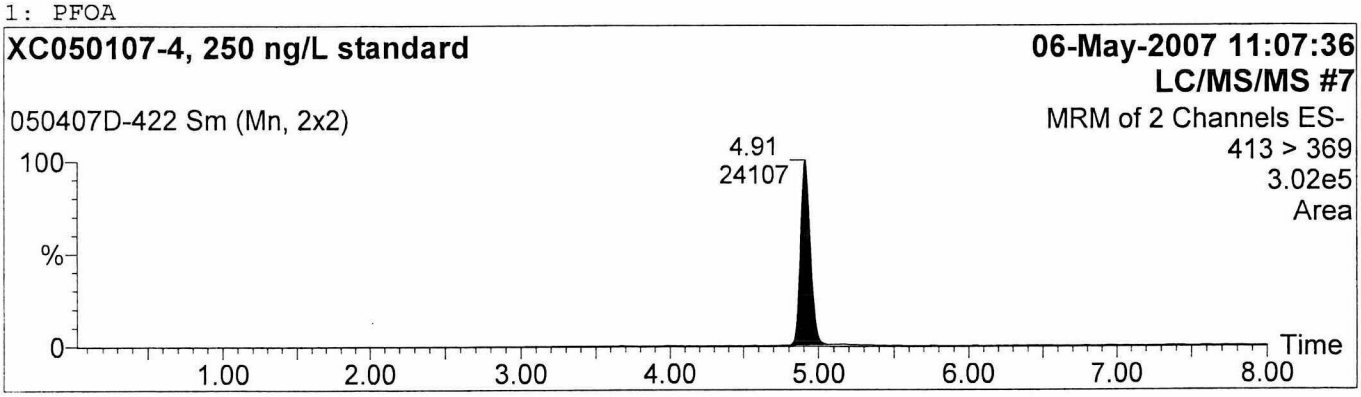
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

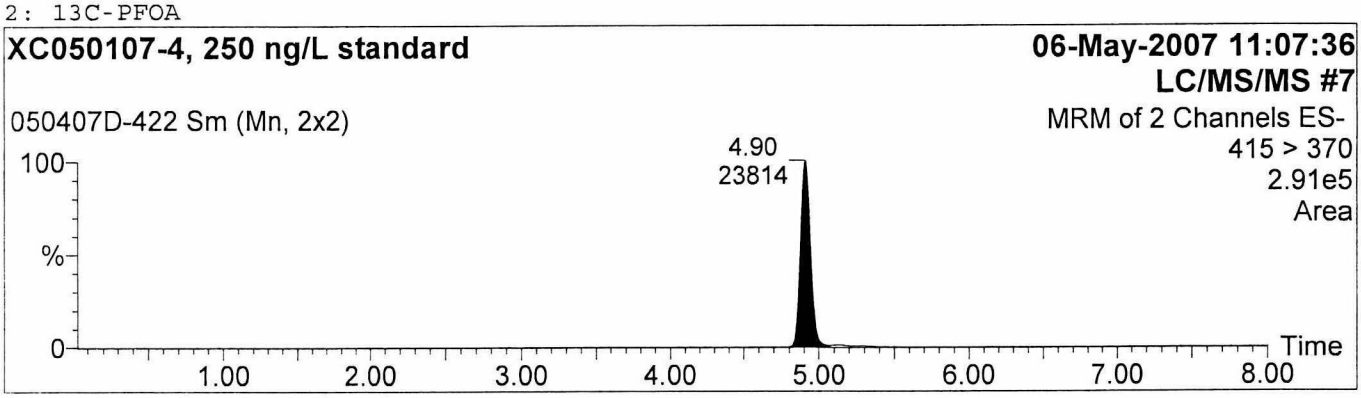
Printed: Thu May 10 10:56:27 2007

Name: 050407D-422
Text:

1: PFOA



2: 13C-PFOA



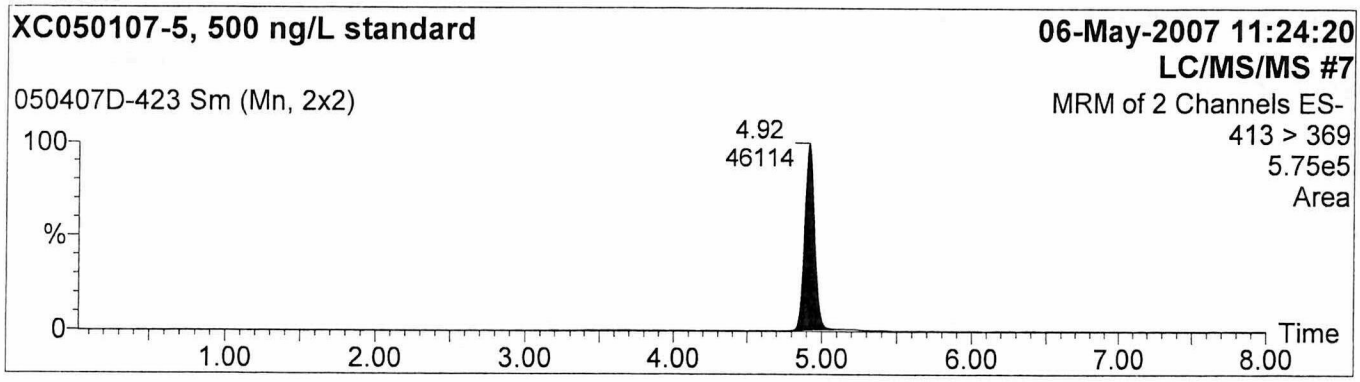
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

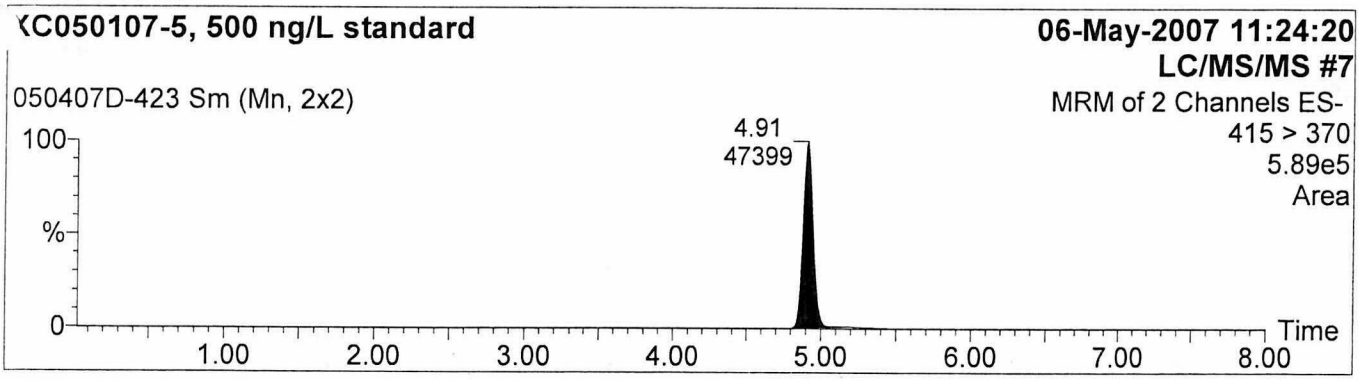
Printed: Thu May 10 10:56:27 2007

Name: 050407D-423
Text:

1: PFOA



2: 13C-PFOA



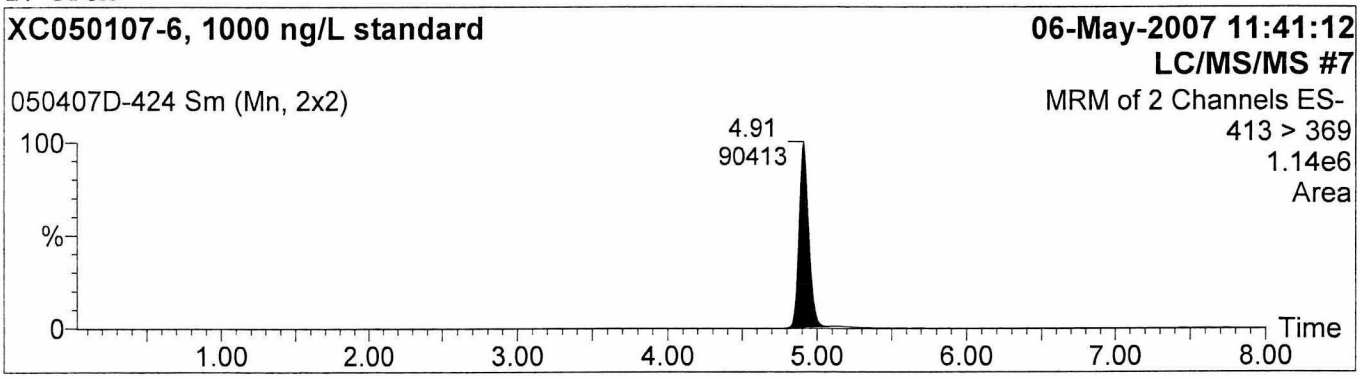
Study No: L11423, Set No: 050407D. Ext.Date: 05/02&04/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050407D MeOH Rinse
Last modified: Thu May 10 10:52:10 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\050307 PFOA & C13PFOA
Last modified: Thu May 03 10:45:11 2007
Job Code:

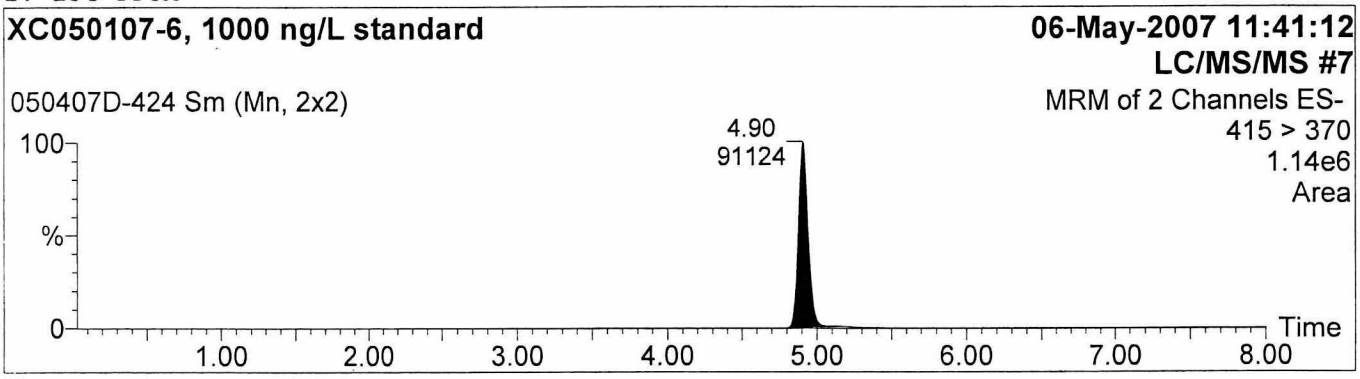
Printed: Thu May 10 10:56:27 2007

Name: 050407D-424
Text:

1: PFOA



2: 13C-PFOA



7.5 Attachment E: Dispersion Analytical Data

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	50 ng/L	Set No:	050207D
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	Dispersions	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	0.4 mL	Extraction Date:	05/02/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/13/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050207D-601	0	-	-	0	-	-	-	-
XC050107-1	-	CS	050207D-602	25	-	-	2931	-	-	-	-
XC050107-2	-	CS	050207D-603	50	-	-	5431	-	-	-	-
XC050107-3	-	CS	050207D-604	100	-	-	11171	-	-	-	-
XC050107-4	-	CS	050207D-605	250	-	-	26493	-	-	-	-
XC050107-5	-	CS	050207D-606	500	-	-	55539	-	-	-	-
XC050107-6	-	CS	050207D-607	1000	-	-	103522	-	-	-	-
Methanol Wash	-	C	050207D-608	-	-	-	0	-	-	-	-
Reagent Control	NA	C	050207D-609	-	-	1	0	ND	ND	-	-
Reagent Spk A	NA	LCS	050207D-610	-	-	1	5932	52.2	54.3	50	104
Reagent Spk B	NA	LCS	050207D-611	-	-	1	55164	507	527	500	101
L11423-21 Spk C	SG-L-001	LF	050207D-612	-	225	10000000	51766	^	-	5000000000	-
L11423-21	SG-L-001	S	050207D-613	-	225	10000000	151	*	-	-	-
L11423-21	SG-L-001	S	050207D-614	-	225	1000000	969	*	-	-	-
L11423-21	SG-L-001	S	050207D-615	-	225	10000	88389	8130000	8460000	-	-
Methanol Wash	-	C	050207D-616	-	-	-	0	-	-	-	-
XC050107-1	-	CS	050207D-617	25	-	-	2961	-	-	-	-
XC050107-2	-	CS	050207D-618	50	-	-	5713	-	-	-	-
XC050107-3	-	CS	050207D-619	100	-	-	11715	-	-	-	-
L11423-21 Rep	SG-L-001	S	050207D-620	-	225	10000000	181	*	-	-	-
L11423-21 Rep	SG-L-001	S	050207D-621	-	225	1000000	1068	*	-	-	-
L11423-21 Rep	SG-L-001	S	050207D-622	-	225	10000	92455	8510000	8860000	-	-
L11423-22	SG-L-002	S	050207D-623	-	235	10000000	239	*	-	-	-
L11423-22	SG-L-002	S	050207D-624	-	235	1000000	931	*	-	-	-
L11423-22	SG-L-002	S	050207D-625	-	235	10000	90707	8350000	8690000	-	-
Methanol Wash	-	C	050207D-626	-	-	-	0	-	-	-	-
XC050107-4	-	CS	050207D-627	250	-	-	27733	-	-	-	-
XC050107-5	-	CS	050207D-628	500	-	-	56783	-	-	-	-
XC050107-6	-	CS	050207D-629	1000	-	.	110500	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 277.394
Slope = 108.354
Coef. Of Det. = 0.997700

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 25 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 50 ng/L (LOQ)

*Sample was analyzed at several dilution levels in this data set. The appropriate results are reported.
^Sample requires a lower spiking level. For re-extraction and analysis see data set 051607A.

Spreadsheet prepared by: MLC / 5/15/07

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	50 ng/L	Set No:	050207D
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	^{13}C-PFOA	Matrix:	Dispersions	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	415 -> 370	Sample Volume Extracted:	0.4 mL	Extraction Date:	05/02/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/13/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	13C-PFOA Found (ng/L)	Amount 13C-PFOA Added (ng/L)	Recovery (%)
XC050107-0	-	CS	050207D-601	0	-	-	0	-	-	-
XC050107-1	-	CS	050207D-602	25	-	-	2711	-	-	-
XC050107-2	-	CS	050207D-603	50	-	-	5337	-	-	-
XC050107-3	-	CS	050207D-604	100	-	-	10504	-	-	-
XC050107-4	-	CS	050207D-605	250	-	-	25969	-	-	-
XC050107-5	-	CS	050207D-606	500	-	-	55903	-	-	-
XC050107-6	-	CS	050207D-607	1000	-	-	103336	-	-	-
Methanol Wash	-	C	050207D-608	-	-	-	0	-	-	-
Reagent Control	NA	C	050207D-609	-	-	1	0	ND	-	-
Reagent Spk A	NA	LCS	050207D-610	-	-	1	5450	50.0	50	100
Reagent Spk B	NA	LCS	050207D-611	-	-	1	54193	506	500	101
L11423-21 Spk C	SG-L-001	LF	050207D-612	-	225	10000000	44002	^	5000000000	-
L11423-21	SG-L-001	S	050207D-613	-	225	10000000	117	*	5000000	-
L11423-21	SG-L-001	S	050207D-614	-	225	1000000	522	*	5000000	-
L11423-21	SG-L-001	S	050207D-615	-	225	10000	54326	5070000	5000000	101
Methanol Wash	-	C	050207D-616	-	-	-	0	-	-	-
XC050107-1	-	CS	050207D-617	25	-	-	2812	-	-	-
XC050107-2	-	CS	050207D-618	50	-	-	5362	-	-	-
XC050107-3	-	CS	050207D-619	100	-	-	11226	-	-	-
L11423-21 Rep	SG-L-001	S	050207D-620	-	225	10000000	93	*	5000000	-
L11423-21 Rep	SG-L-001	S	050207D-621	-	225	1000000	536	*	5000000	-
L11423-21 Rep	SG-L-001	S	050207D-622	-	225	10000	54080	5050000	5000000	101
L11423-22	SG-L-002	S	050207D-623	-	235	10000000	134	*	5000000	-
L11423-22	SG-L-002	S	050207D-624	-	235	1000000	614	*	5000000	-
L11423-22	SG-L-002	S	050207D-625	-	235	10000	53269	4980000	5000000	100
Methanol Wash	-	C	050207D-626	-	-	-	0	-	-	-
XC050107-4	-	CS	050207D-627	250	-	-	27790	-	-	-
XC050107-5	-	CS	050207D-628	500	-	-	54208	-	-	-
XC050107-6	-	CS	050207D-629	1000	-	.	107463	-	-	-

13C-PFOA Found (ng/L) = (peak area - intercept) / slope x DF

Recovery (%) = $\frac{\text{13C-PFOA found (ng/L)}}{\text{amount 13C-PFOA added (ng/L)}} \times 100$

Standard Curve : Linear (1/x weighted)
Intercept = 104.347
Slope = 106.849
Coef. Of Det. = 0.998650

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 25 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 50 ng/L (LOQ)

*Sample was analyzed at several dilution levels in this data set. The appropriate results are reported.
^Sample requires a lower spiking level. For re-extraction and analysis see data set 051607A.

Spreadsheet prepared by: MLC / 5/15/07

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

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423 Matrix: Water
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 34
Time 8:25 AM Date 5/02/2007 Initials EDD

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-L-001	L11423-21 Spk C*	225 mL	40.0	20000.0
SG-L-001	L11423-21*	225 mL	40.0	-
SG-L-001	L11423-21 Rep*	225 mL	40.0	-
SG-L-002	L11423-22*	235 mL	40.0	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
L11423-21 Spk C*	SS20794 (100000 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
L11423-21 Spk C*	SS18238 (100000 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
All samples	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/2/07
-	-	-	-
-	-	-	-

All samples were measured:
Time 10:25 AM Date 5/02/2007 Initials EDD

After measuring samples were returned to refrigerator No. 34
Time 3:30 PM Date 5/02/2007 Initials EDD

Comments: *Sample is pre-diluted 10000 times by taking 0.4 mL of sample and bringing the volume up to 40 mL with HPLC water, then taking 0.4 mL of that solution and bringing the volume up to 40 mL.
Spiking is done on a 10000x diluted sample for Spk C.

Analysis Summary:
Data Set: 050207D Initials/Date: MLC / 5/11/07
Data Set: - Initials/Date: - / -
Data Set: - Initials/Date: - / -

Set extraction/analysis data verified by: CEE Date: 05/24/07

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

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007A$$ PROTOCOL NUMBER: NA

MATRIX: Water ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot # or Exygen ID
na	Reagent Control	↓	↓	–	↓	–	–	Methanol	46303
na	Reagent Spk A			–		–	–	C18 SPE	0053370818
na	Reagent Spk B			–		–	–	HPLC Water	46335
SG-L-001	L11423-21 Spk C*			①0.1/10, 0.1/1.0		–	–	-	-
SG-L-001	L11423-21*			②0.1/10, 0.1/1.0		–	–	-	-
SG-L-001	L11423-21 Rep*			②0.1/10, 0.1/1.0		–	–	-	-
SG-L-002	L11423-22*			②0.1/10, 0.1/1.0		–	–	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	*Initials/Date*	EDD 5/2/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C44404
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date	-	EDD 5/2/07	EDD 5/2/07	MLC 5/2/07	MLC 5/11/07	–	–	*Initials/Date*	MLC 5/11/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

①DF = 1000
②DF = 100 AND 1000 > MLC 5/2/07

*Sample was diluted 10000 times prior to extraction.
**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 by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:

Final extracts stored in refrigerator 32 Initials: EDD Date: 5/2/2007

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

Exygen STUDY NO: L11423

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Protocol No.: None
Method No.: $$
Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA
Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC050107-0	Methanol	46803
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC050107-1	C18 SPE	0053370818
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC050107-2	Water	46335
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC050107-3	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC050107-4	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC050107-5	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC050107-6	-	-
-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/01/07

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) / freezer # 34 Time: 9:10 AM — Initials/Date: EDD 1 5/01/07

40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD 1 5/01/07

After measuring, samples returned to (refrigerator) / freezer # 34 Time: 11:10 AM — Initials/Date: EDD 1 5/01/07

Samples fortified: Initials/Date: AMB 1 05/01/07

SPE clean-up: Initials/Date: EDD 1 5/01/07

Final volume adjusted to 5 mL: Initials/Date: EDD 1 5/01/07

Extracts placed in refrigerator # 32 Initials/Date: EDD 1 5/01/07

Comments:

STANDARD EXPIRATION DATE: 5/15/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/15/07

Masslynx - Sample List

Exygen STUDY NO. L11423

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050207D Dispersion Water.SPL
Printed: Fri May 11 16:06:56 2007 MLC 5/11/07

Page Position: (1, 1)

L (RE) MLC 5/11/07

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/~~mL~~L)	Conc B	Conc C
1	94	050207D-601	---	---	XC050107-0, 0 ng/L standard	---	Standard	0	0	---
2	95	050207D-602	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---
3	96	050207D-603	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---
4	97	050207D-604	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---
5	98	050207D-605	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---
6	99	050207D-606	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---
7	100	050207D-607	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---
8	92	050207D-608	---	---	Methanol Wash	---	Blank	---	---	---
9	83	050207D-609	---	---	Reagent Control	---	Blank	---	---	---
10	84	050207D-610	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---
11	85	050207D-611	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500	---
12	86	050207D-612	---	---	L11423-21 Spk C, 5,000,000,000 ng/L, DF=10,000,000	---	QC	5000000000	5000000000	---
13	87	050207D-613	---	---	L11423-21, DF=10,000,000	---	Analyte	---	5000000	---
14	88	050207D-614	---	---	L11423-21, DF=1,000,000	---	Analyte	---	5000000	---
15	89	050207D-615	---	---	L11423-21, DF=10,000	---	Analyte	---	5000000	---
16	92	050207D-616	---	---	Methanol Wash	---	Blank	---	---	---
17	95	050207D-617	---	---	XC050107-1, 25 ng/L standard	---	Standard	25	25	---
18	96	050207D-618	---	---	XC050107-2, 50 ng/L standard	---	Standard	50	50	---
19	97	050207D-619	---	---	XC050107-3, 100 ng/L standard	---	Standard	100	100	---
20	90	050207D-620	---	---	L11423-21 Rep, DF=10,000,000	---	Analyte	---	5000000	---
21	29	050207D-621	---	---	L11423-21 Rep, DF=1,000,000	---	Analyte	---	5000000	---
22	30	050207D-622	---	---	L11423-21 Rep, DF=10,000	---	Analyte	---	5000000	---
23	36	050207D-623	---	---	L11423-22, DF=10,000,000	---	Analyte	---	5000000	---
24	37	050207D-624	---	---	L11423-22, DF=1,000,000	---	Analyte	---	5000000	---
25	38	050207D-625	---	---	L11423-22, DF=10,000	---	Analyte	---	5000000	---
26	92	050207D-626	---	---	Methanol Wash	---	Blank	---	---	---
27	98	050207D-627	---	---	XC050107-4, 250 ng/L standard	---	Standard	250	250	---
28	99	050207D-628	---	---	XC050107-5, 500 ng/L standard	---	Standard	500	500	---
29	100	050207D-629	---	---	XC050107-6, 1000 ng/L standard	---	Standard	1000	1000	---

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\050207D Dispersion Water.SPL

Exygen STUDY NO. L 11423

Page Position: (2, 1)

Printed: Fri May 11 16:06:56 2007

MLC 5/11/07

	Test ID	DF	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	0	10000000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	0	10000000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	0	1000000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	0	10000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
20	0	10000000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
21	0	1000000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
22	0	10000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
23	0	10000000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
24	0	1000000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
25	0	10000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
26	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
27	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
28	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
29	0	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA

Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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 Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES**CHROMATOGRAPHY: Part No. FK5962EJ)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/11/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/15/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Fri May 11 16:07:09 2007 MLC 5/11/07

Solvent Delay (mins) : 0.00

Function : 1 **MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Friday, May 11, 2007 15:57:32

Printed: Friday, May 11, 2007 16:07:37 MLC 5/11/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

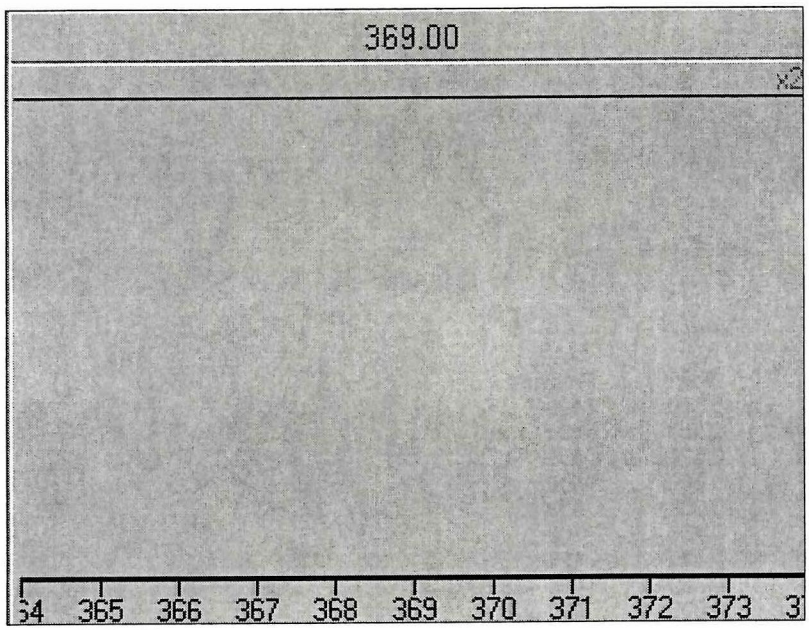
HP1100 Autosampler Initial Conditions

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	96

Tuning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Fri May 11 16:07:47 2007 MLC 5/11/07



Dau 413.00

SOURCE (ESP-)	Set	Rdbk
Capillary	3.00	-2.95
Cone	10	-10
Hexapole 1	0.0	
Aperture 1	0.0	
Hexapole 2	0.0	
Source Block Temp.	100	102
Desolvation Temp.	300	300

Analyser	Set	Rdbk
LM Res 1	14.0	
HM Res 1	14.0	
IEnergy 1	1.0	
Entrance	-2	8
Collision	10	9
Exit	2	12
LM Res 2	14.0	
HM Res 2	14.0	
IEnergy 2	2.0	
Multiplier	700	-695

Pressures	Rdbk
Analyser Vacuum	OFF
Gas Cell	2.3e-3

Gas Flows	Rdbk
Cone Gas	91.1
Desolvation	664.8

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 050207D
Extraction Date: 05/02/07
Analyzed on: 05/13/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2931
XC050107-2	50	5431
XC050107-3	100	11171
XC050107-4	250	26493
XC050107-5	500	55539
XC050107-6	1000	103522

Coefficient of Determination: 0.998724079

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999361836
R Square	0.998724079
Adjusted R Square	0.998405099
Standard Error	1562.729071
Observations	6

MLC
5/15/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	7646260400	7646260400	3130.990151	6.1075E-07
Residual	4	9768488.603	2442122.151		
Total	5	7656028889			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	839.9835221	872.9621033	0.962222208	0.390427758	-1583.747837	3263.714881	-1583.747837	3263.714881
X Variable 1	103.9205708	1.857208544	55.95525132	6.1075E-07	98.76413327	109.0770084	98.76413327	109.0770084

Initial Calibration Curve for ^{13}C-PFOA

Exygen Study No.: L11423
Set No: 050207D
Extraction Date: 05/02/07
Analyzed on: 05/13/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC050107-1	25	2711
XC050107-2	50	5337
XC050107-3	100	10504
XC050107-4	250	25969
XC050107-5	500	55903
XC050107-6	1000	103336

Coefficient of Determination: 0.998238678

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999118951
R Square	0.998238678
Adjusted R Square	0.997798348
Standard Error	1841.861903
Observations	6

MLC 5/15/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	7690770951	7690770951	2267.022066	1.16403E-06
Residual	4	13569821.07	3392455.268		
Total	5	7704340772			

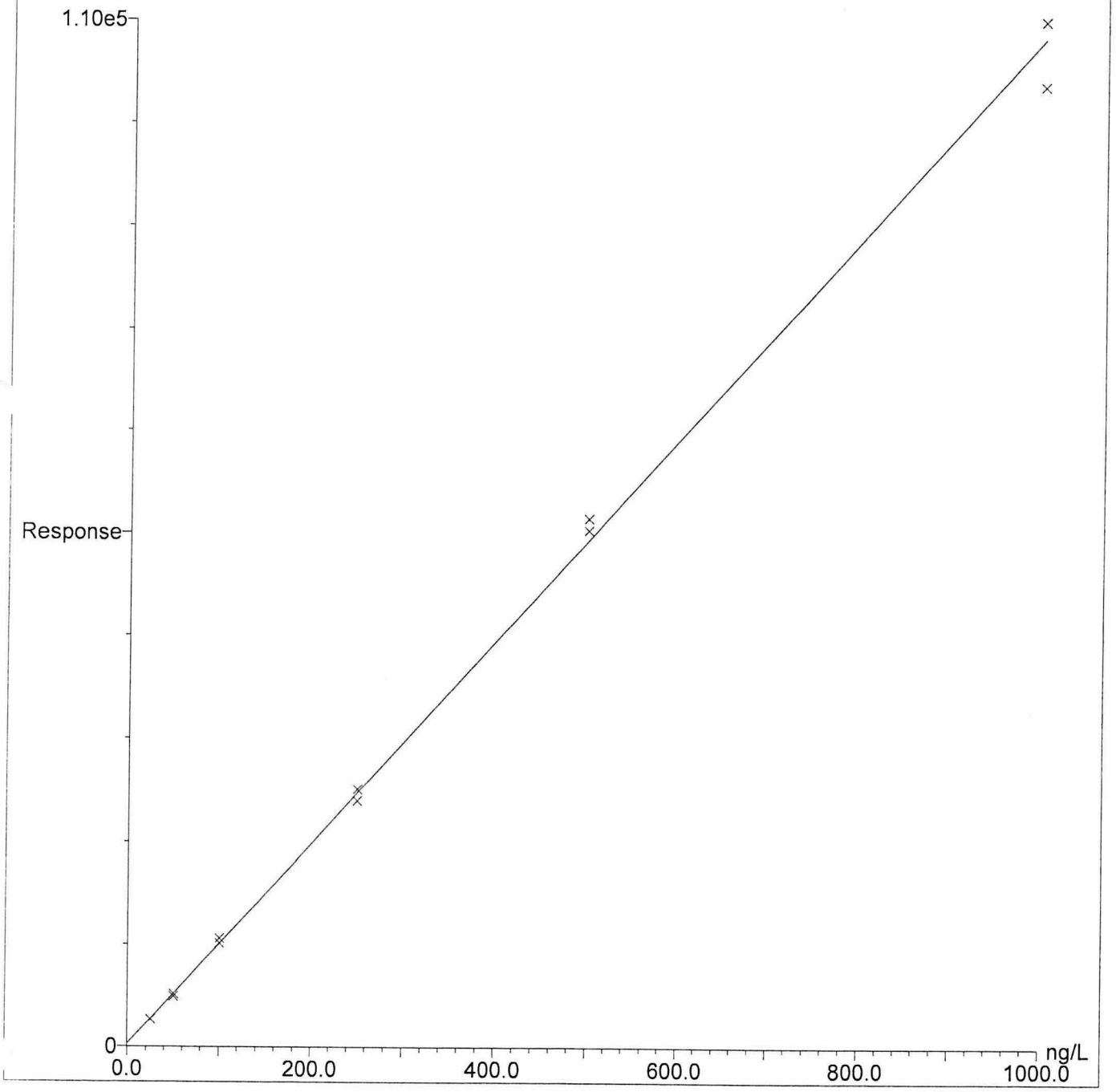
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	521.9145211	1028.889569	0.507259998	0.638660742	-2334.740886	3378.569928	-2334.740886	3378.569928
X Variable 1	104.2226041	2.188940953	47.61325515	1.16403E-06	98.1451297	110.3000785	98.1451297	110.3000785

Quantify Calibration Report
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050207D Dispersion
Last modified: Tue May 15 10:51:46 2007
Printed: Tue May 15 11:13:45 2007

MLC 5/15/07

Compound 1 name: PFOA
Coefficient of Determination: 0.997700
Calibration curve: 108.354 * x + 277.394
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

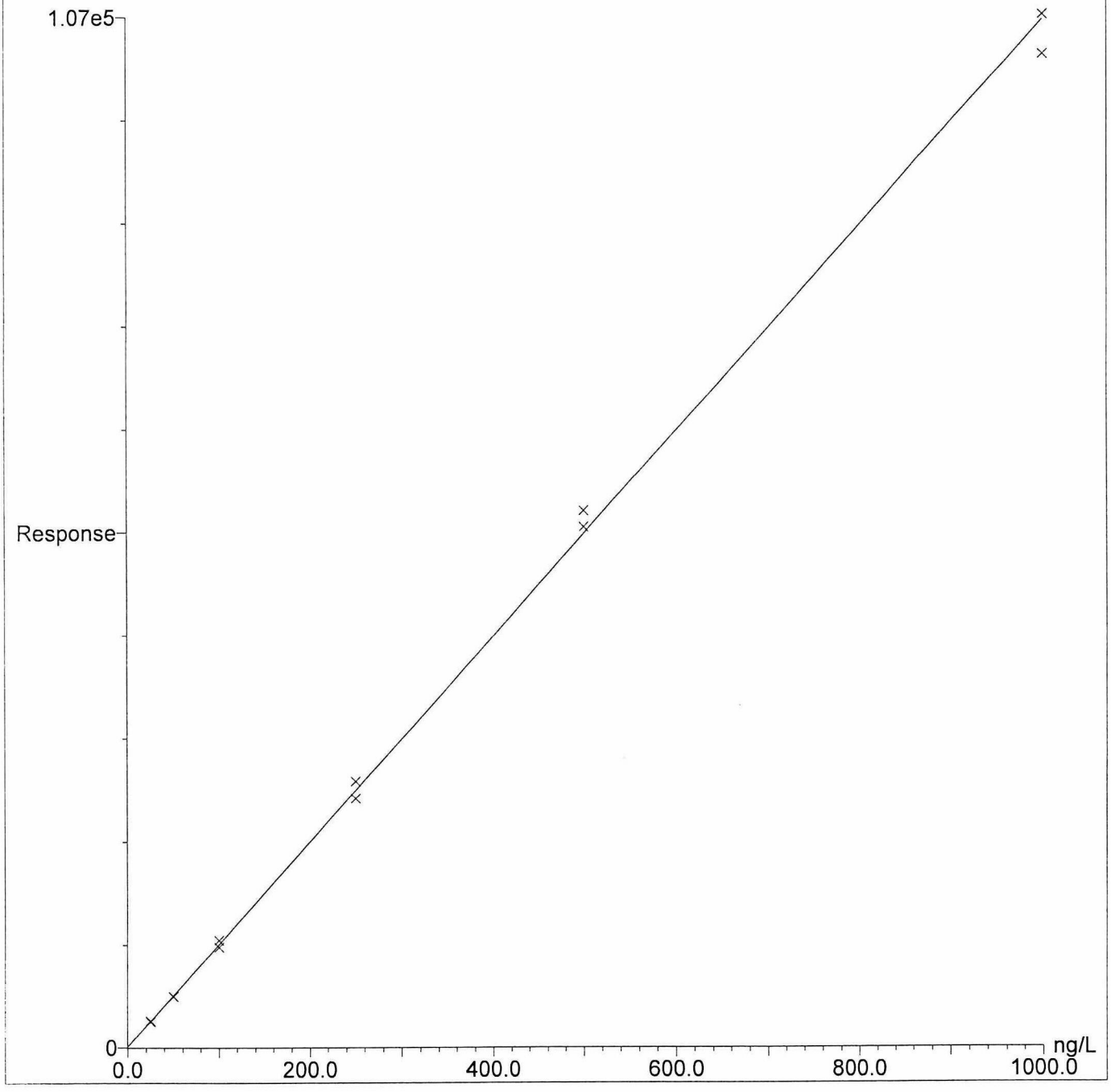


Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\050207D Dispersion
Last modified: Tue May 15 10:51:46 2007
Printed: Tue May 15 11:13:45 2007

MLC 5/15/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.998650
Calibration curve: 106.849 * x + 104.347
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

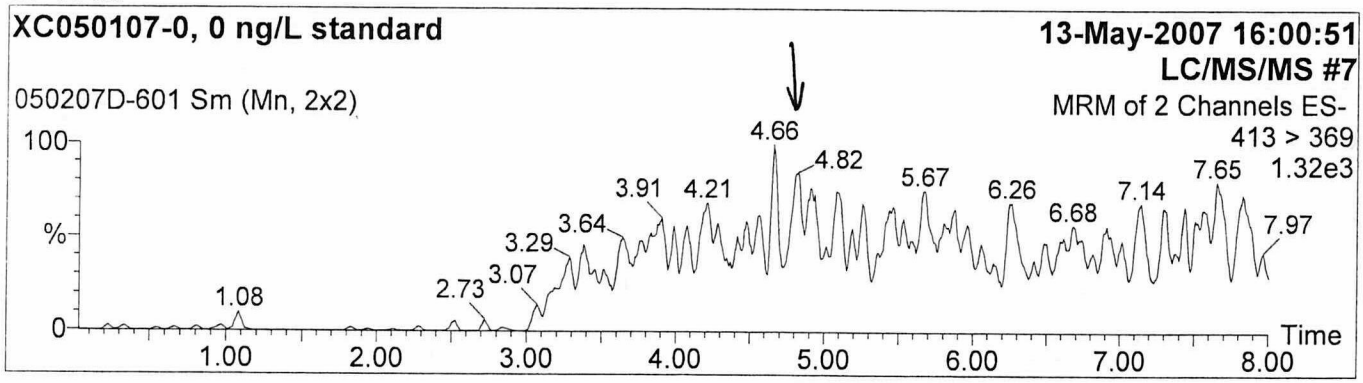
Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

Printed: Tue May 15 11:13:46 2007

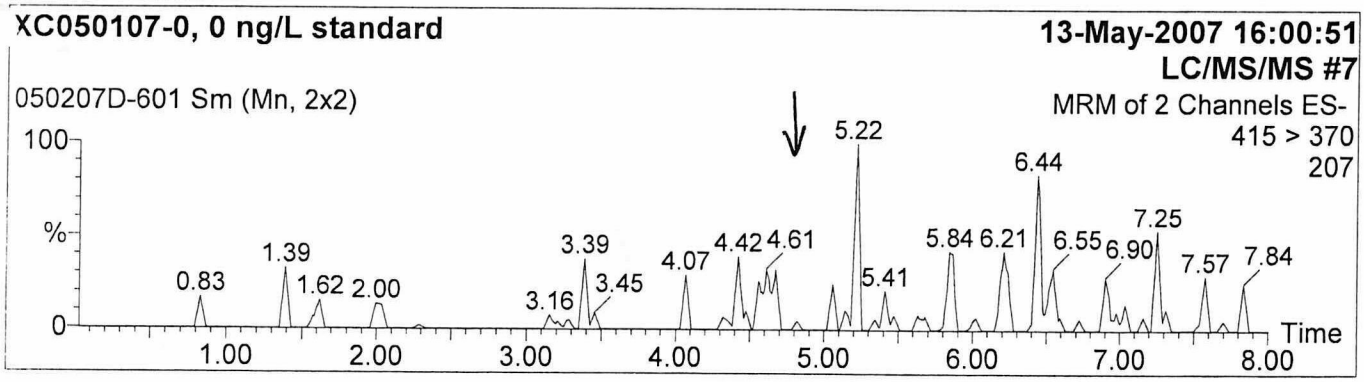
Initials MLC
Date 5/15/07
Run#050207D-601 To 050207D-629

Name: 050207D-601
Text:

1: PFOA



2: 13C-PFOA



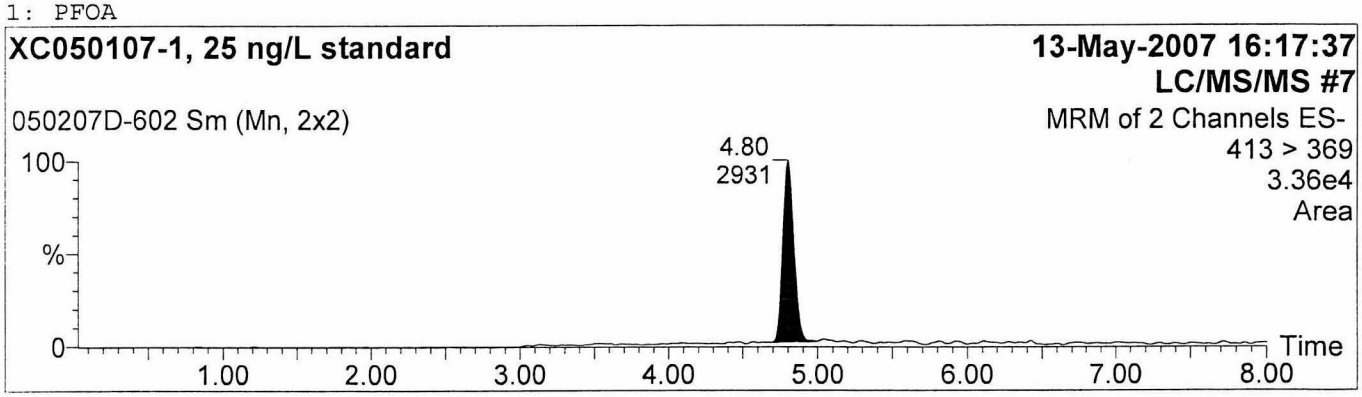
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

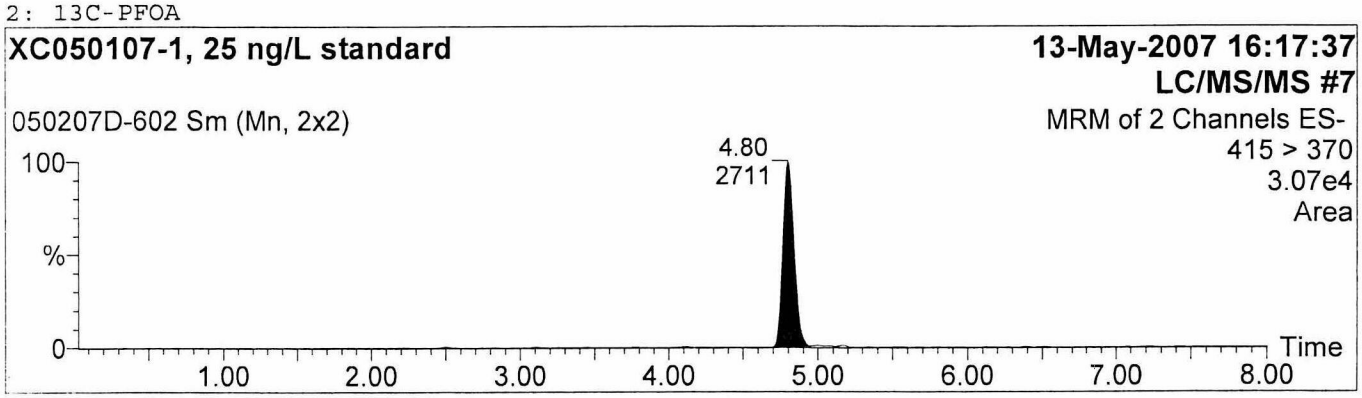
Printed: Tue May 15 11:13:46 2007

Name: 050207D-602
Text:

1: PFOA



2: 13C-PFOA



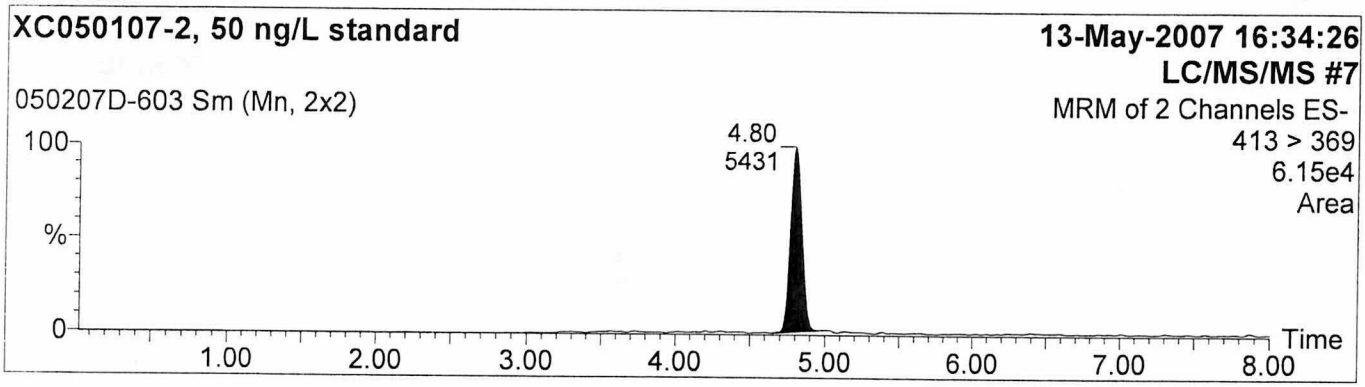
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

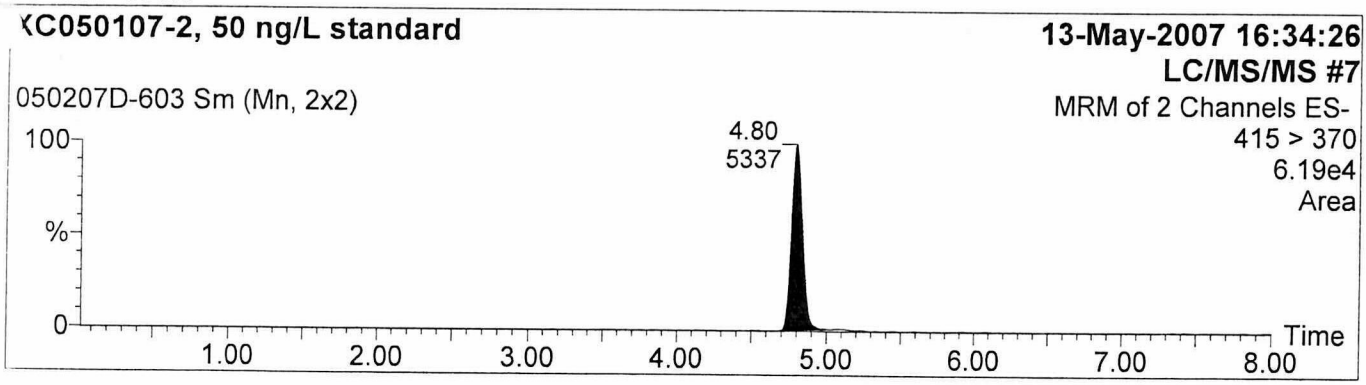
Printed: Tue May 15 11:13:46 2007

Name: 050207D-603
Text:

1: PFOA



2: 13C-PFOA



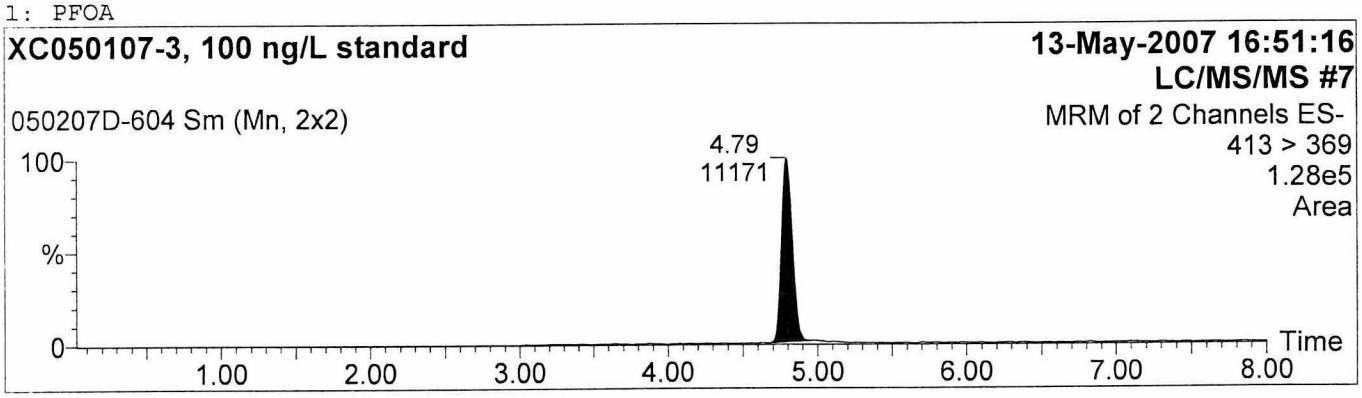
Quantify Sample Report
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

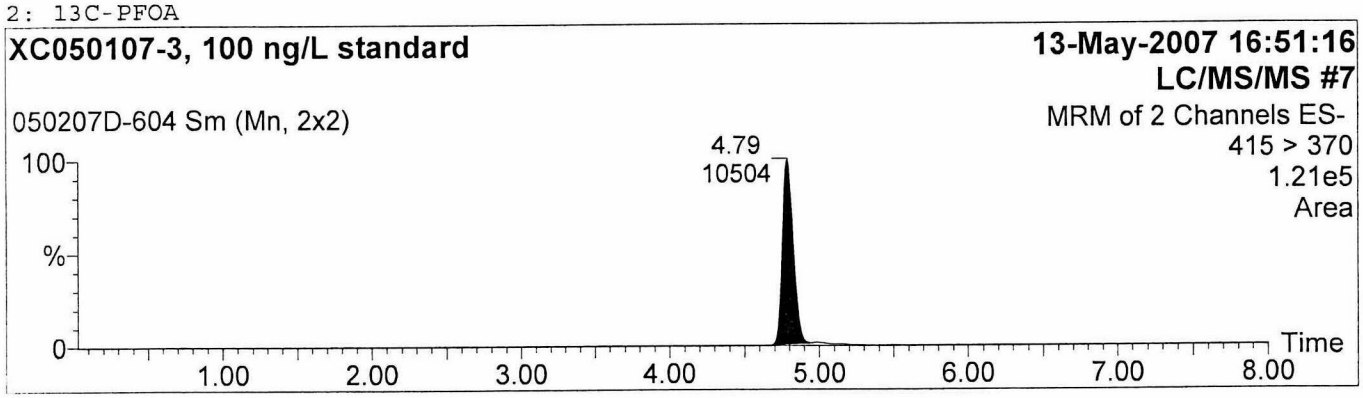
Printed: Tue May 15 11:13:46 2007

Name: 050207D-604
Text:

1: PFOA



2: 13C-PFOA



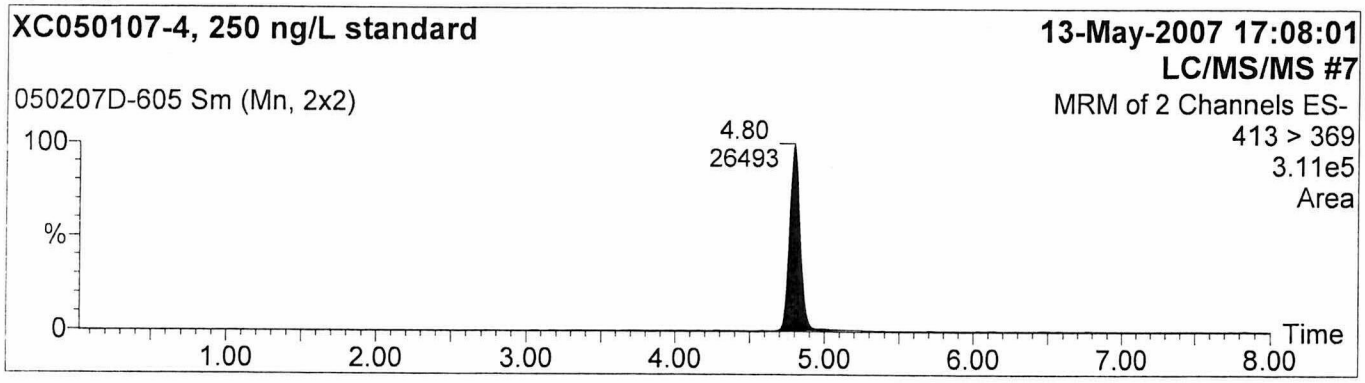
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

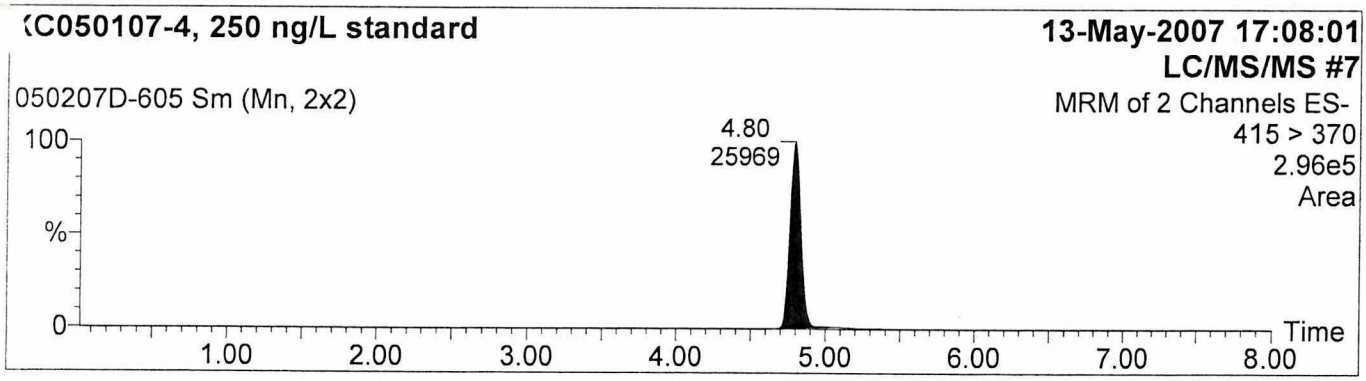
Printed: Tue May 15 11:13:46 2007

Name: 050207D-605
Text:

1: PFOA



2: 13C-PFOA



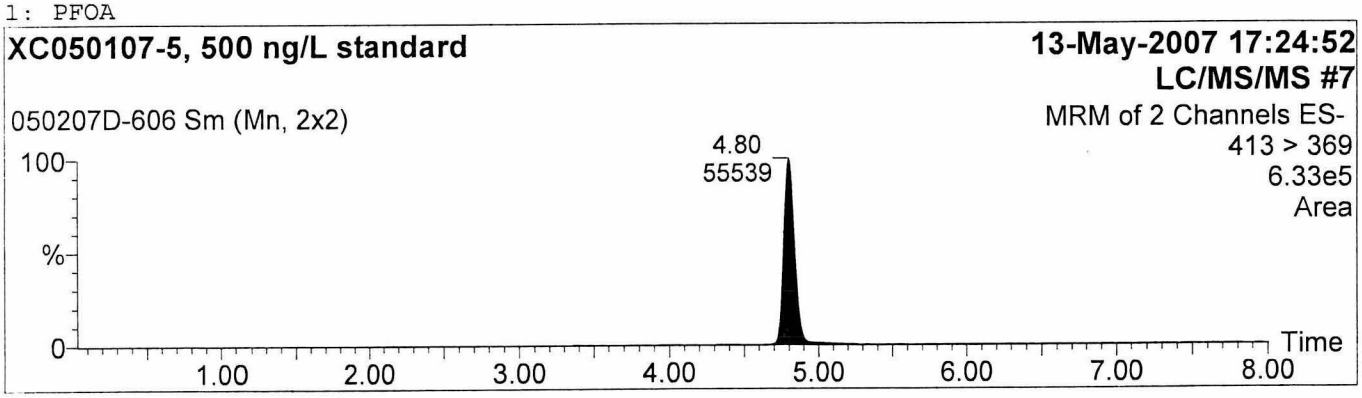
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

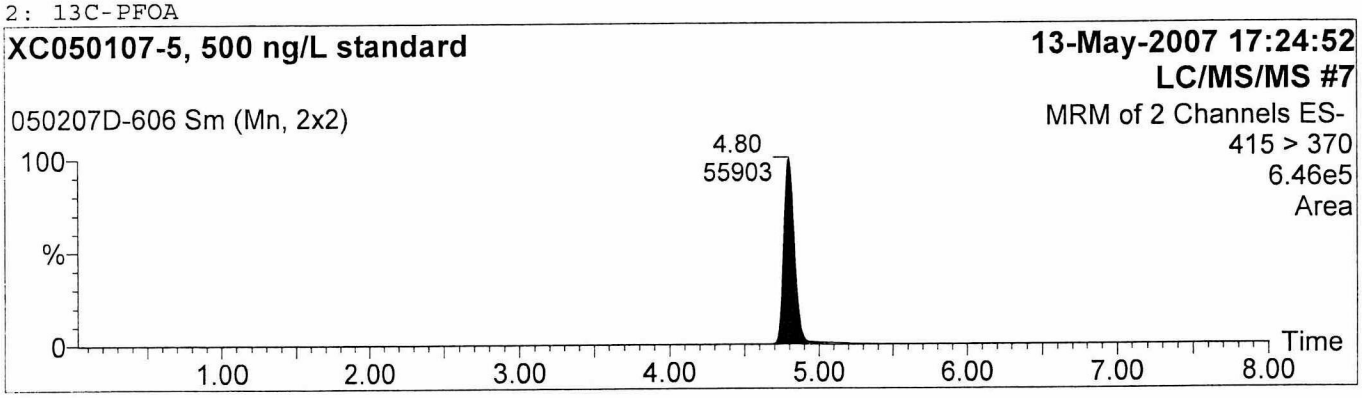
Printed: Tue May 15 11:13:46 2007

Name: 050207D-606
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
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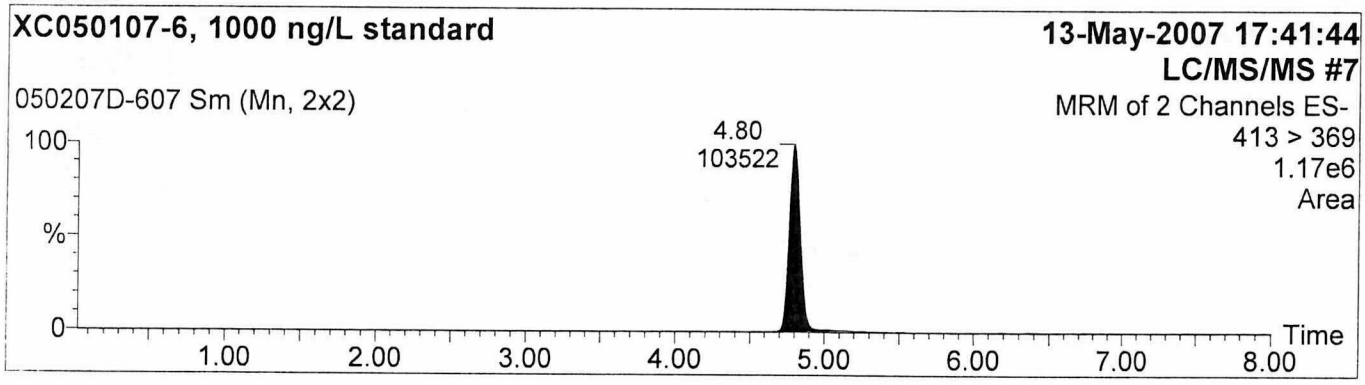
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

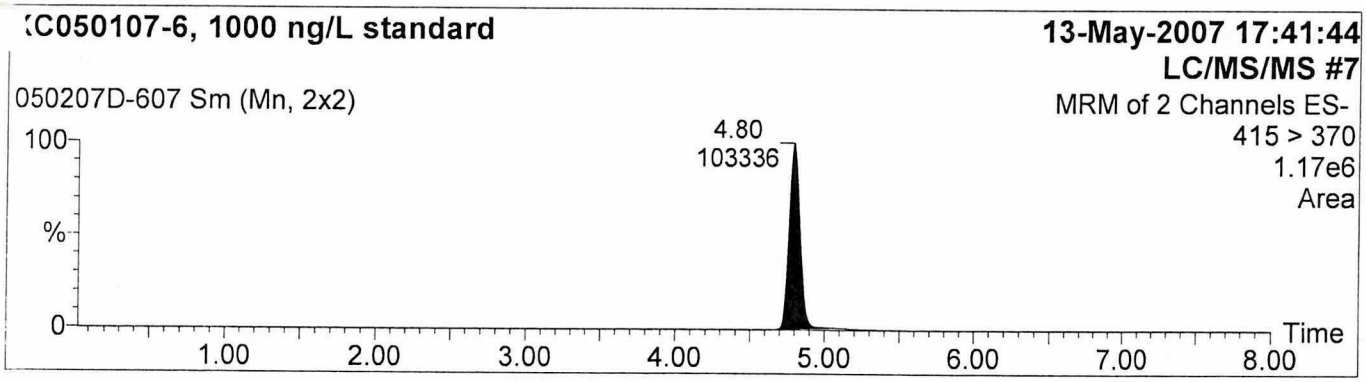
Printed: Tue May 15 11:13:46 2007

Name: 050207D-607
Text:

1: PFOA



2: 13C-PFOA



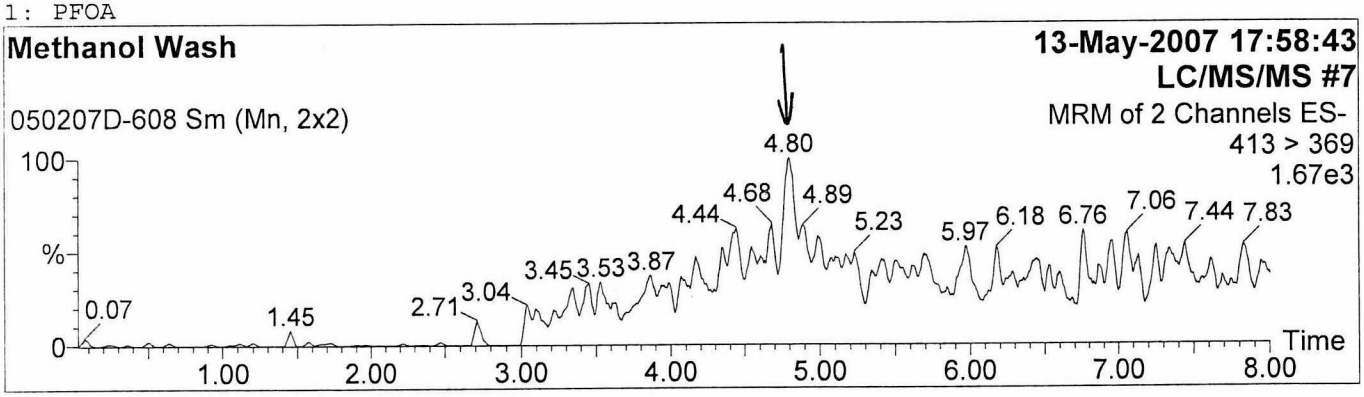
Quantify Sample Report
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

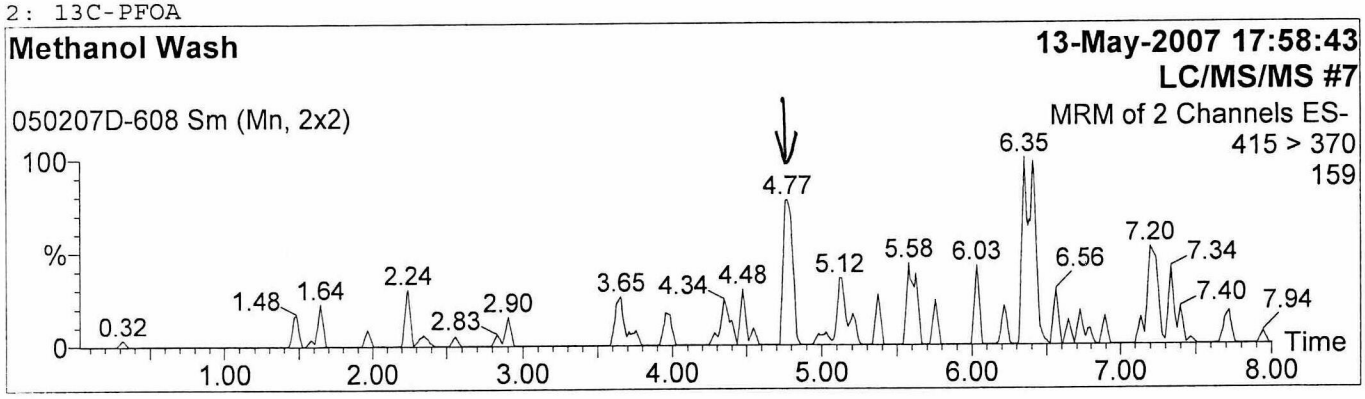
Printed: Tue May 15 11:13:46 2007

Name: 050207D-608
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
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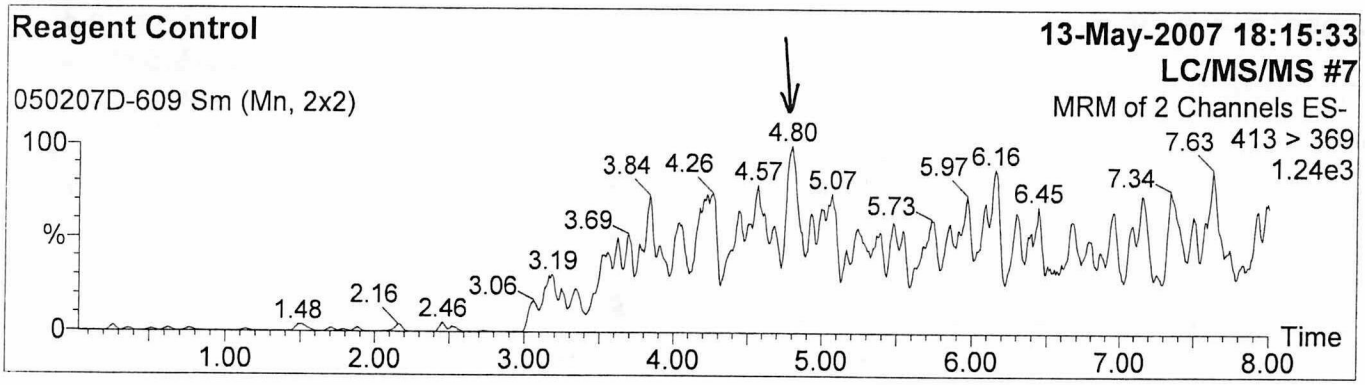
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

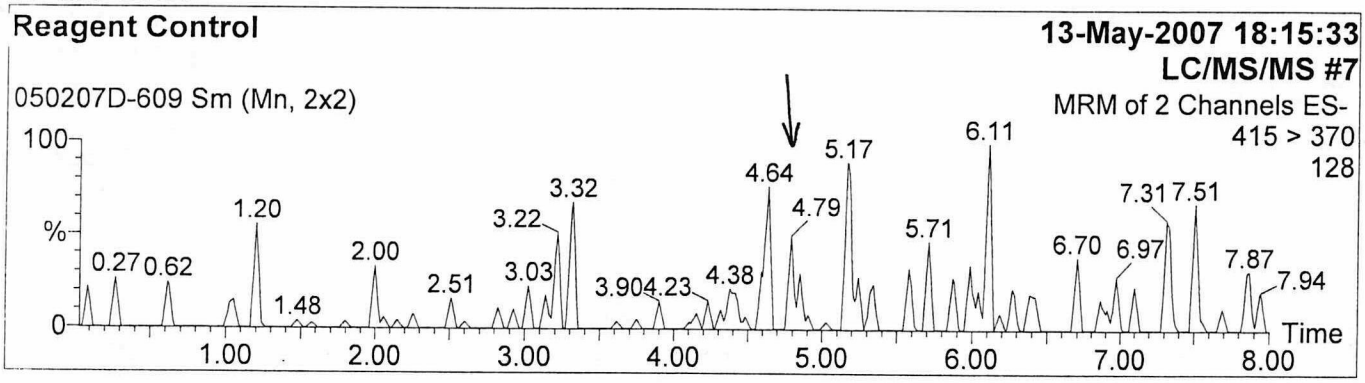
Printed: Tue May 15 11:13:46 2007

Name: 050207D-609
Text:

1: PFOA



2: 13C-PFOA

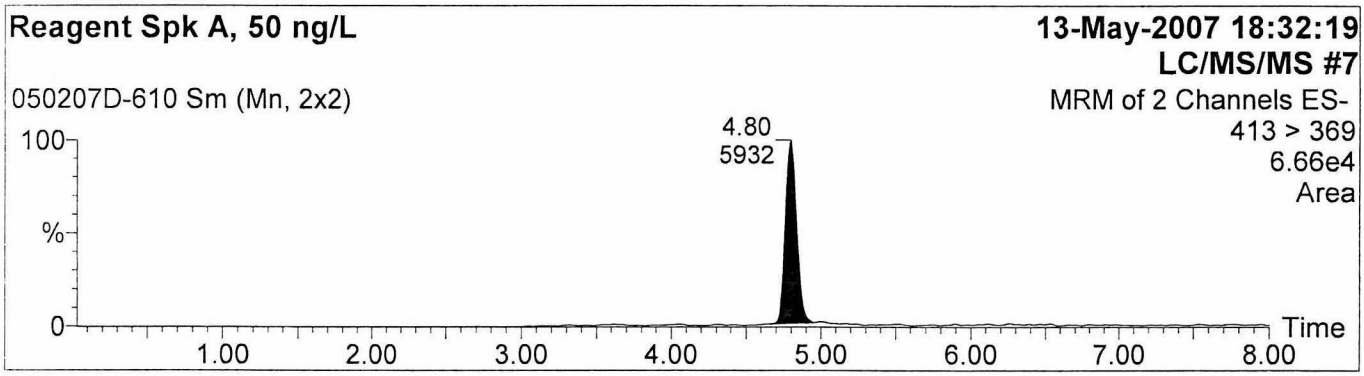


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

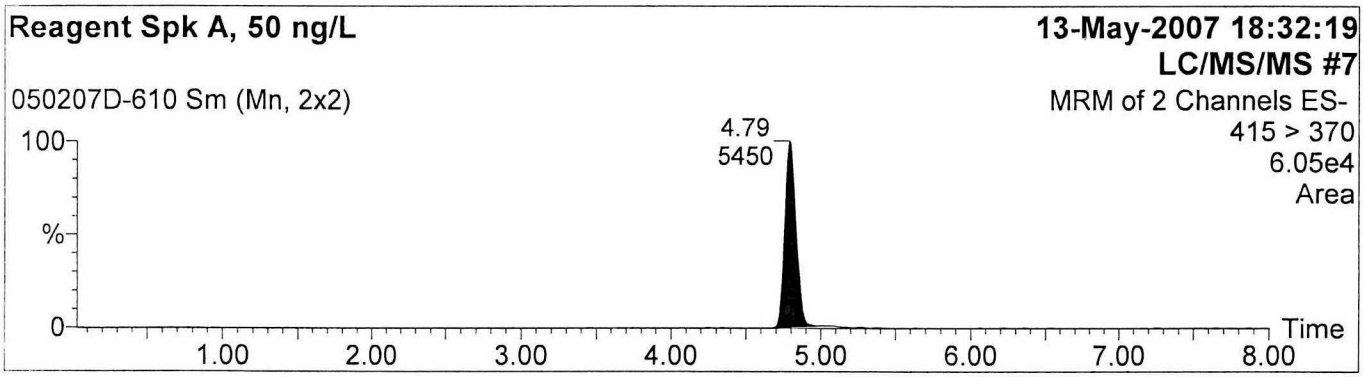
Printed: Tue May 15 11:13:46 2007

Name: 050207D-610
Text:

1: PFOA



2: 13C-PFOA



Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
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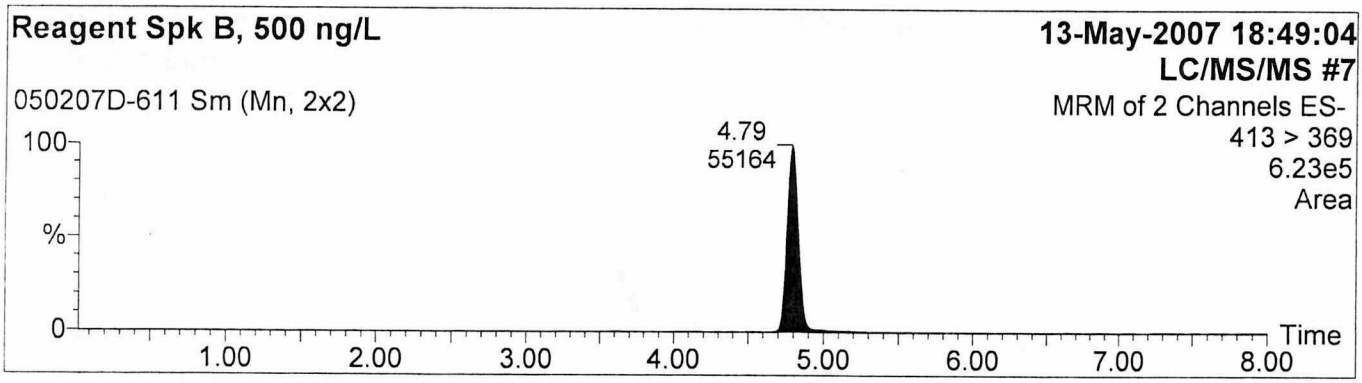
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

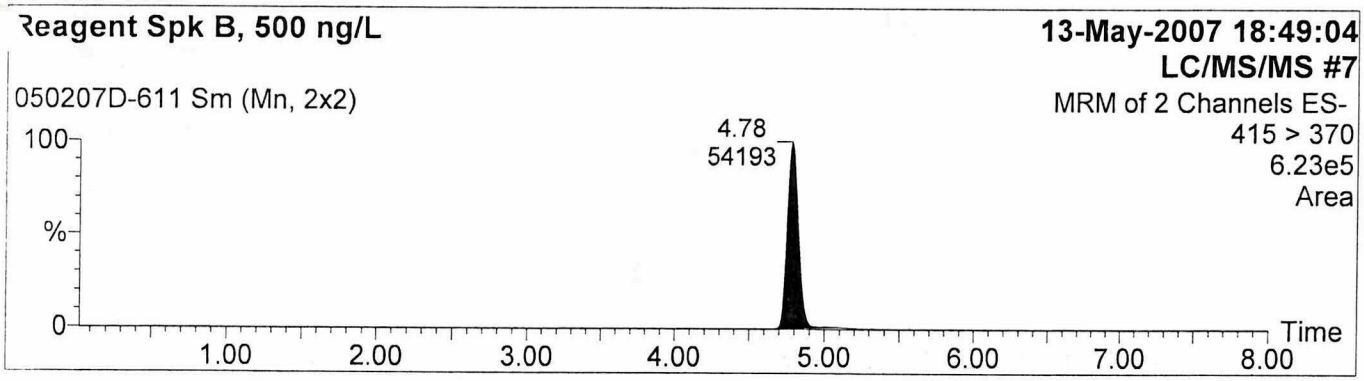
Printed: Tue May 15 11:13:46 2007

Name: 050207D-611
Text:

1: PFOA



2: 13C-PFOA



Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

Printed: Tue May 15 11:13:46 2007

Name: 050207D-612
Text:

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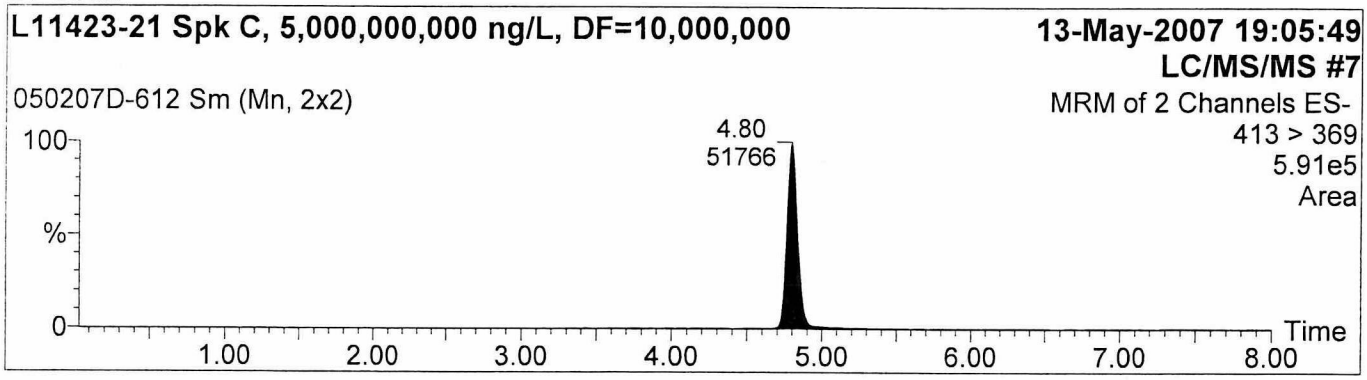
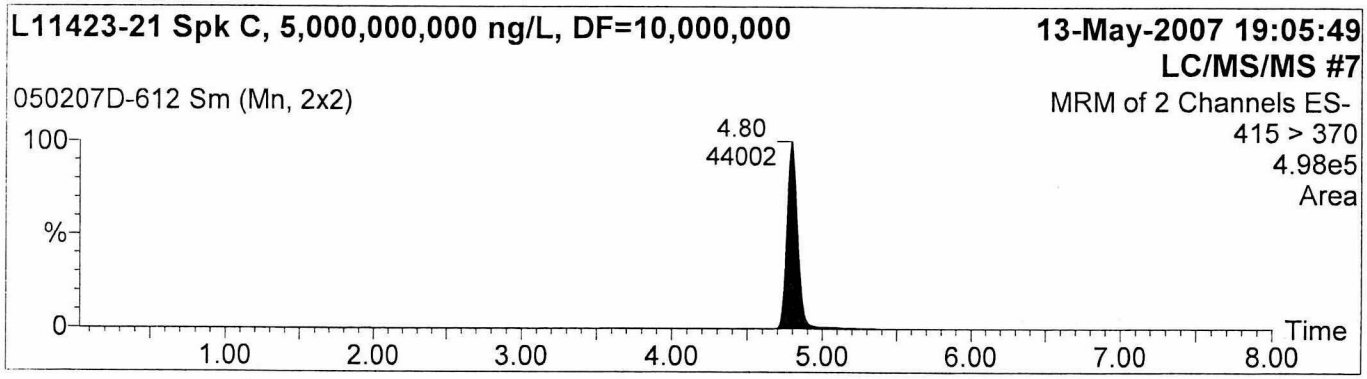


2: 13C-PFOA



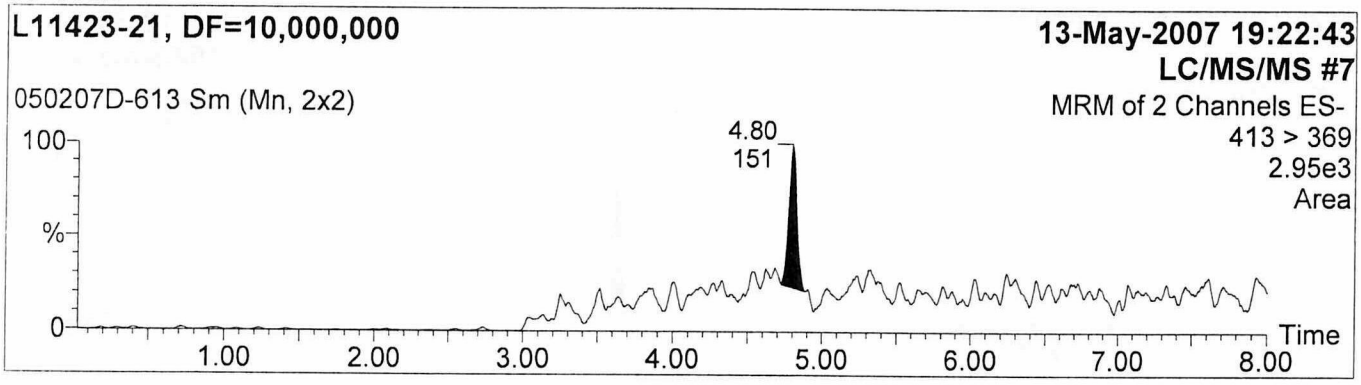
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

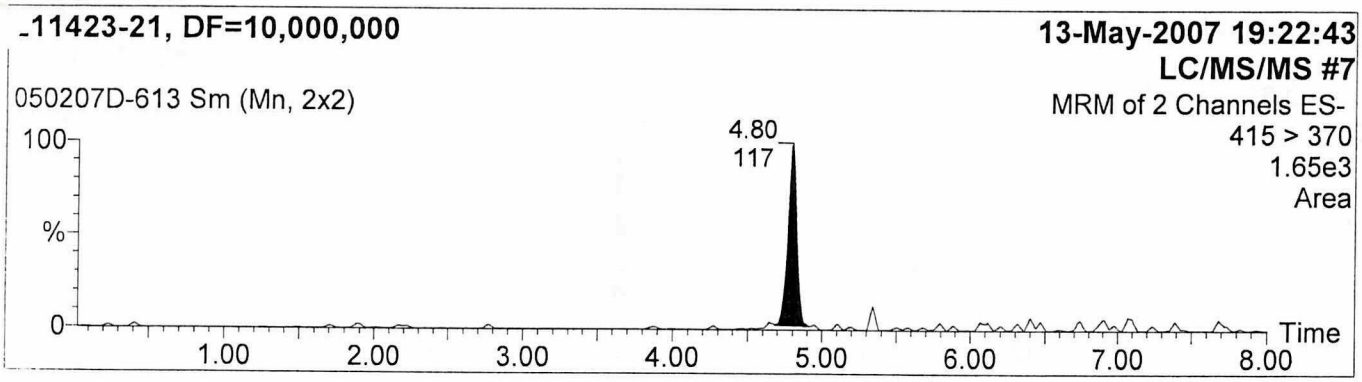
Printed: Tue May 15 11:13:46 2007

Name: 050207D-613
Text:

1: PFOA



2: 13C-PFOA



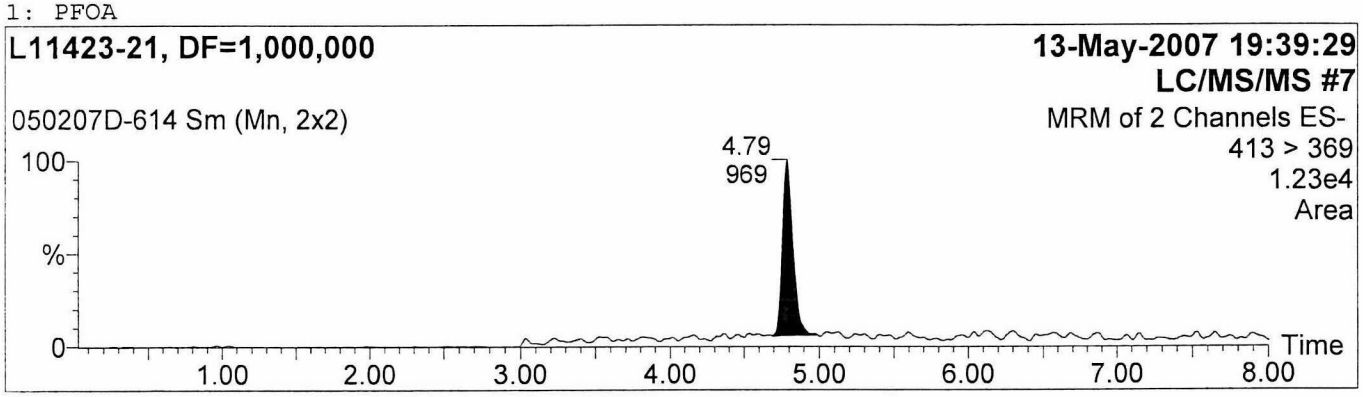
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

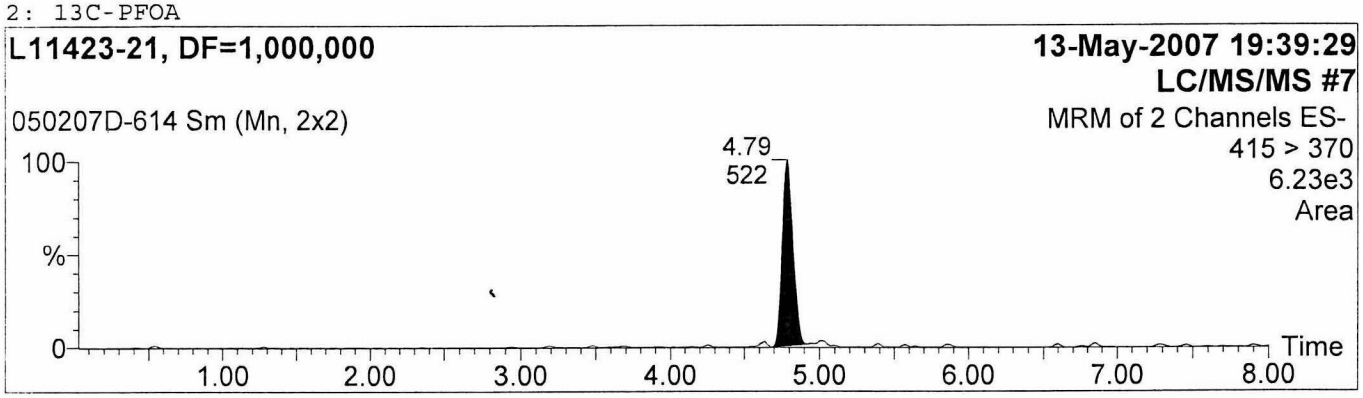
Printed: Tue May 15 11:13:46 2007

Name: 050207D-614
Text:

1: PFOA



2: 13C-PFOA



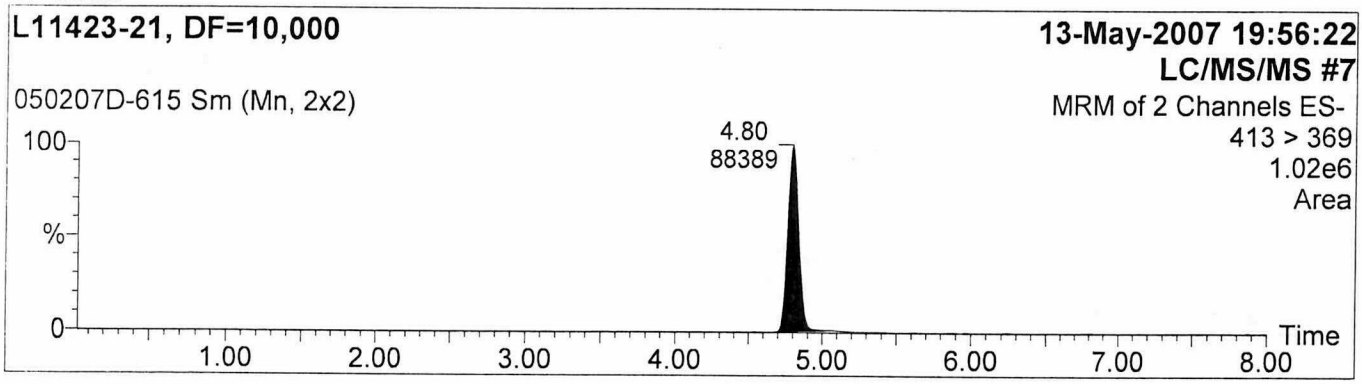
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

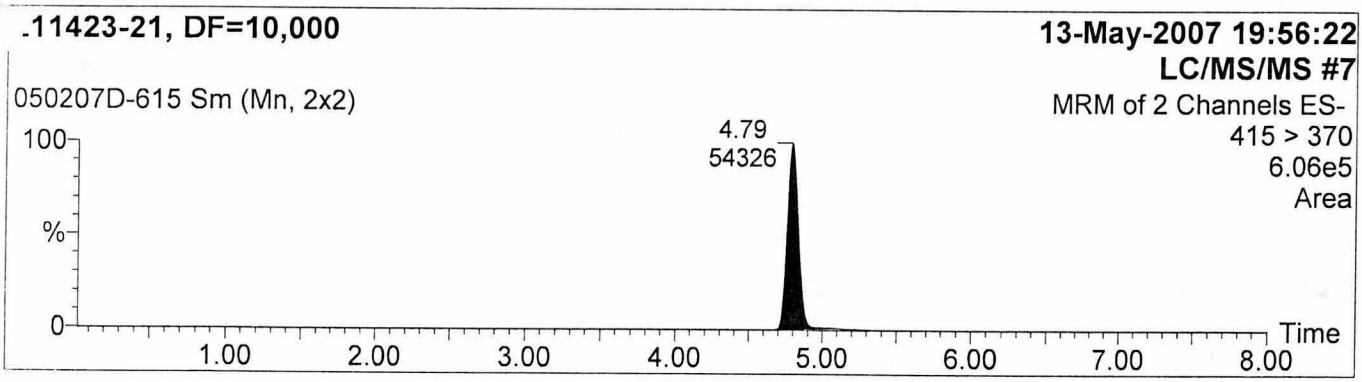
Printed: Tue May 15 11:13:46 2007

Name: 050207D-615
Text:

1: PFOA



2: 13C-PFOA



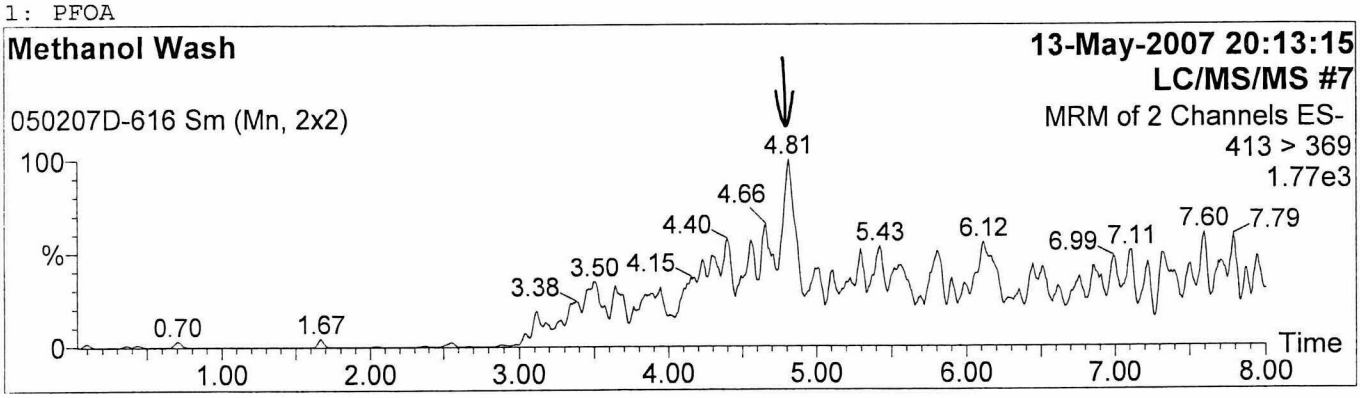
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

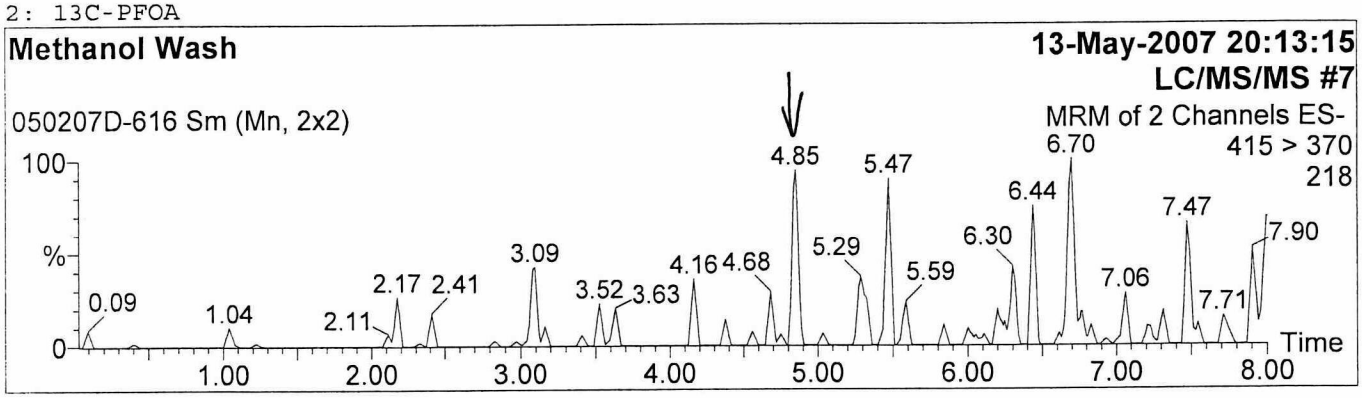
Printed: Tue May 15 11:13:46 2007

Name: 050207D-616
Text:

1: PFOA



2: 13C-PFOA



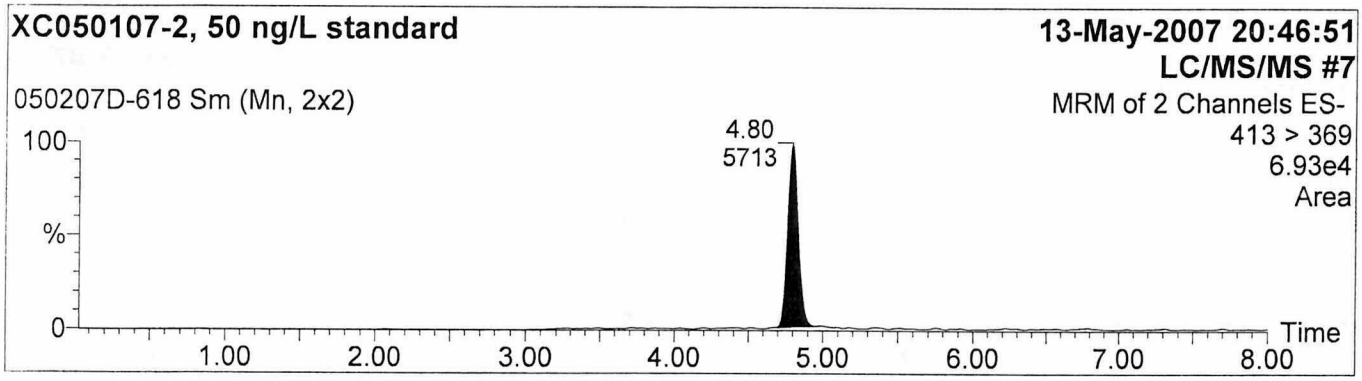
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

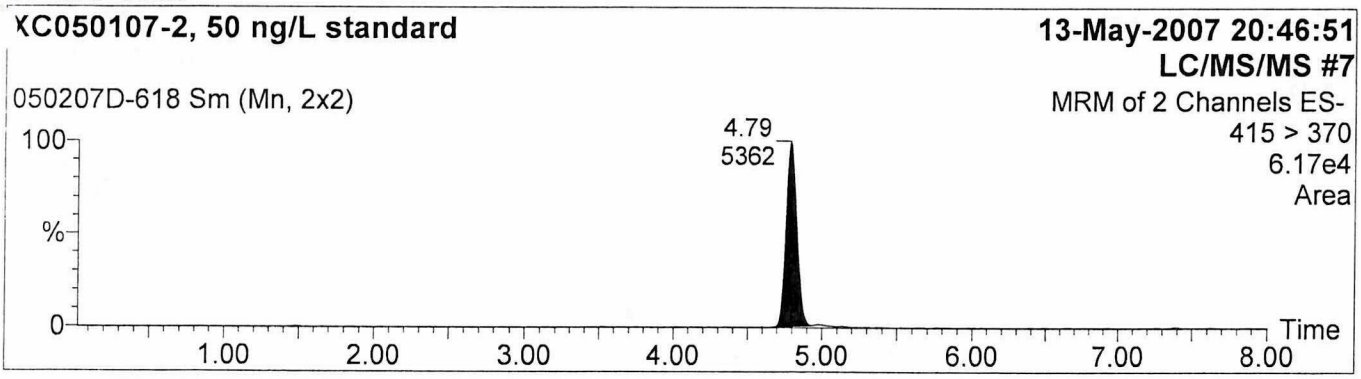
Printed: Tue May 15 11:13:46 2007

Name: 050207D-618
Text:

1: PFOA



2: 13C-PFOA



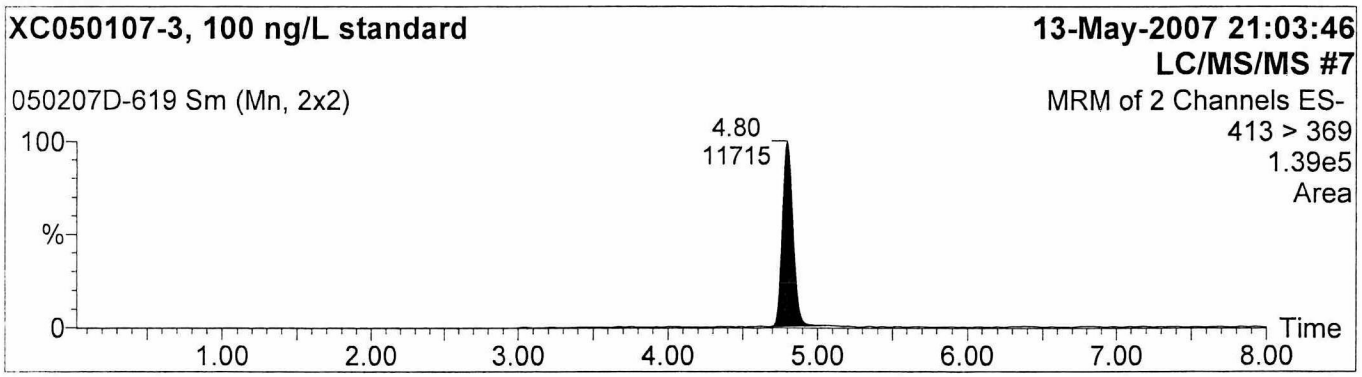
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

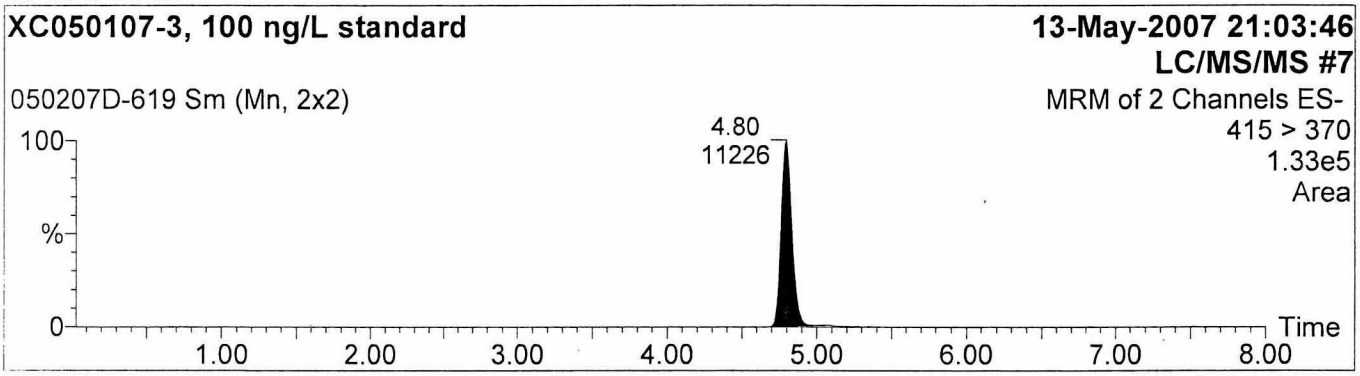
Printed: Tue May 15 11:13:46 2007

Name: 050207D-619
Text:

1: PFOA



2: 13C-PFOA



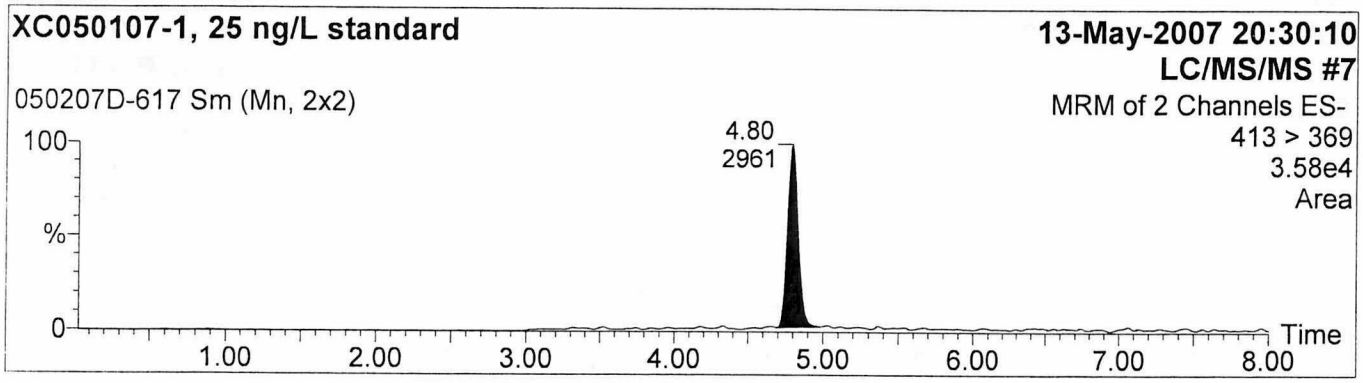
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

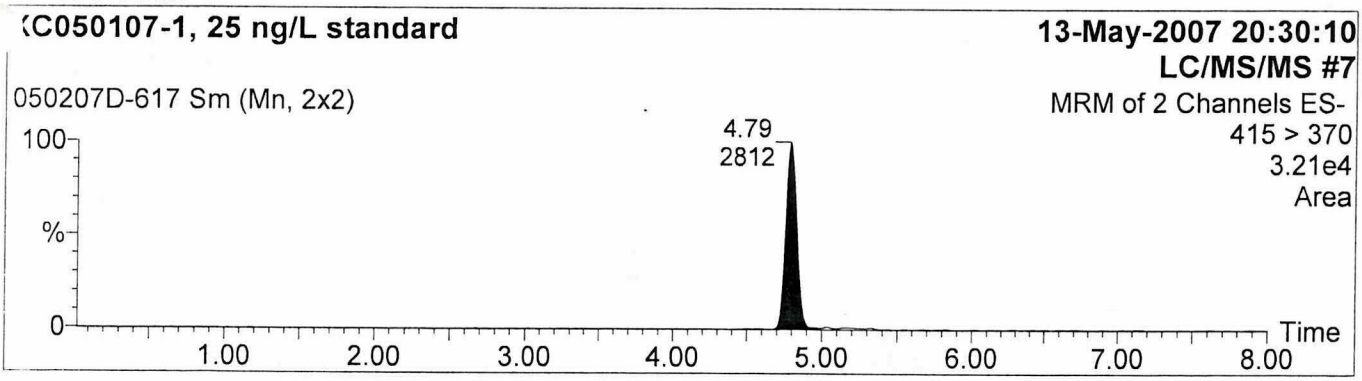
Printed: Tue May 15 11:13:46 2007

Name: 050207D-617
Text:

1: PFOA



2: 13C-PFOA



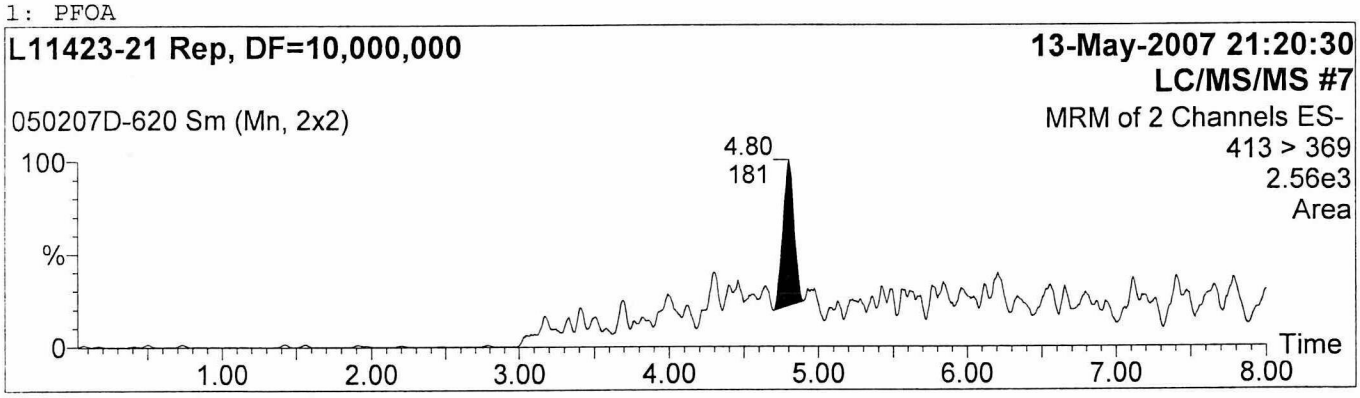
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

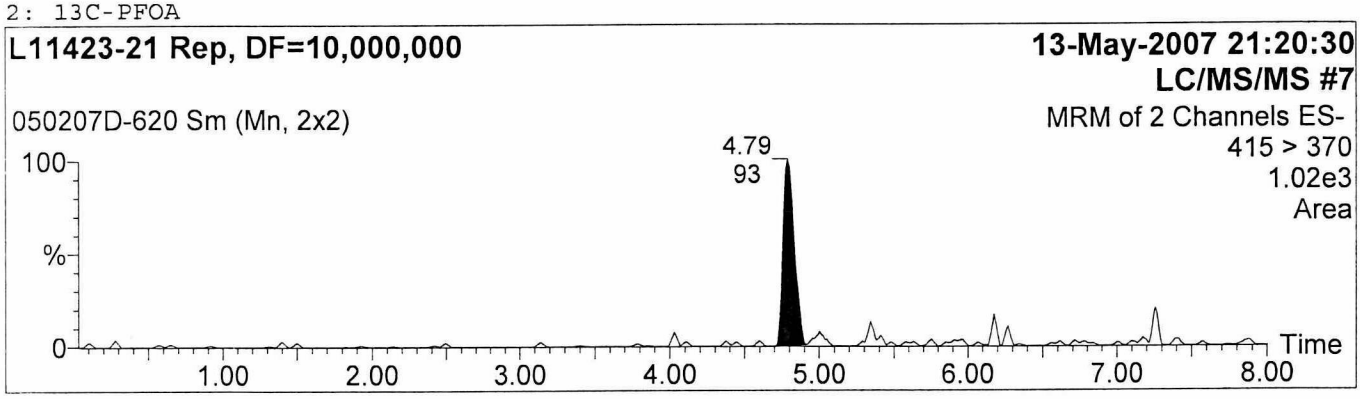
Printed: Tue May 15 11:13:46 2007

Name: 050207D-620
Text:

1: PFOA



2: 13C-PFOA



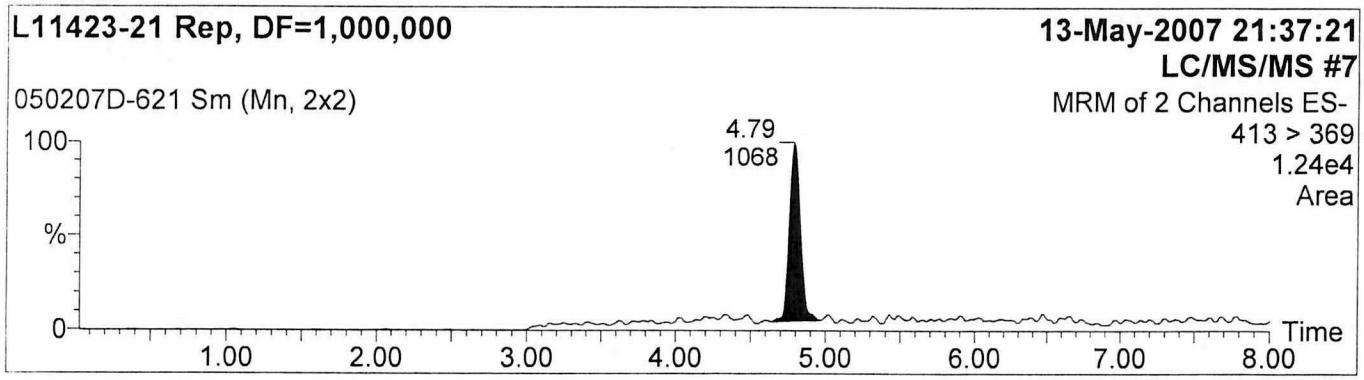
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

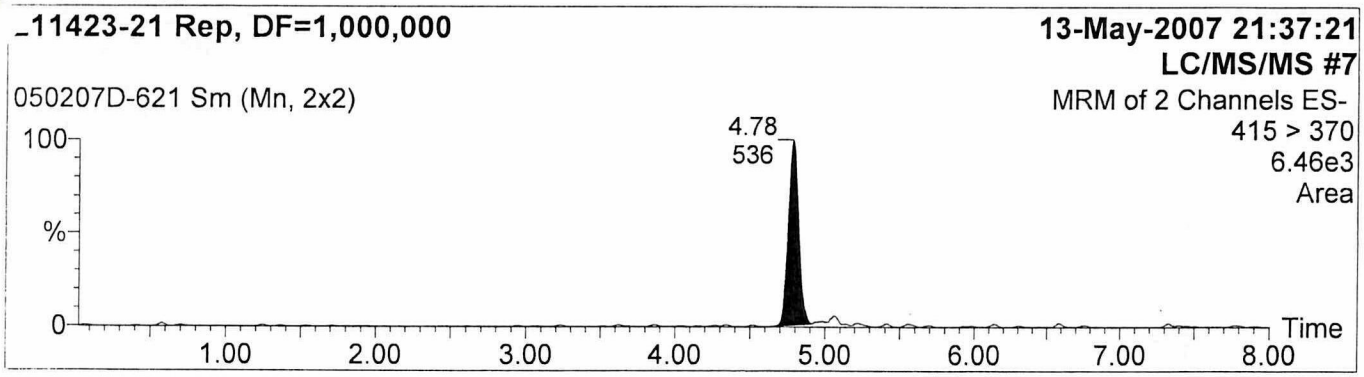
Printed: Tue May 15 11:13:46 2007

Name: 050207D-621
Text:

1: PFOA



2: 13C-PFOA



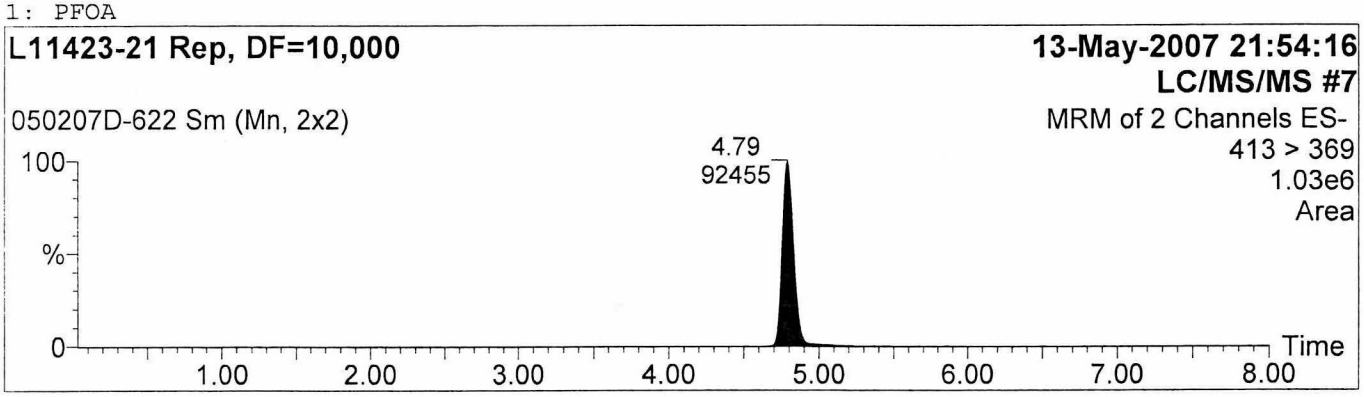
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

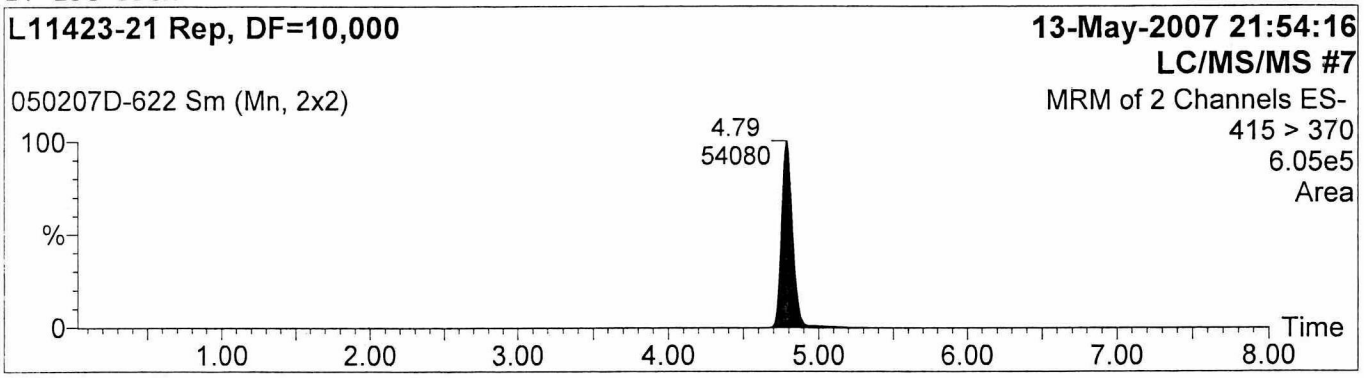
Printed: Tue May 15 11:13:46 2007

Name: 050207D-622
Text:

1: PFOA



2: 13C-PFOA



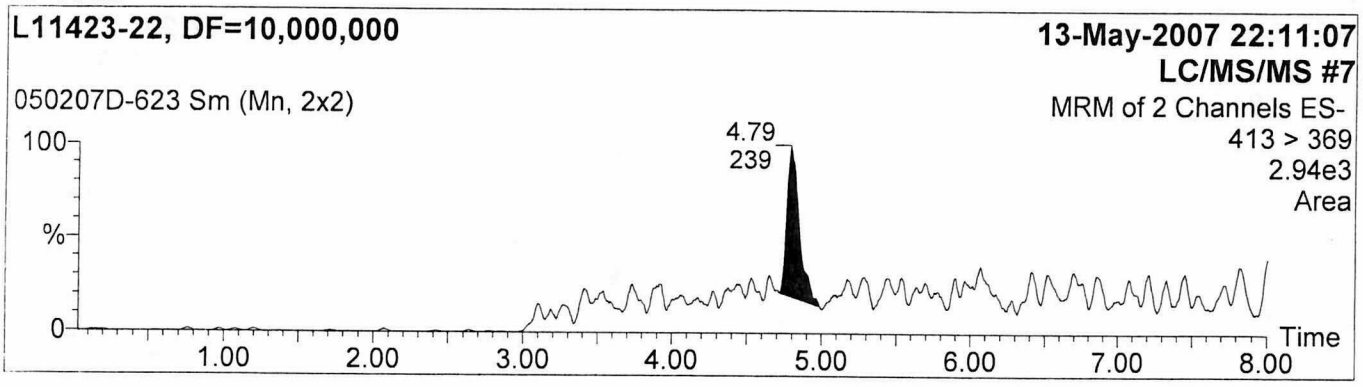
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

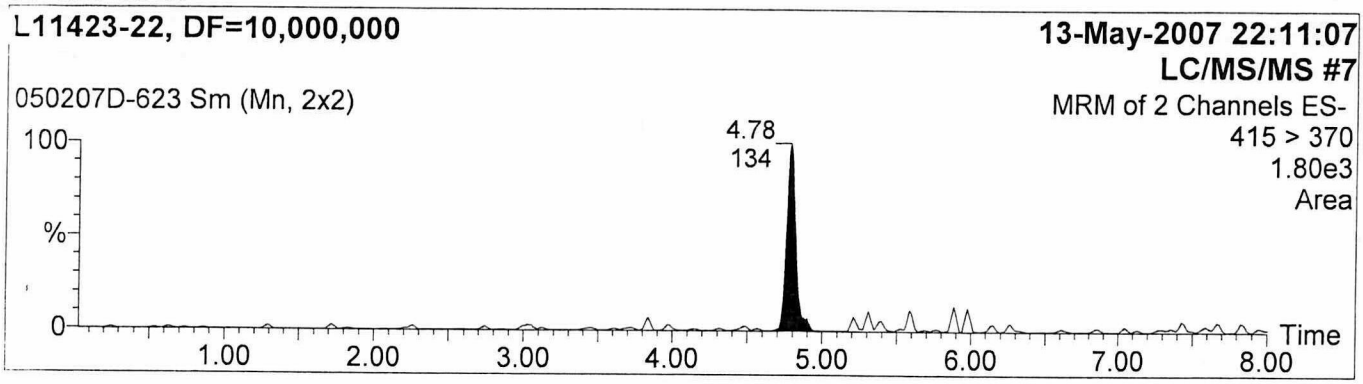
Printed: Tue May 15 11:13:46 2007

Name: 050207D-623
Text:

1: PFOA



2: 13C-PFOA

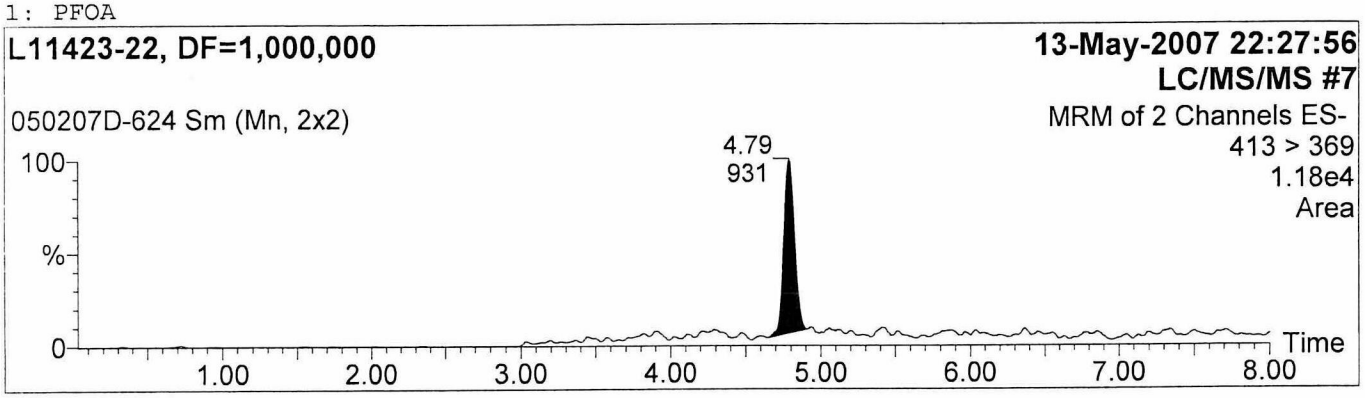


Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

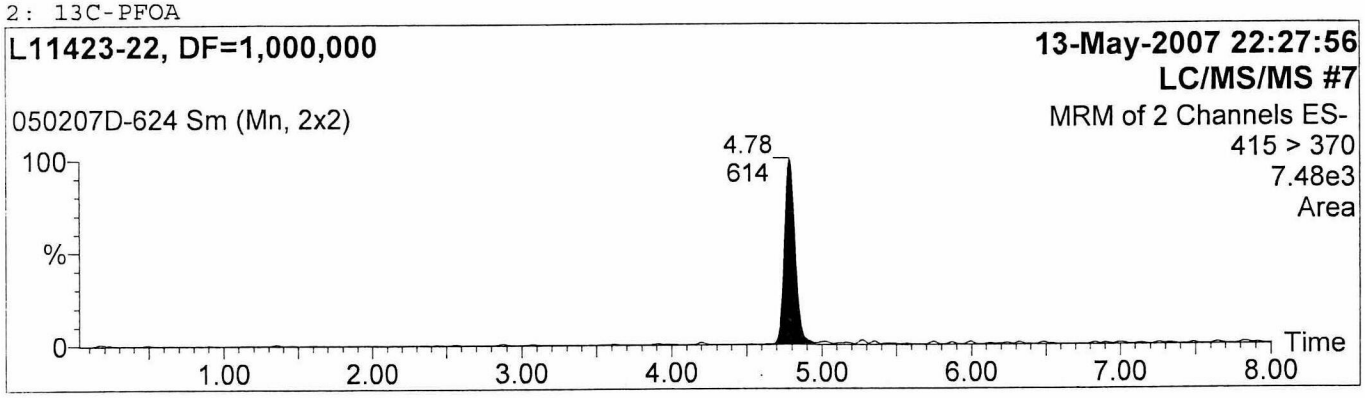
Printed: Tue May 15 11:13:46 2007

Name: 050207D-624
Text:

1: PFOA



2: 13C-PFOA



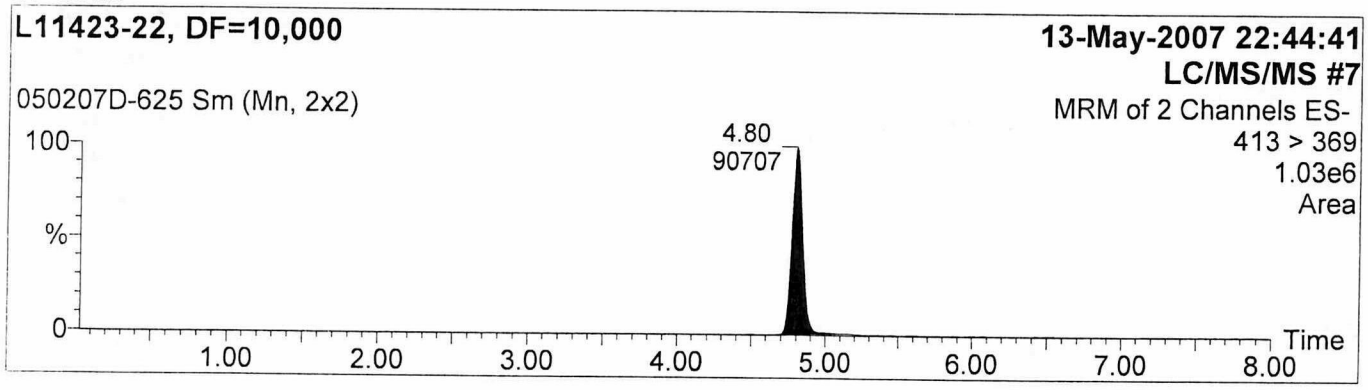
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

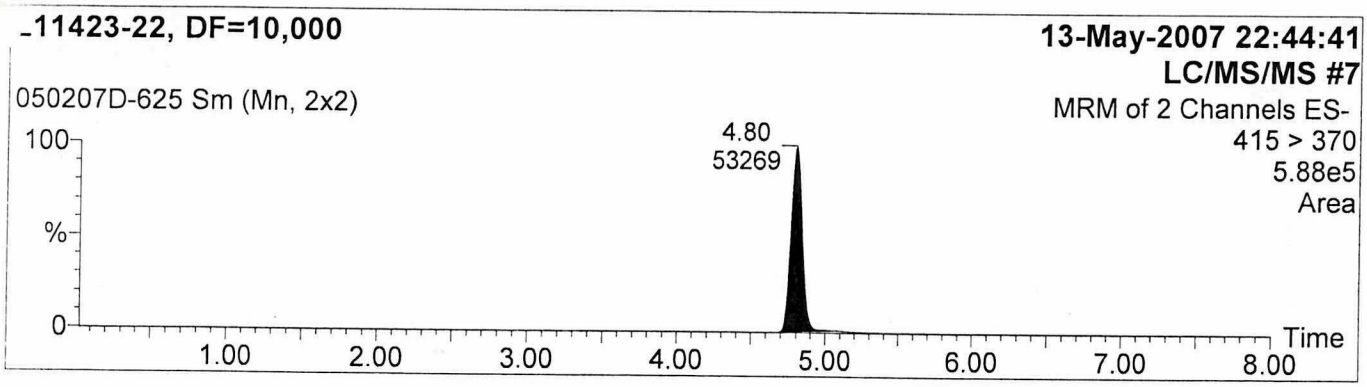
Printed: Tue May 15 11:13:46 2007

Name: 050207D-625
Text:

1: PFOA



2: 13C-PFOA



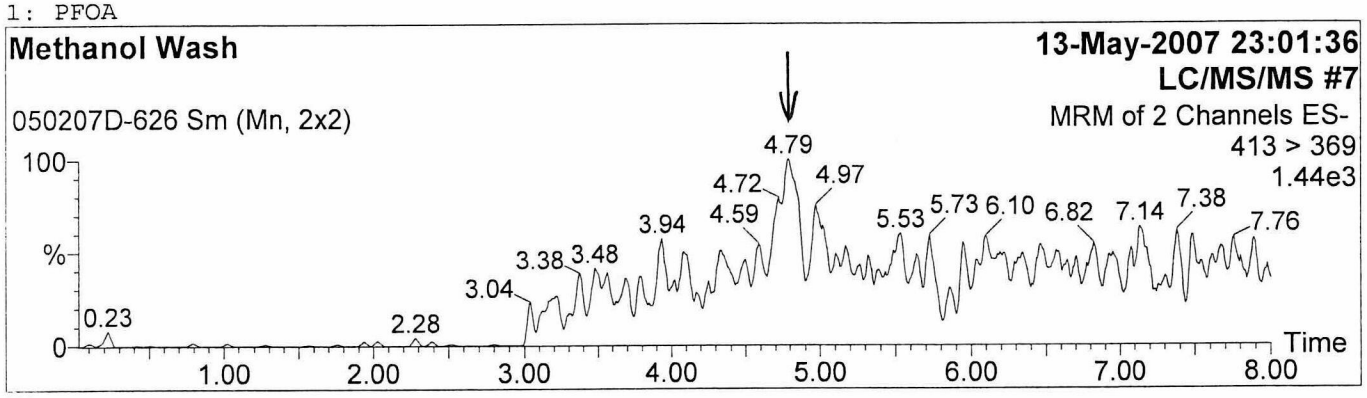
Quantify Sample Report
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

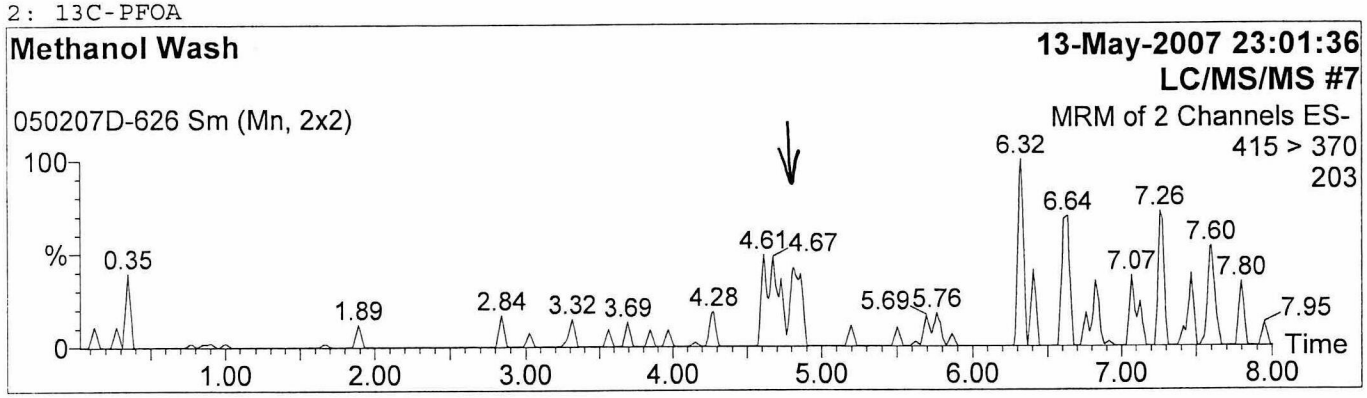
Printed: Tue May 15 11:13:46 2007

Name: 050207D-626
Text:

1: PFOA



2: 13C-PFOA



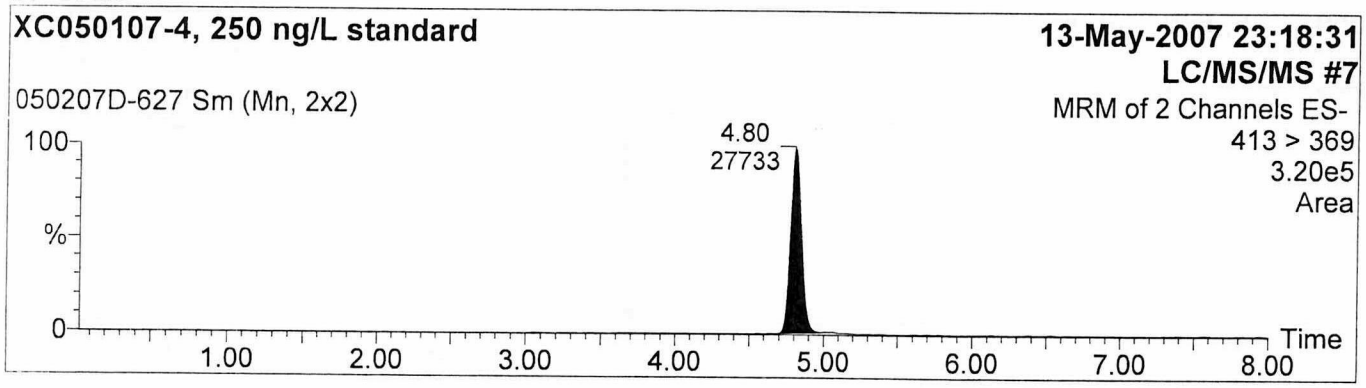
Quantify Sample Report
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

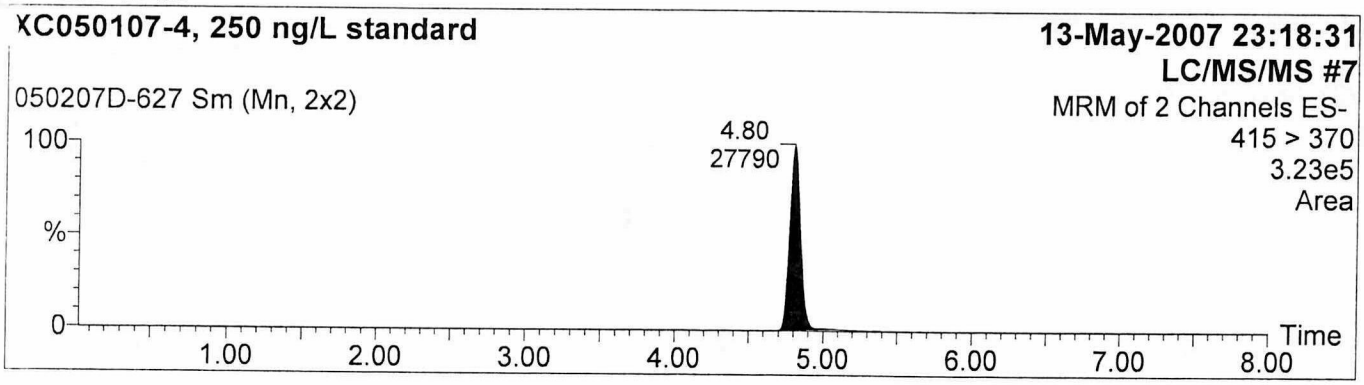
Printed: Tue May 15 11:13:46 2007

Name: 050207D-627
Text:

1: PFOA



2: 13C-PFOA



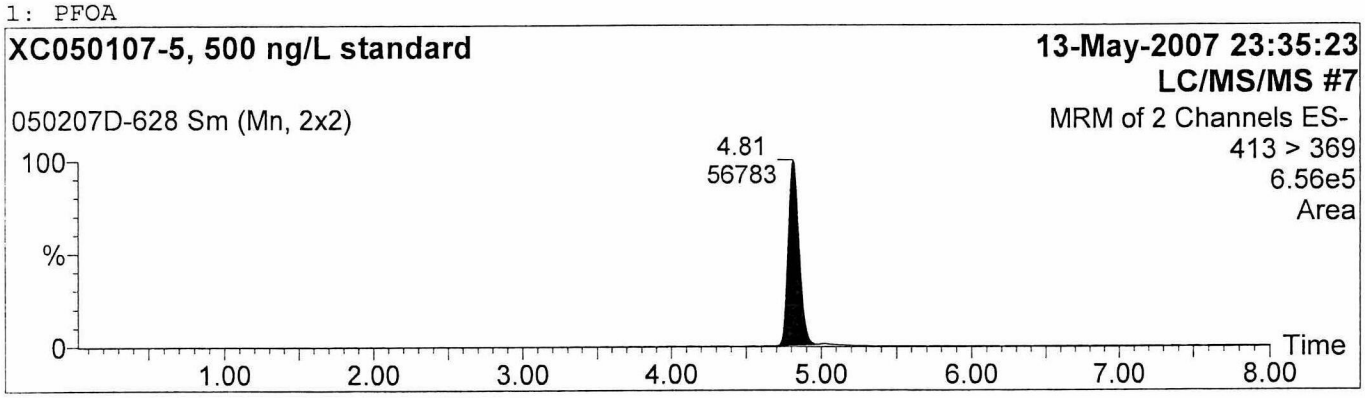
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

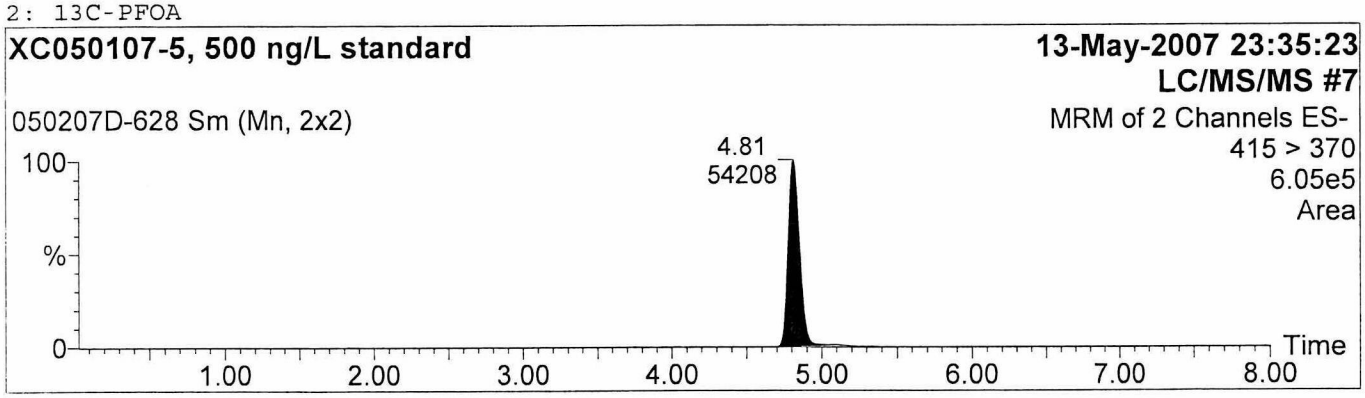
Printed: Tue May 15 11:13:46 2007

Name: 050207D-628
Text:

1: PFOA



2: 13C-PFOA



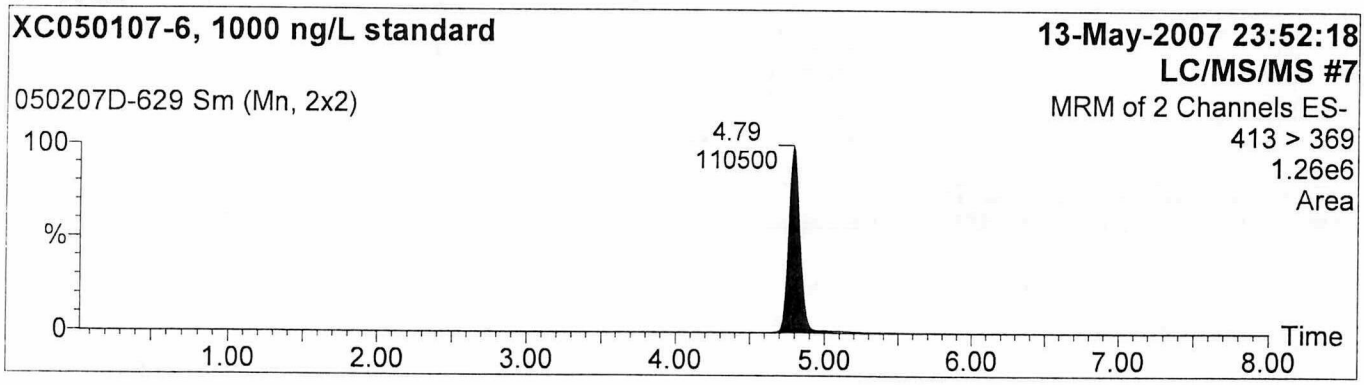
Study No: L11423, Set No: 050207D. Ext.Date: 05/02/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\050207D Dispersion Water
Last modified: Tue May 15 10:45:25 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

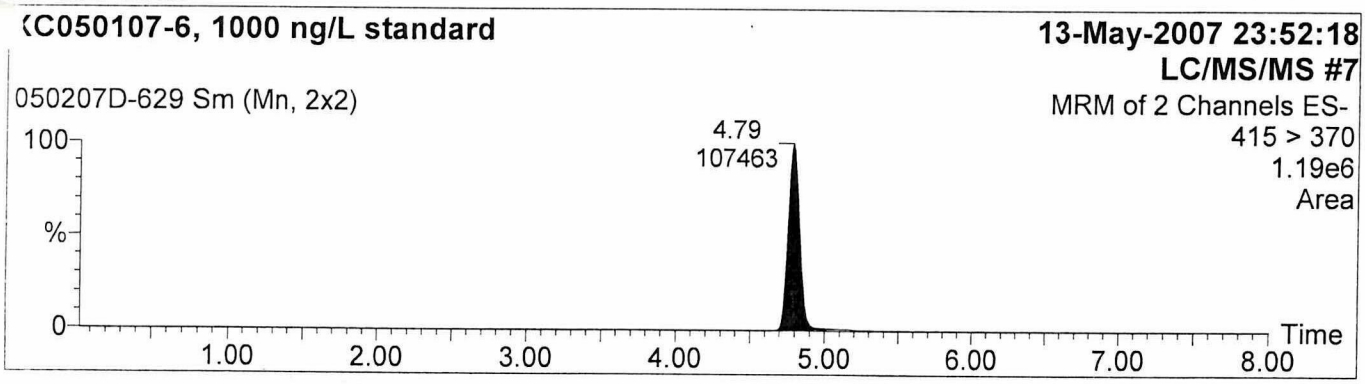
Printed: Tue May 15 11:13:46 2007

Name: 050207D-629
Text:

1: PFOA



2: 13C-PFOA



RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	50 ng/L	Set No:	051607A
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	**APFO**	Matrix:	Dispersions	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume Extracted:	0.4 mL	Extraction Date:	05/16/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/18/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC051707-0	-	CS	051607A-101	0	-	-	0	-	-	-	-
XC051707-1	-	CS	051607A-102	25	-	-	1733	-	-	-	-
XC051707-2	-	CS	051607A-103	50	-	-	3242	-	-	-	-
XC051707-3	-	CS	051607A-104	100	-	-	6436	-	-	-	-
XC051707-4	-	CS	051607A-105	250	-	-	14738	-	-	-	-
XC051707-5	-	CS	051607A-106	500	-	-	30310	-	-	-	-
XC051707-6	-	CS	051607A-107	1000	-	-	61355	-	-	-	-
Methanol Wash	-	C	051607A-108	-	-	-	0	-	-	-	-
Reagent Control	NA	C	051607A-109	-	-	1	0	ND	ND	-	-
Reagent Spk A	NA	LCS	051607A-110	-	-	1	3330	52.3	54.4	50	105
Reagent Spk B	NA	LCS	051607A-111	-	-	1	29697	502	523	500	100
L11423-21 Spk C	SG-L-001	LF	051607A-112	-	225	100000	11948	19900000	20700000	10000000	118*
Methanol Wash	-	C	051607A-113	-	-	-	0	-	-	-	-
XC051707-1	-	CS	051607A-114	25	-	-	1582	-	-	-	-
XC051707-2	-	CS	051607A-115	50	-	-	3067	-	-	-	-
XC051707-3	-	CS	051607A-116	100	-	-	6607	-	-	-	-
XC051707-4	-	CS	051607A-117	250	-	-	15520	-	-	-	-
XC051707-5	-	CS	051607A-118	500	-	-	27745	-	-	-	-
XC051707-6	-	CS	051607A-119	1000	-	.	56601	-	-	-	-

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

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

Standard Curve : Linear (1/x weighted)
Intercept = 264.049
Slope = 58.6412
Coef. Of Det. = 0.996710

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 25 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 50 ng/L (LOQ)

*Number used to calculate % Recovery is found in data set 050207D for the sample L11423-21.

Spreadsheet prepared by: MLC / 5/21/07

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	50 ng/L	Set No:	051607A
Exygen Study No:	L11423	Injection Volume:	15 µL	Analyst:	Mindy Cressley
Analyte:	^{13}C-PFOA	Matrix:	Dispersions	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	415 -> 370	Sample Volume Extracted:	0.4 mL	Extraction Date:	05/16/07
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	05/18/07

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Initial Sample Volume (mL)	Dilution Factor	Peak Area	13C-PFOA Found (ng/L)	Amount 13C-PFOA Added (ng/L)	Recovery (%)
XC051707-0	-	CS	051607A-101	0	-	-	0	-	-	-
XC051707-1	-	CS	051607A-102	25	-	-	1741	-	-	-
XC051707-2	-	CS	051607A-103	50	-	-	3200	-	-	-
XC051707-3	-	CS	051607A-104	100	-	-	6759	-	-	-
XC051707-4	-	CS	051607A-105	250	-	-	14382	-	-	-
XC051707-5	-	CS	051607A-106	500	-	-	32258	-	-	-
XC051707-6	-	CS	051607A-107	1000	-	-	63568	-	-	-
Methanol Wash	-	C	051607A-108	-	-	-	0	-	-	-
Reagent Control	NA	C	051607A-109	-	-	1	0	ND	-	-
Reagent Spk A	NA	LCS	051607A-110	-	-	1	3169	48.3	50	97
Reagent Spk B	NA	LCS	051607A-111	-	-	1	29749	486	500	97
L11423-21 Spk C	SG-L-001	LF	051607A-112	-	225	100000	6196	9820000	10000000	98
Methanol Wash	-	C	051607A-113	-	-	-	0	-	-	-
XC051707-1	-	CS	051607A-114	25	-	-	1754	-	-	-
XC051707-2	-	CS	051607A-115	50	-	-	3131	-	-	-
XC051707-3	-	CS	051607A-116	100	-	-	6528	-	-	-
XC051707-4	-	CS	051607A-117	250	-	-	15667	-	-	-
XC051707-5	-	CS	051607A-118	500	-	-	29711	-	-	-
XC051707-6	-	CS	051607A-119	1000	-	.	57890	-	-	-

13C-PFOA Found (ng/L) = (peak area - intercept) / slope x DF

Recovery (%) = $\frac{\text{13C-PFOA found (ng/L)}}{\text{amount 13C-PFOA added (ng/L)}} \times 100$

Standard Curve : Linear (1/x weighted)
Intercept = 235.555
Slope = 60.7178
Coef. Of Det. = 0.995941

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 25 ng/L (LOD)
NQ = Not quantifiable = Response between LOD and 50 ng/L (LOQ)

Spreadsheet prepared by: MLC / 5/21/07

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

Internal Chain of Custody/Fortification Sheet

Exygen Study Number: L11423 Matrix: Water
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 34
Time 9:15 AM Date 5/16/2007 Initials EDD

CLIENT SAMPLE ID	EXYGEN ID NUMBER	INITIAL VOL (mL)	EXT'N VOL (mL)	FORTIFICATION (ng)
na	Reagent Control	-	40.0	-
na	Reagent Spk A	-	40.0	2.0
na	Reagent Spk B	-	40.0	20.0
SG-L-001	L11423-21 Spk C*	225 mL	40.0	40.0
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-
-	-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
Reagent Spk A	SS20798 (10 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/16/07
Reagent Spk A	F012104-4 (10 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/16/07
Reagent Spk B	SS20797 (100 ng/mL PFOA)	200 µL (200 µL micropipet)	MLC / 5/16/07
Reagent Spk B	F012104-3 (100 ng/mL ^{13}C-PFOA)	200 µL (200 µL micropipet)	MLC / 5/16/07
L11423-21 Spk C*	SS20797 (100 ng/mL PFOA)	400 µL (200 µL micropipet)	MLC / 5/16/07
L11423-21 Spk C*	F012104-3 (100 ng/mL ^{13}C-PFOA)	400 µL (200 µL micropipet)	MLC / 5/16/07
-	-	-	-
-	-	-	-
-	-	-	-

All samples were measured:
Time 11:15 AM Date 5/16/2007 Initials EDD

After measuring samples were returned to refrigerator No. 34
Time 12:18 PM Date 5/16/2007 Initials EDD

Comments: *Sample is pre-diluted 10000 times by taking 0.4 mL of sample and bringing the volume up to 40 mL with HPLC water, then taking 0.4 mL of that solution and bringing the volume up to 40 mL.
Spiking is done on a 10000x diluted sample for Spk C.

Analysis Summary:
Data Set: 051607A Initials/Date: MLC / 5/17/07
Data Set: - Initials/Date: - / -
Data Set: - Initials/Date: - / -

Set extraction/analysis data verified by: CEE Date: 05/24/07

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

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

EXYGEN STUDY NUMBER: L11423 METHOD: ExM-079-007A[$$] PROTOCOL NUMBER: NA

MATRIX: Water ANALYTES: PFOA and ^{13}C-PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot # or Exygen ID
na	Reagent Control	↓	↓	-	↓	-	-	Methanol	47089
na	Reagent Spk A			-		-	-	C18 SPE	065437115A
na	Reagent Spk B			-		-	-	HPLC Water	46355
SG-L-001	L11423-21 Spk C*	↓	↓	0.1/1.0	↓	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	*Initials/Date*	EDD 5/16/07
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	**HPLC**	
-	-	-	-	-	-	-	-	Methanol	46356
-	-	-	-	-	-	-	-	Ammonium Acetate	C44H04
-	-	-	-	-	-	-	-	HPLC Water	46271
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-
**Initials/Date*	-	EDD 5/16/07	EDD 5/16/07	MLC 5/16/07	MLC 5/17/07	-	-	*Initials/Date*	MLC 5/17/07

STEP 1: SPE column clean-up
STEP 2: Final volume to 5 mL collected in 15 mL polypropolene tubes
STEP 3: LC/MS/MS analysis
STEP 4: LC/MS/MS reanalysis.

*Sample was diluted 10000 times prior to extraction.
**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 by LC/MS/MS for SPI Processors Mass Balance Study.

COMMENTS:

Final extracts stored in refrigerator 32 Initials: EDD Date: 5/16/2007

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

Exygen STUDY NO. L11423

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Protocol No.: None
Method No.: $$
Exygen Study No.: NA
Analytes: PFOA and ^{13}C-PFOA
Matrix: Water
Sample Vol: 40 mL

Sponsor Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # ^^	Reagents/ Materials	Lot #
NA	Water	-	-	-	-	-	XC051707-10	Methanol	47089
NA	Water	SS20798* & F012104-4**	10	100	200	25	XC051707-11	C18 SPE	005437115A
NA	Water	SS20798* & F012104-4**	10	200	200	50	XC051707-12	Water	47067
NA	Water	SS20798* & F012104-4**	10	400	200	100	XC051707-13	-	-
NA	Water	SS20797* & F012104-3**	100	100	200	250	XC051707-14	-	-
NA	Water	SS20797* & F012104-3**	100	200	200	500	XC051707-15	-	-
NA	Water	SS20797* & F012104-3**	100	400	200	1000	XC051707-16	-	-
-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	Initials/Date:	EDD 5/17/07

Vertical arrows in a column indicate identical values.

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

Samples removed from (refrigerator) / freezer # 34 Time: 8:30AM — Initials/Date: EDD 1 5/17/07

40 mL of each sample measured using a 50 mL graduated cylinder. — Initials/Date: EDD 1 5/17/07

After measuring, samples returned to (refrigerator) / freezer # 34 Time: 10:30AM — Initials/Date: EDD 1 5/17/07

Samples fortified: — Initials/Date: MLC 1 5/17/07

SPE clean-up: — Initials/Date: EDD 1 5/17/07

Final volume adjusted to 5 mL: — Initials/Date: EDD 1 5/17/07

Extracts placed in refrigerator # 32 — Initials/Date: EDD 1 5/17/07

Comments:

STANDARD EXPIRATION DATE: 5/31/2007

$$ Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study.

*Solution contains PFOA.

**Solution contains ^{13}C-PFOA.

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY MLC DATE 5/21/07

Masslynx - Sample List

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\051607A Dispersion Water.SPL
Printed: Thu May 17 15:01:21 2007 MLC 5/17/07

Exygen STUDY NO. L11423

Page Position: (1, 1)

L (RE) MLC 5/17/07

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ng/~~mL~~)	Conc B	Conc C	Test ID
1	94	051607A-101	---	---	① XC050~~4~~07-0, 0 ng/L standard	---	Standard	0	0	---	0
2	95	051607A-102	---	---	XC050~~4~~07-1, 25 ng/L standard	---	Standard	25	25	---	0
3	96	051607A-103	---	---	XC050~~4~~07-2, 50 ng/L standard	---	Standard	50	50	---	0
4	97	051607A-104	---	---	XC050~~4~~07-3, 100 ng/L standard	---	Standard	100	100	---	0
5	98	051607A-105	---	---	XC050~~4~~07-4, 250 ng/L standard	---	Standard	250	250	---	0
6	99	051607A-106	---	---	XC050~~4~~07-5, 500 ng/L standard	---	Standard	500	500	---	0
7	100	051607A-107	---	---	XC050~~4~~07-6, 1000 ng/L standard	---	Standard	1000	1000	---	0
8	92	051607A-108	---	---	Methanol Wash	---	Blank	---	---	---	0
9	81	051607A-109	---	---	Reagent Control	---	Blank	---	---	---	0
10	82	051607A-110	---	---	Reagent Spk A, 50 ng/L	---	QC	50	50	---	0
11	83	051607A-111	---	---	Reagent Spk B, 500 ng/L	---	QC	500	500	---	0
12	84	051607A-112	---	---	L11423-21 Spk C, 10,000,000 ng/L, DF=100,000	---	QC	10000000	10000000	---	0
13	92	051607A-113	---	---	① Methanol Wash	---	Blank	---	---	---	0
14	95	051607A-114	---	---	XC050~~4~~07-1, 25 ng/L standard	---	Standard	25	25	---	0
15	96	051607A-115	---	---	XC050~~4~~07-2, 50 ng/L standard	---	Standard	50	50	---	0
16	97	051607A-116	---	---	XC050~~4~~07-3, 100 ng/L standard	---	Standard	100	100	---	0
17	98	051607A-117	---	---	XC050~~4~~07-4, 250 ng/L standard	---	Standard	250	250	---	0
18	99	051607A-118	---	---	XC050~~4~~07-5, 500 ng/L standard	---	Standard	500	500	---	0
19	100	051607A-119	---	---	XC050~~4~~07-6, 1000 ng/L standard	---	Standard	1000	1000	---	0

① (RE) XC051707 MLC 5/17/07

Sample List: C:\MASSLYNX\P593.PRO\SampleDB\051607A Dispersion Water.SPL

Exygen STUDY NO. L11423

Page Position: (2, 1)

Printed: Thu May 17 15:01:21 2007 MLC 5/17/07

	DF	MS Method	HPLC Method	MS Tune File	Inj Volume (µL)
1	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
2	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
3	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
4	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
5	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
6	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
7	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
8	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
9	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
10	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
11	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
12	100000	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
13	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
14	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
15	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
16	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
17	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
18	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15
19	1	PFOA AND 13C PFOA	mass balance water	PFOA Tune	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA

Exygen Study No: L11423

Instrument:	Micromass Quattro Ultima (LC/MS/MS Unit #7)
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:	Agilent Technologies (HP) Series 1100 HP Quat Pump — HP Vacuum Degasser HP Autosampler — HP Column Oven
HPLC Column:	Genesis C-8, 5 cm x 2.1 mm i.d. x 4 µ (Exygen ID: MA0034061) (**JONES** CHROMATOGRAPHY: Part No. FK5962E)
Mobile Phase (A) :	2 mM Ammonium Acetate in Water
Mobile Phase (B) :	Methanol
Analyst:	Mindy Cressley MLC 5/17/07 Exygen Research 3058 Research Drive, State College, PA 16801 Phone: (814) 272-1039 FAX: (814) 231-1580

NOTE: The next 3 pages are computer generated printouts from the masslynx software program. The pages contain the instrument settings used for the analysis of this data set.

All Handwritten Peak ID's by: MLC 5/21/07

Scanning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA AND 13C PFOA
Last Modified: Thu Jan 29 16:40:36 2004

Printed: Thu May 17 15:01:51 2007 MLC 5/17/07

Solvent Delay (mins) : 0.00

Function : 1 MRM of 2 Mass Pairs (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
2	415.00	370.00	0.20	10	10

Method File: c:\masslynx\p593.pro\acqudb\mass balance water
Last Modified: Thursday, May 17, 2007 15:02:03

Printed: Thursday, May 17, 2007 15:02:08 MLC 5/17/07

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 5 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	Off	Off	Off	
3.00	Off	Off	On	Off	Off	
8.00	Off	Off	Off	Off	Off	

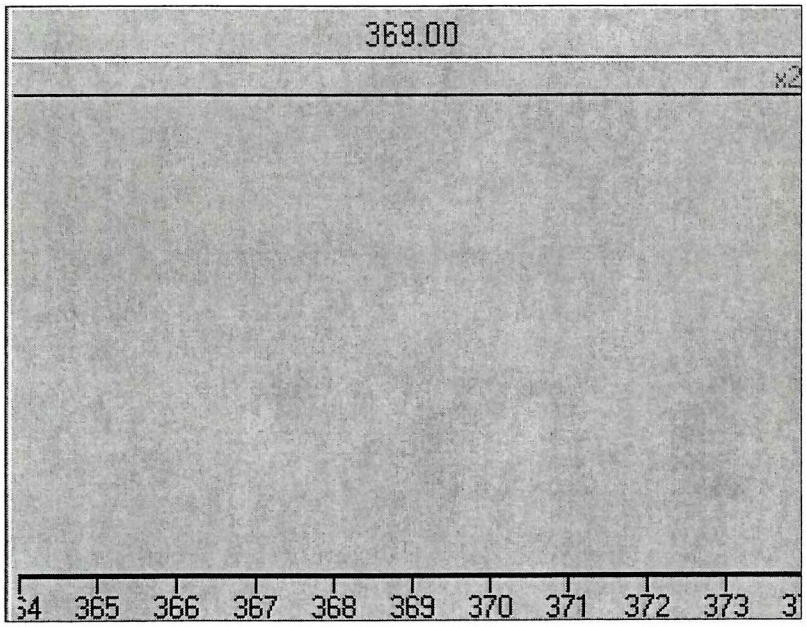
HP1100 Autosampler Initial Conditions

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	100

Tuning Method Report

Method: C:\MASSLYNX\P593.PRO\ACQUDB\PFOA TUNE

Printed: Thu May 17 15:02:24 2007 MLC 5/17/07



Dau 413.00

SOURCE (ESP-)	Set	Rdbk	Analyser	Set	Rdbk
Capillary	3.00	-2.94	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	102	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	97.0
Gas Cell	2.2e-3	Desolvation	683.9

Initial Calibration Curve for PFOA

Exygen Study No.: L11423
Set No: 051607A
Extraction Date: 05/16/07
Analyzed on: 05/18/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC051707-1	25	1733
XC051707-2	50	3242
XC051707-3	100	6436
XC051707-4	250	14738
XC051707-5	500	30310
XC051707-6	1000	61355

Coefficient of Determination: 0.999777545

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999888767
R Square	0.999777545
Adjusted R Square	0.999721932
Standard Error	383.310347
Observations	6

MLC
5/21/07

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	2641332578	2641332578	17977.19804	1.85586E-08
Residual	4	587707.2885	146926.8221		
Total	5	2641920285			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	39.64057673	214.1224687	0.185130393	0.862134777	-554.8587035	634.139857	-554.8587035	634.139857
X Variable 1	61.07852288	0.455541056	134.0790738	1.85586E-08	59.81373814	62.34330761	59.81373814	62.34330761

Initial Calibration Curve for ^{13}C-PFOA

Exygen Study No.: L11423
Set No: 051607A
Extraction Date: 05/16/07
Analyzed on: 05/18/07

The first curve of the data set is used to determine performance. A linear regression (with no weighting) is performed using the first six 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.

Exygen ID	Standard Concentration (ng/L)	Peak Area
XC051707-1	25	1741
XC051707-2	50	3200
XC051707-3	100	6759
XC051707-4	250	14382
XC051707-5	500	32258
XC051707-6	1000	63568

Coefficient of Determination: 0.999076465

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999538126
R Square	0.999076465
Adjusted R Square	0.998845582
Standard Error	814.0728374
Observations	6

MLC 5/21/07

ANOVA

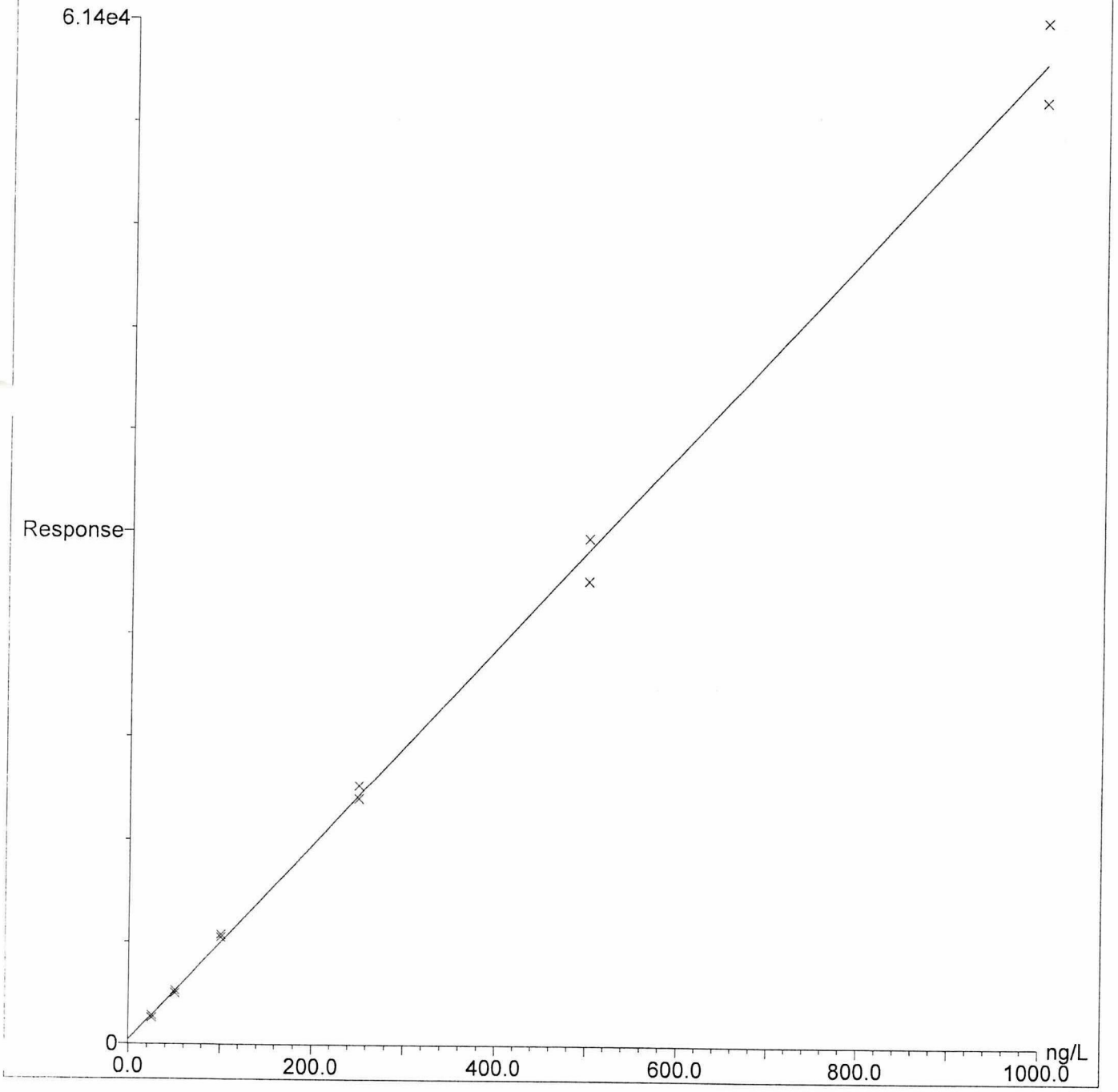
	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	2867688672	2867688672	4327.185094	3.19942E-07
Residual	4	2650858.338	662714.5845		
Total	5	2870339530			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-100.4356334	454.752362	-0.22085786	0.836018619	-1363.030603	1162.159336	-1363.030603	1162.159336
X Variable 1	63.6418773	0.967476101	65.78134305	3.19942E-07	60.95573301	66.32802158	60.95573301	66.32802158

Quantify Calibration Report
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\051607A Dispersion
Last modified: Mon May 21 09:02:14 2007
Printed: Mon May 21 09:05:18 2007 MLC 5/21/07

Compound 1 name: PFOA
Coefficient of Determination: 0.996710
Calibration curve: 58.6412 * x + 264.049
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

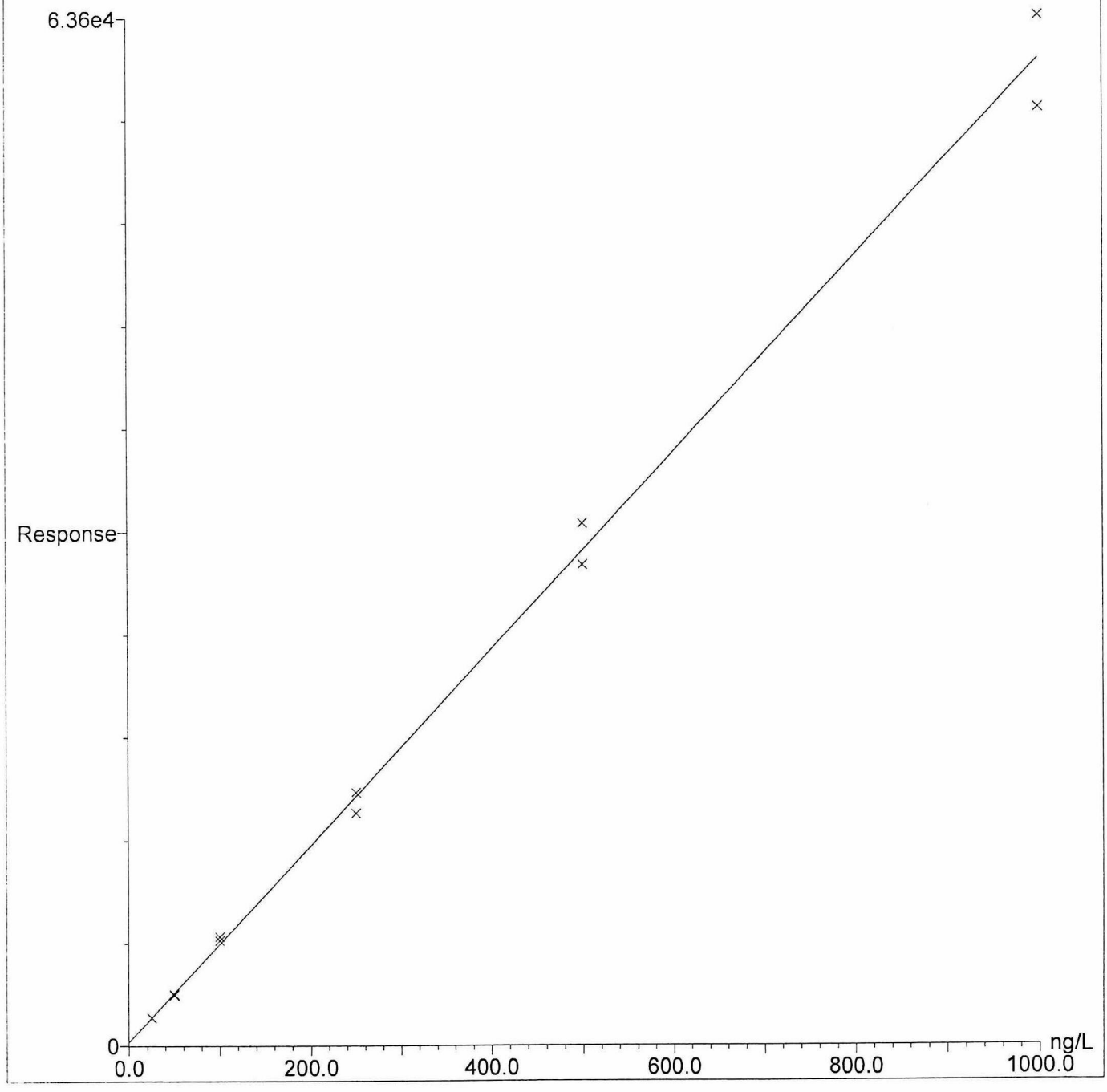


Quantify Calibration Report
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Calibration: P:\Data\LCMSMS7\Masslynx\P593.PRO\CurveDB\051607A Dispersion
Last modified: Mon May 21 09:02:14 2007
Printed: Mon May 21 09:05:18 2007

MLC 5/21/07

Compound 2 name: 13C-PFOA
Coefficient of Determination: 0.995941
Calibration curve: 60.7178 * x + 235.555
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

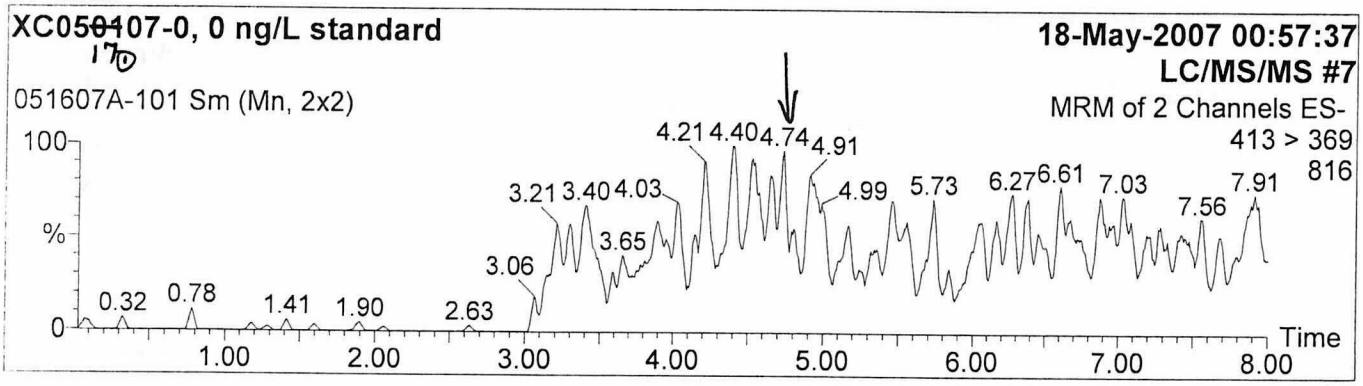
Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

Initials MLC
Date 5/21/07
Run# 051607A-101 To 051607A-119

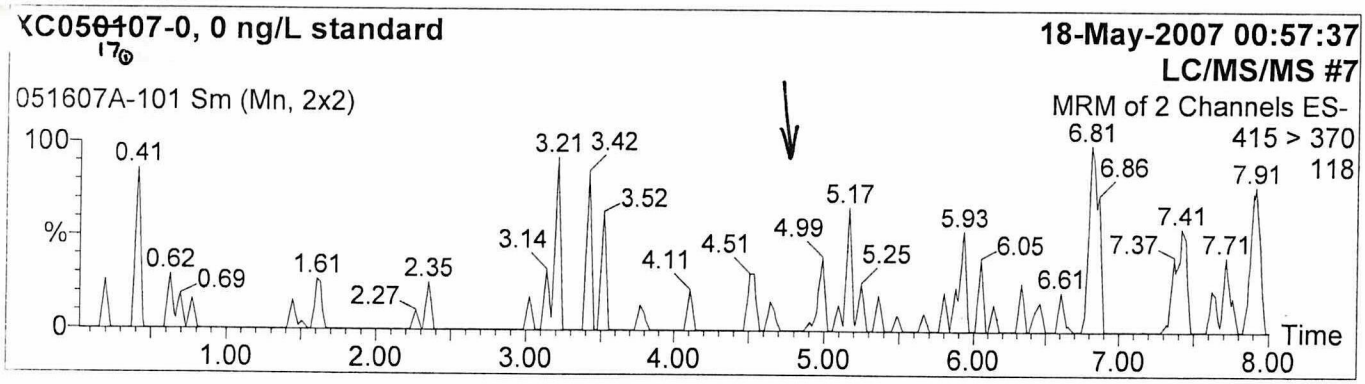
Printed: Mon May 21 09:05:19 2007

Name: 051607A-101
Text:

1: PFOA



2: 13C-PFOA



(RE) MLC 5/21/07

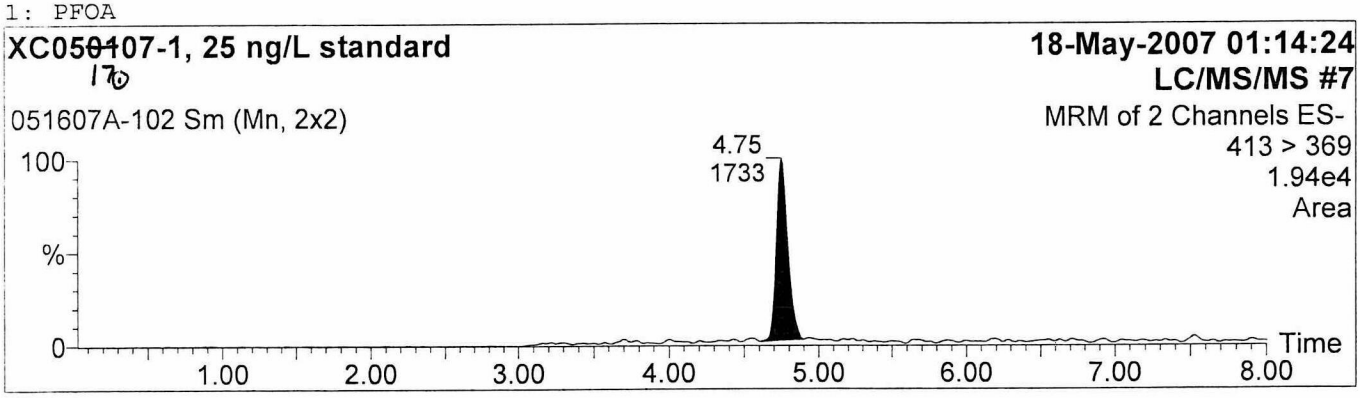
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

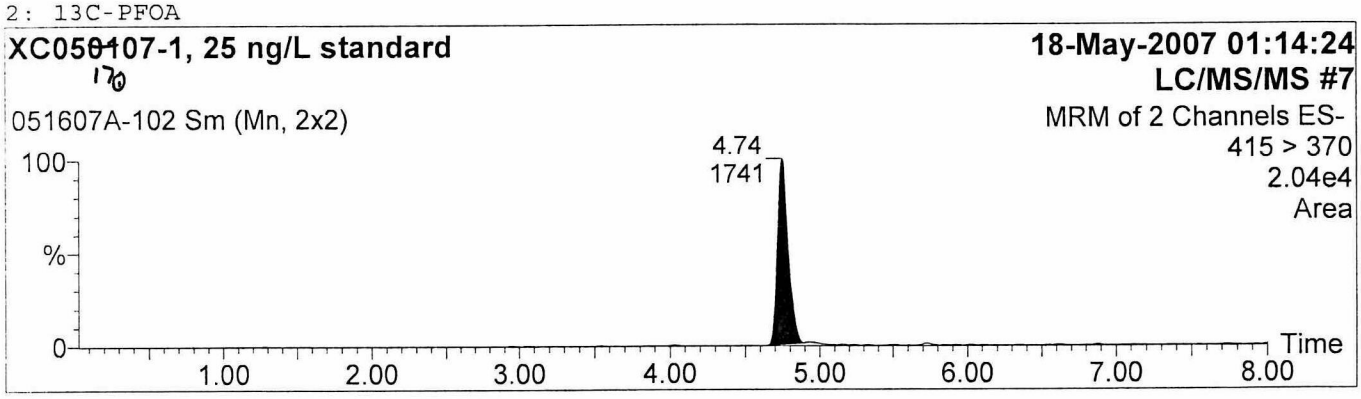
Printed: Mon May 21 09:05:19 2007

Name: 051607A-102
Text:

1: PFOA



2: 13C-PFOA



DRE MLC 5/21/07

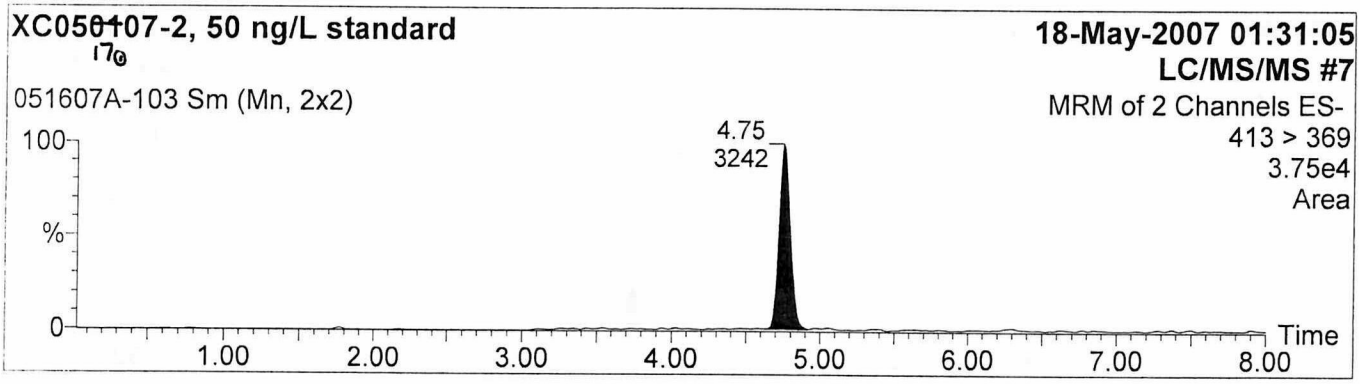
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

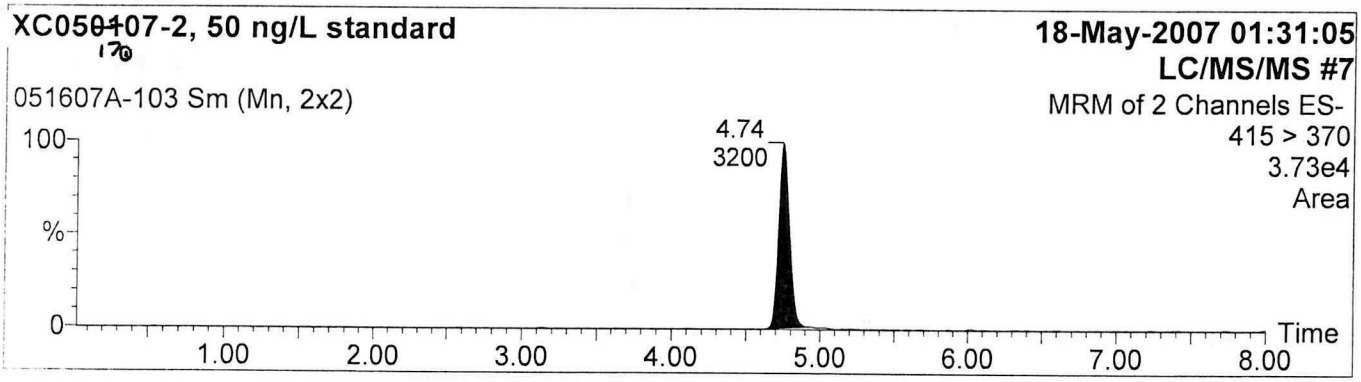
Printed: Mon May 21 09:05:19 2007

Name: 051607A-103
Text:

1: PFOA



2: 13C-PFOA



DRE MLC 5/21/07

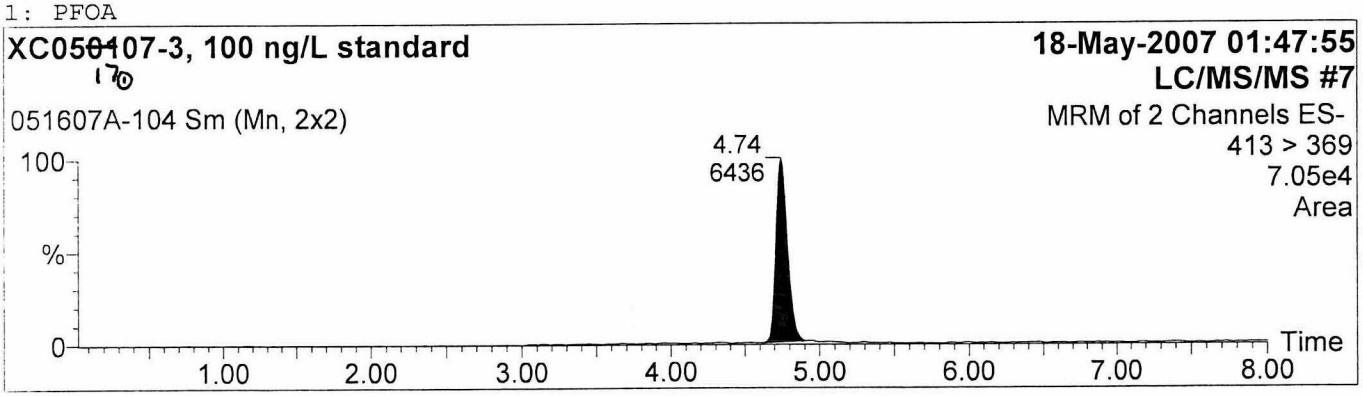
Quantify Sample Report
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

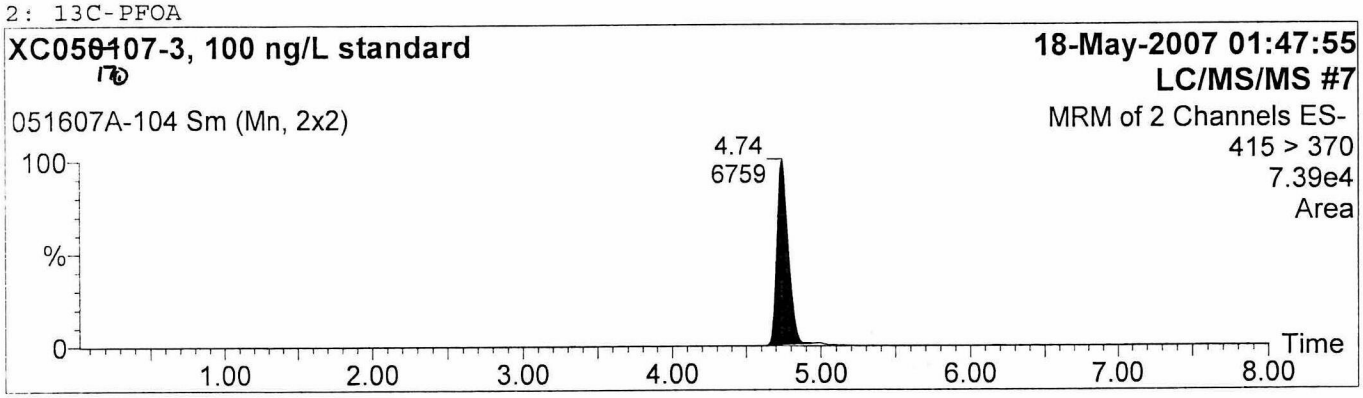
Printed: Mon May 21 09:05:19 2007

Name: 051607A-104
Text:

1: PFOA



2: 13C-PFOA



OK MLC 5/21/07

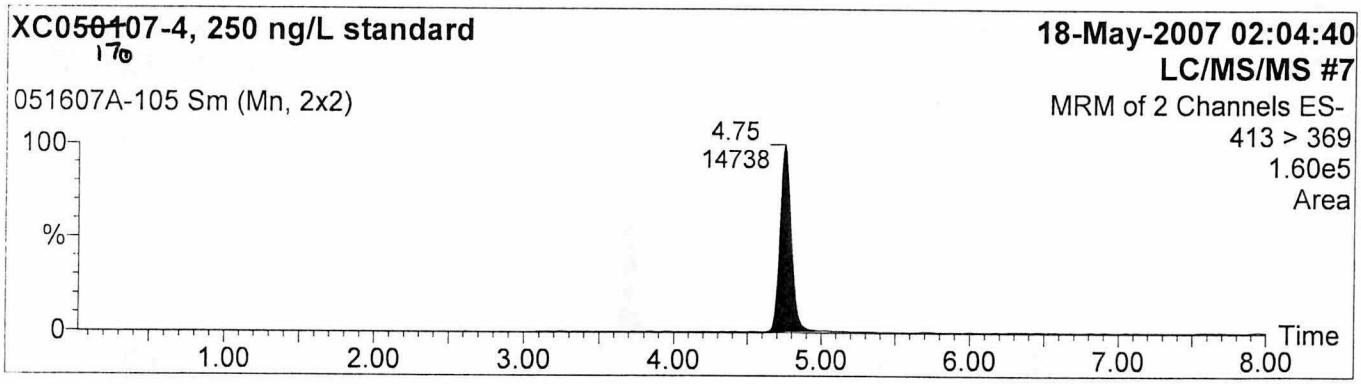
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

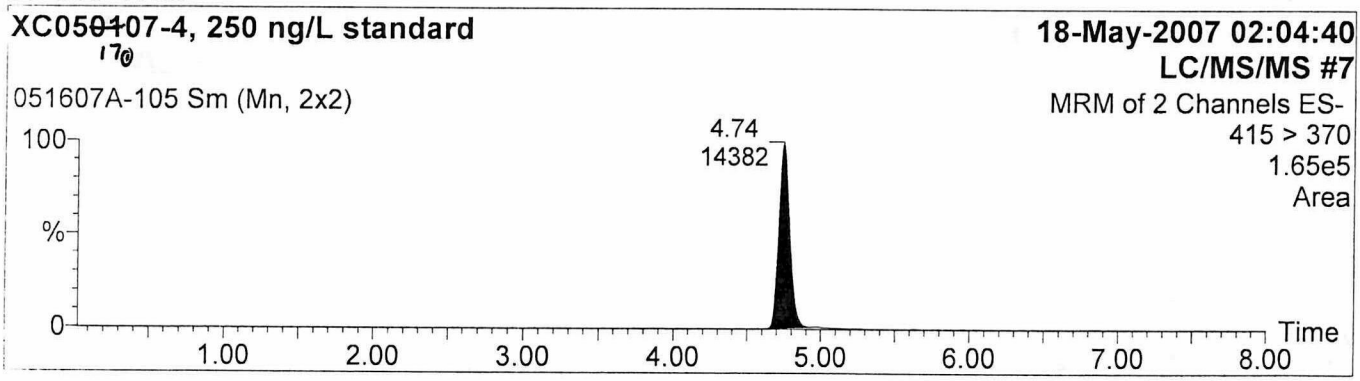
Printed: Mon May 21 09:05:19 2007

Name: 051607A-105
Text:

1: PFOA



2: 13C-PFOA



MLC 5/21/07

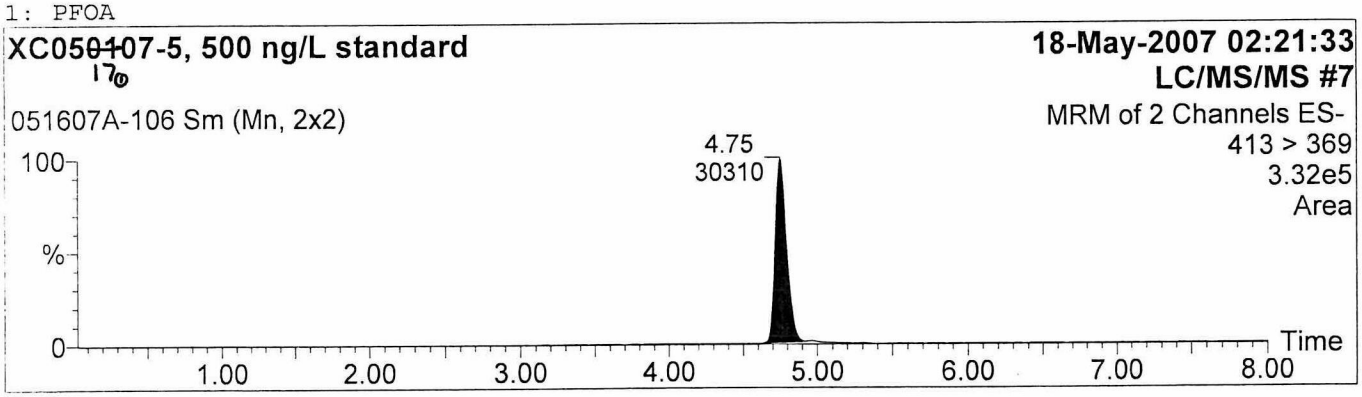
Quantify Sample Report
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

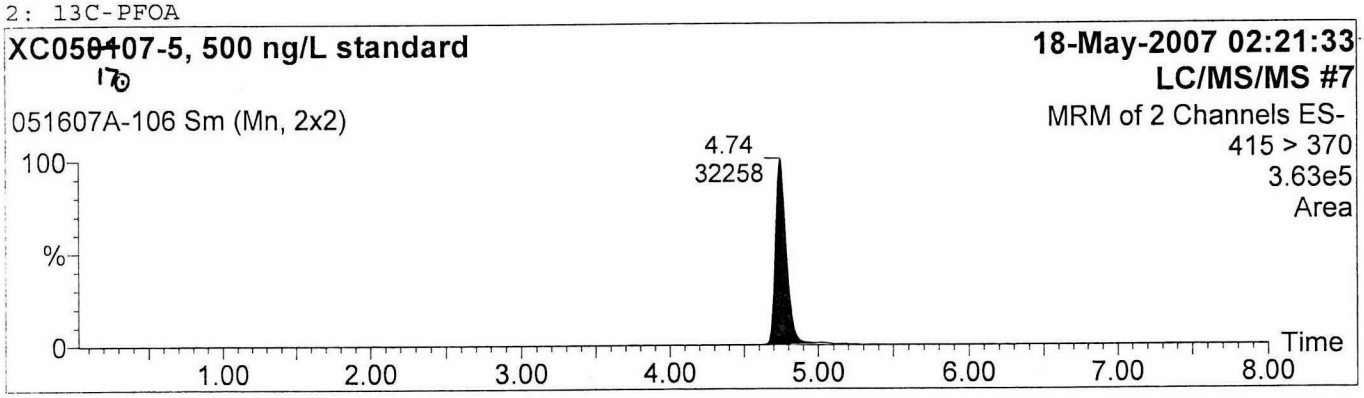
Printed: Mon May 21 09:05:19 2007

Name: 051607A-106
Text:

1: PFOA



2: 13C-PFOA



5/21/07

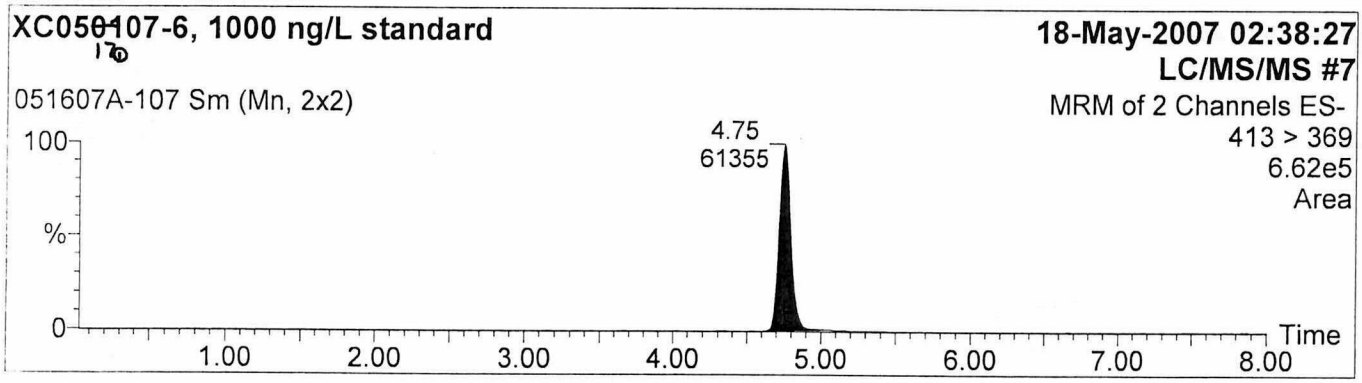
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

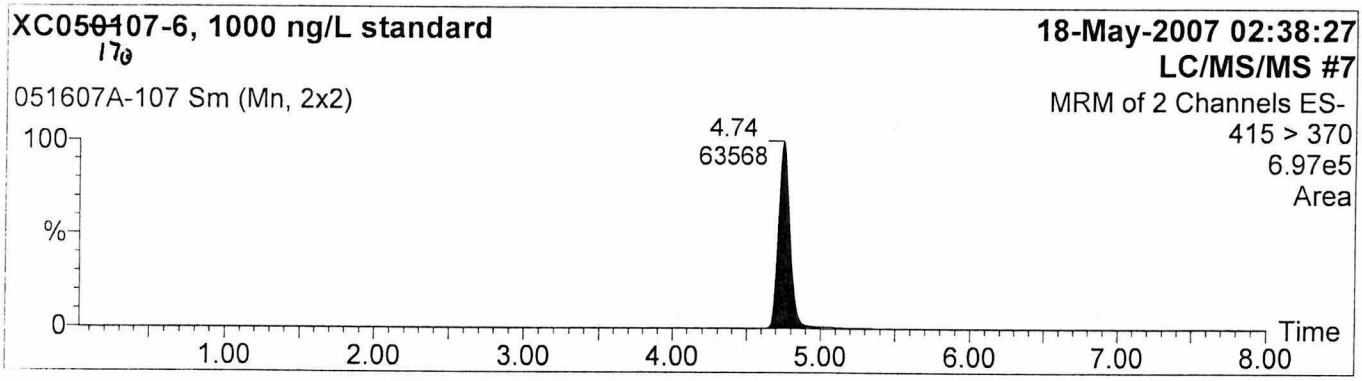
Printed: Mon May 21 09:05:19 2007

Name: 051607A-107
Text:

1: PFOA



2: 13C-PFOA



D PE NLC 5/21/07

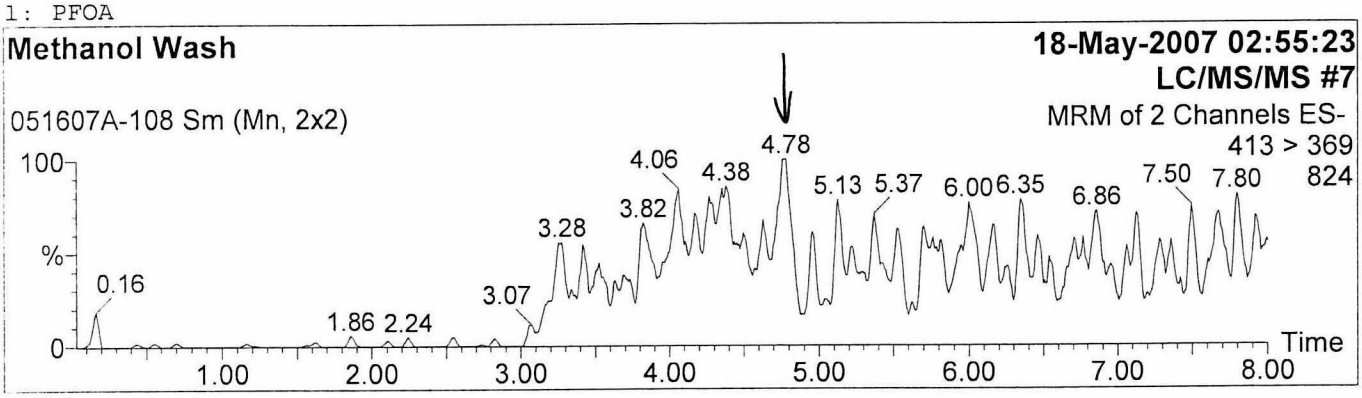
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

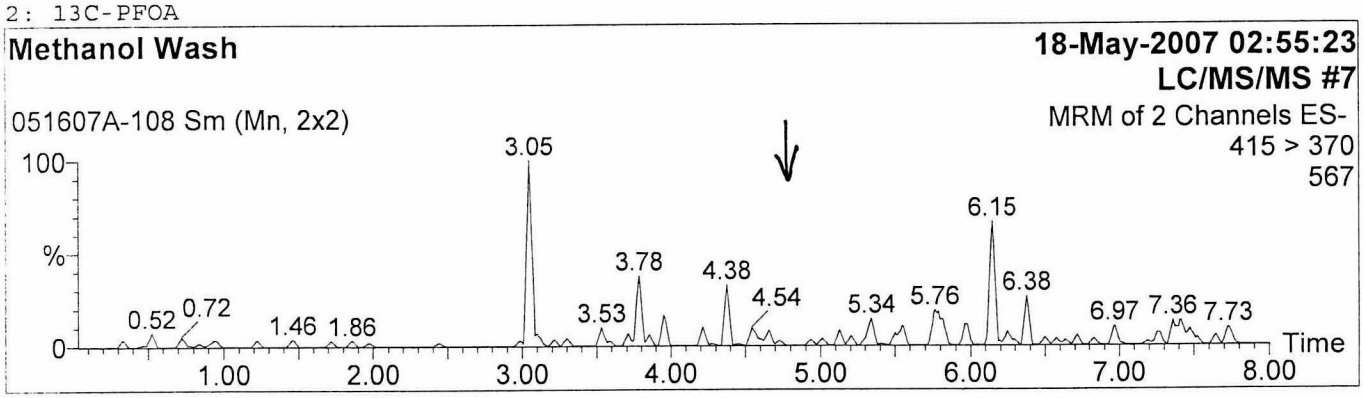
Printed: Mon May 21 09:05:19 2007

Name: 051607A-108
Text:

1: PFOA



2: 13C-PFOA



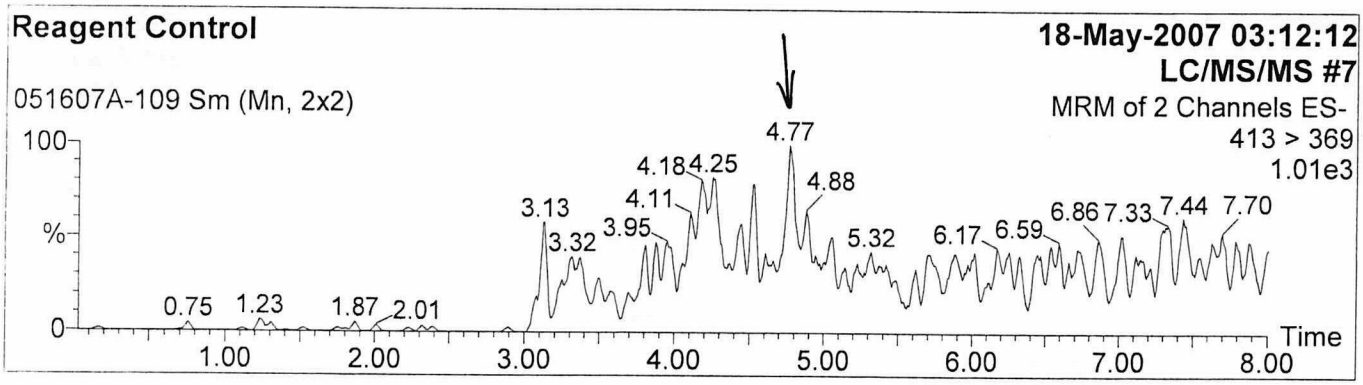
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

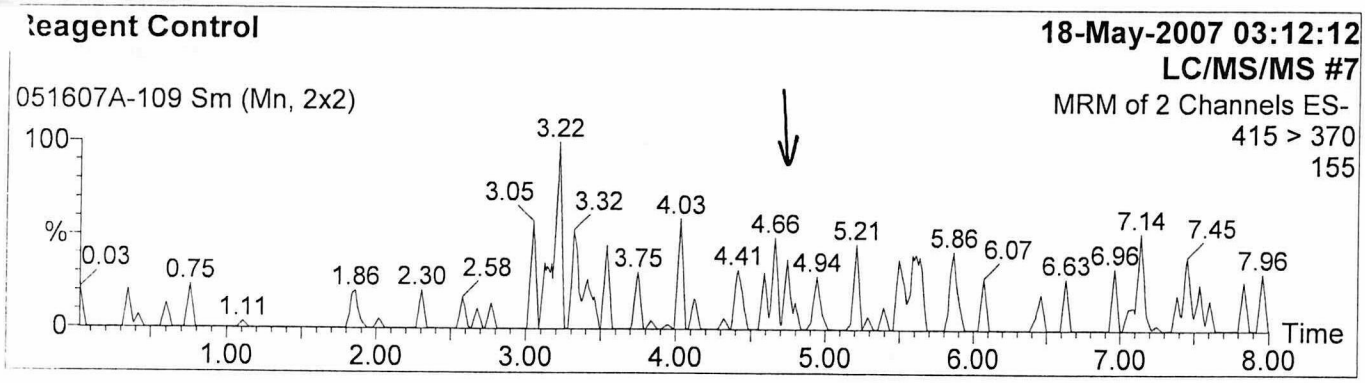
Printed: Mon May 21 09:05:19 2007

Name: 051607A-109
Text:

1: PFOA



2: 13C-PFOA



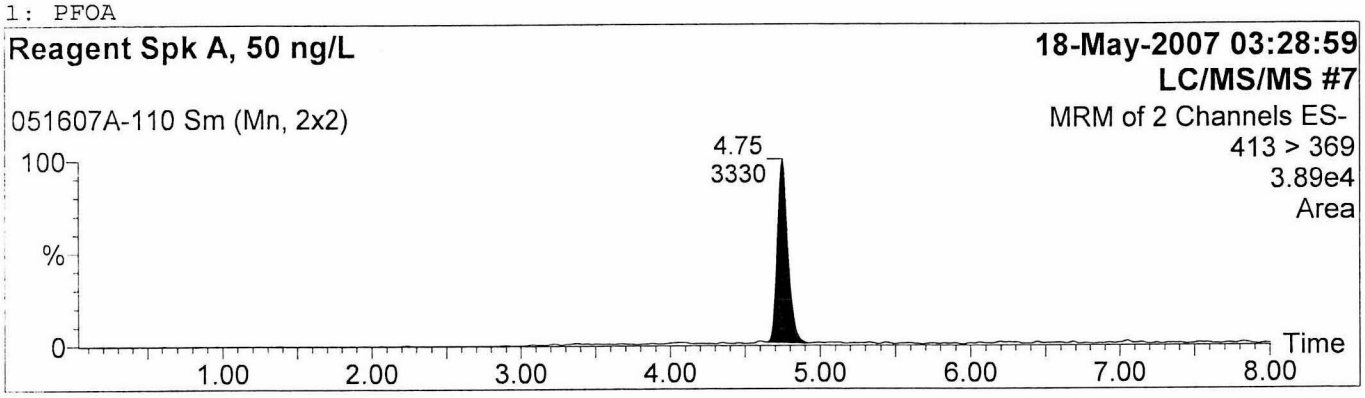
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

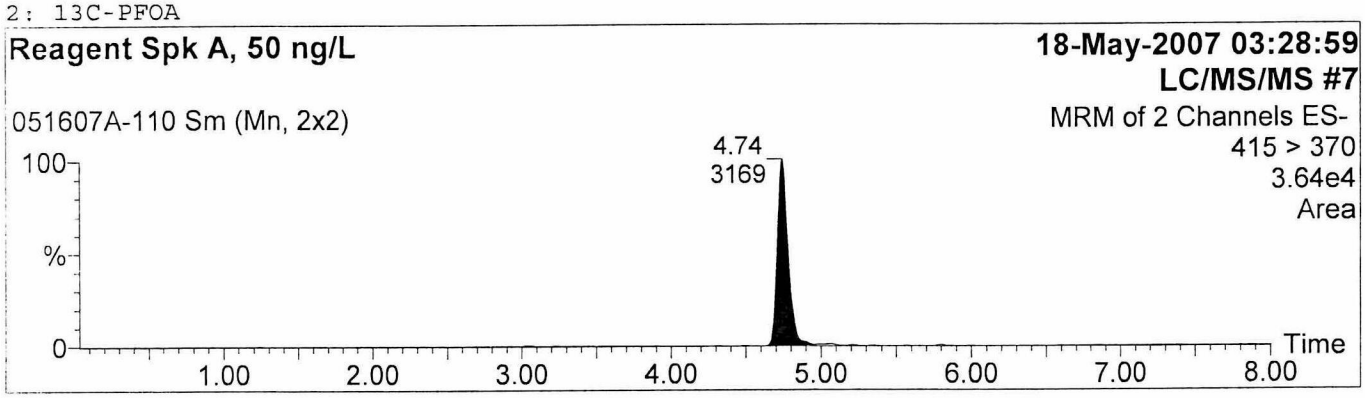
Printed: Mon May 21 09:05:19 2007

Name: 051607A-110
Text:

1: PFOA



2: 13C-PFOA



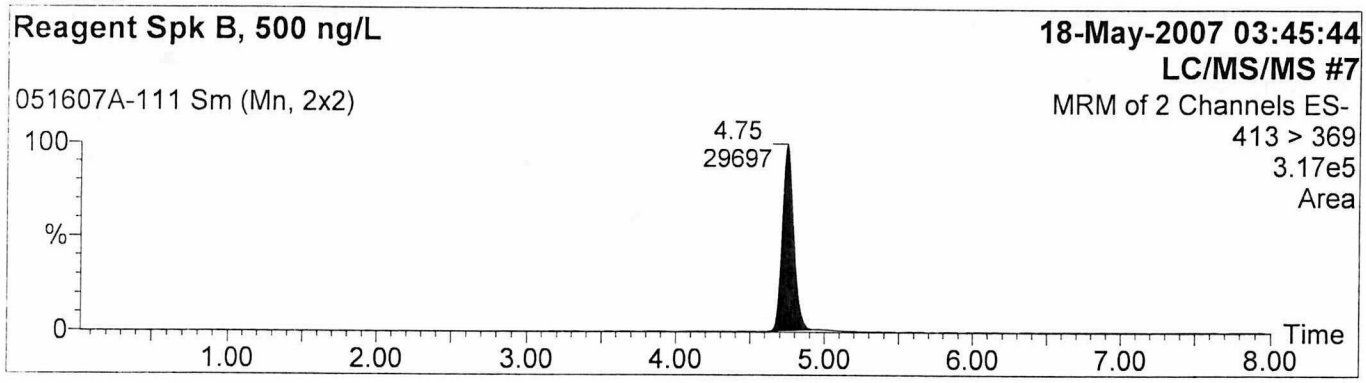
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

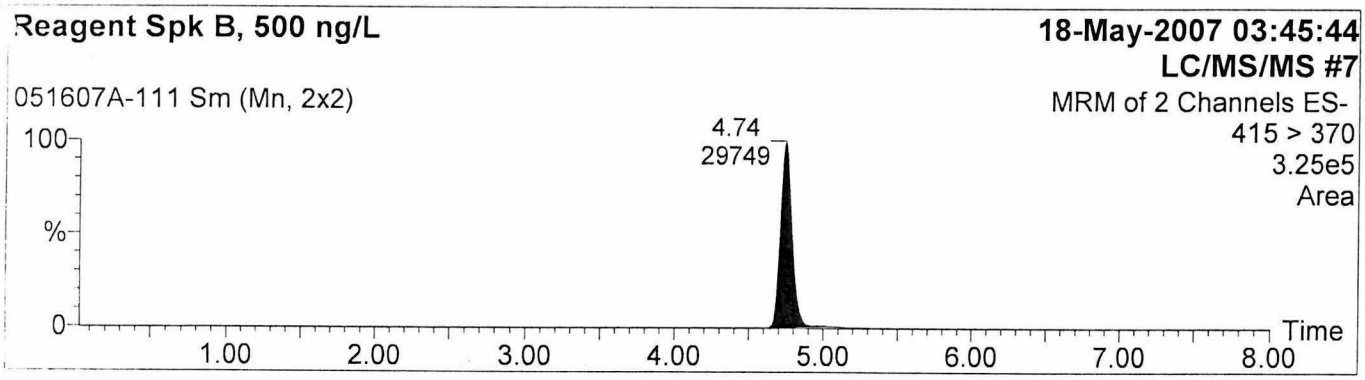
Printed: Mon May 21 09:05:19 2007

Name: 051607A-111
Text:

1: PFOA



2: 13C-PFOA



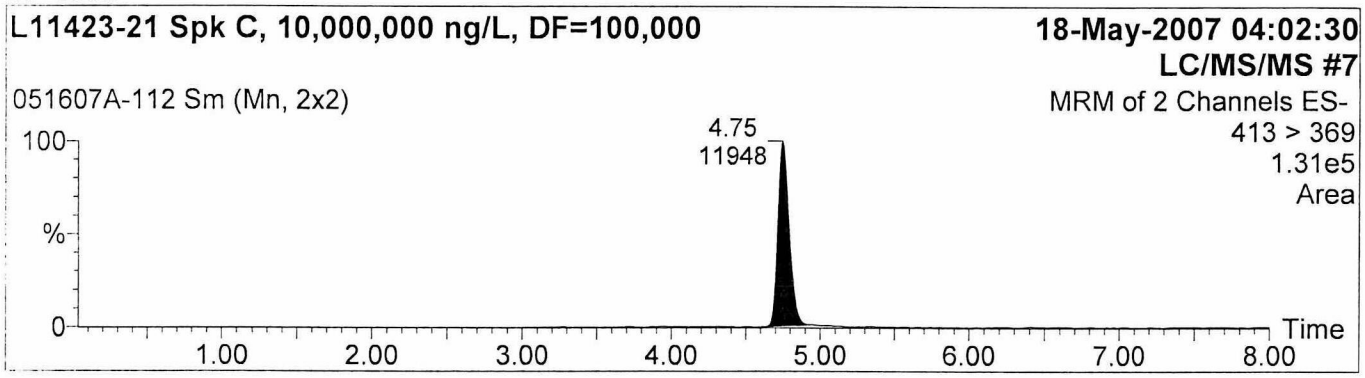
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

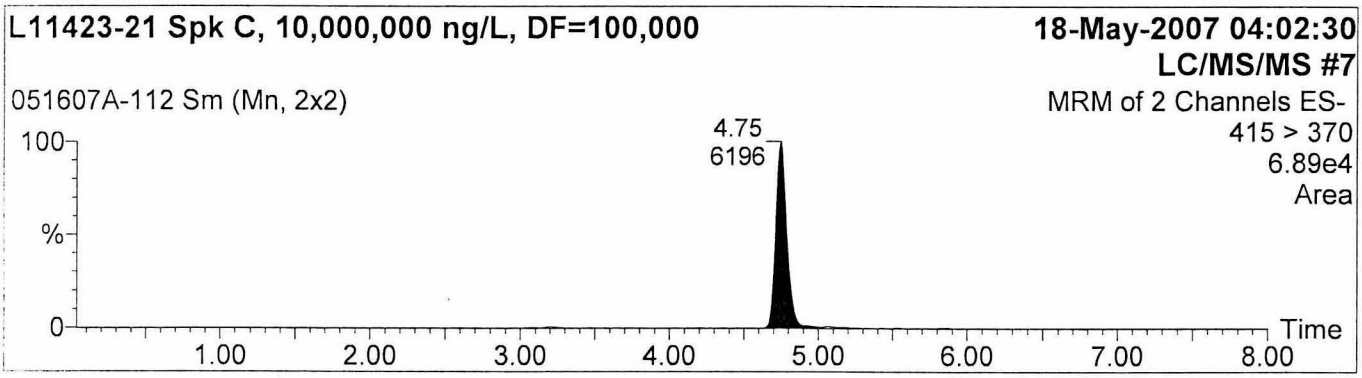
Printed: Mon May 21 09:05:19 2007

Name: 051607A-112
Text:

1: PFOA



2: 13C-PFOA



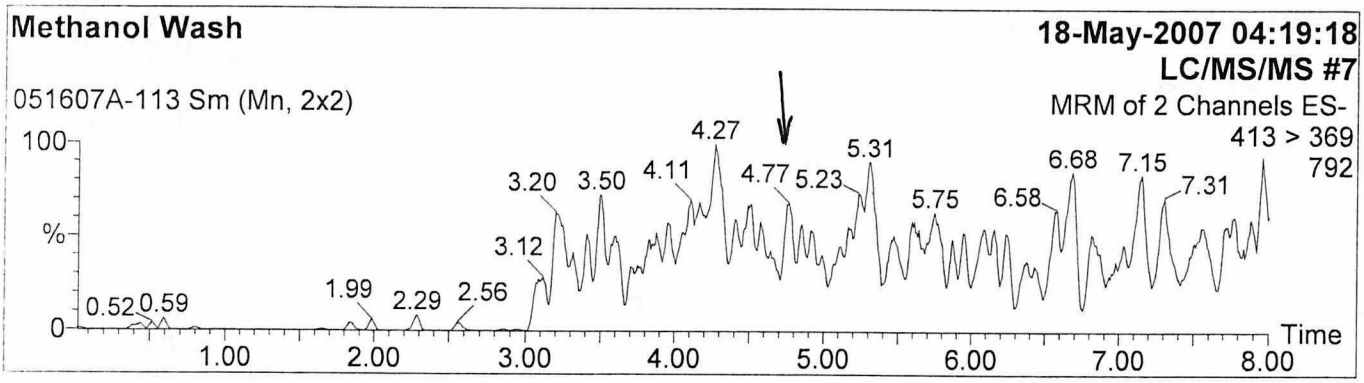
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

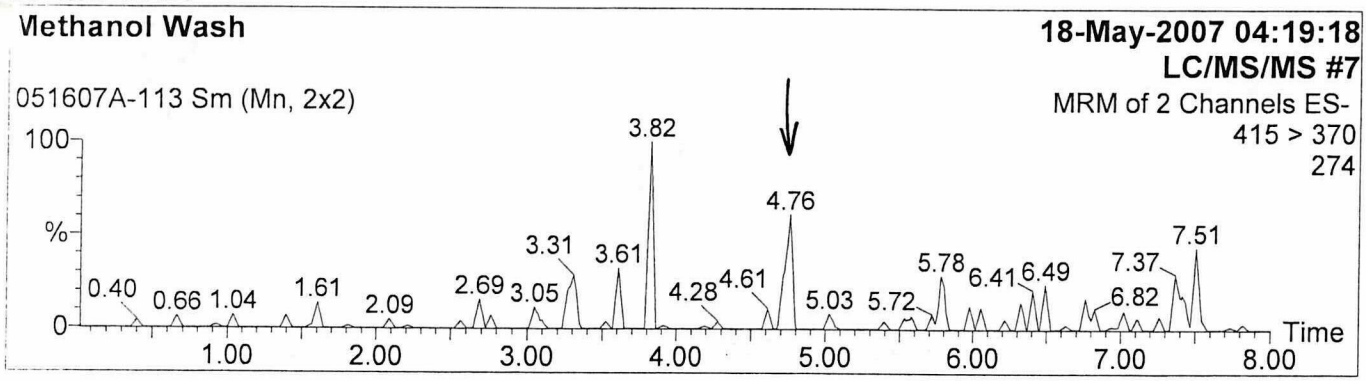
Printed: Mon May 21 09:05:19 2007

Name: 051607A-113
Text:

1: PFOA



2: 13C-PFOA



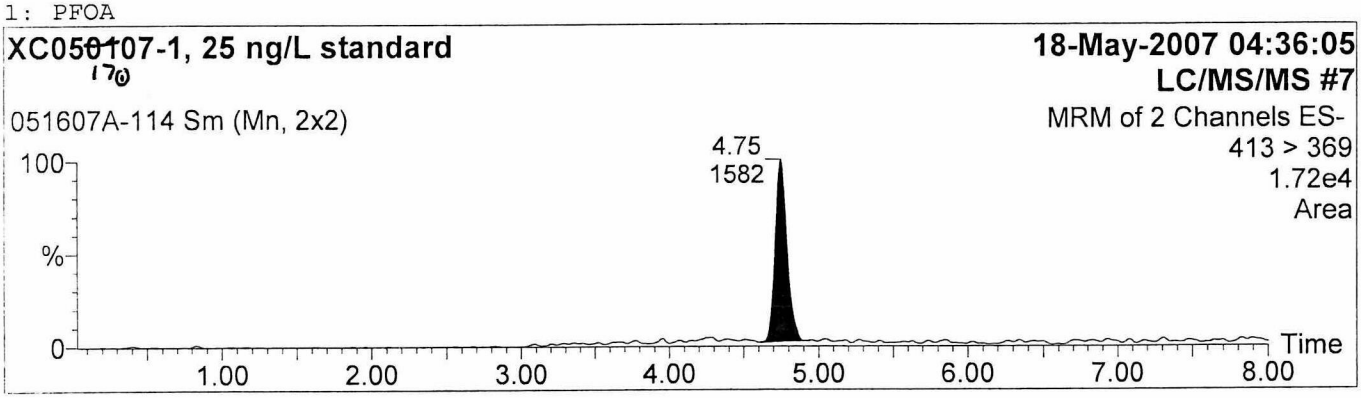
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

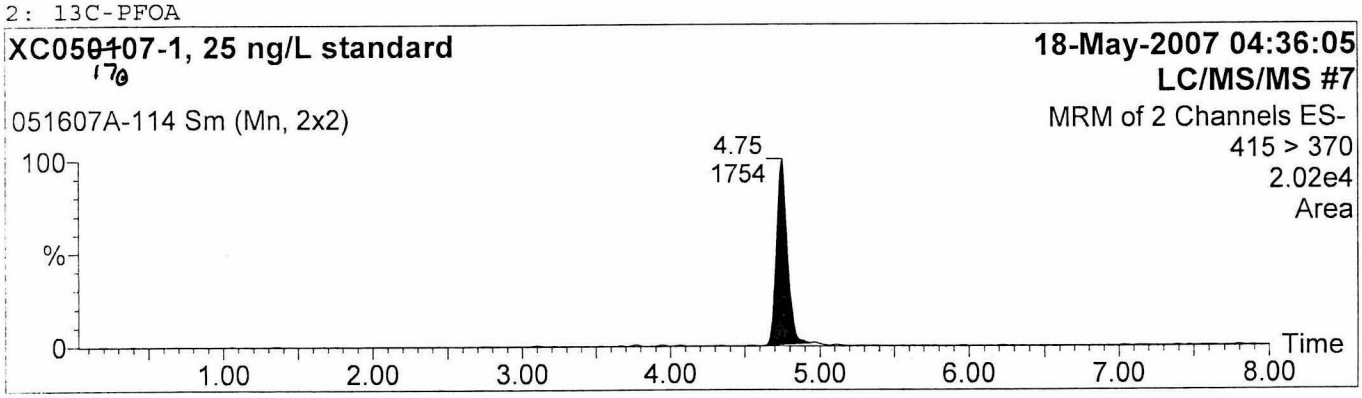
Printed: Mon May 21 09:05:19 2007

Name: 051607A-114
Text:

1: PFOA



2: 13C-PFOA



OK MLC 5/21/07

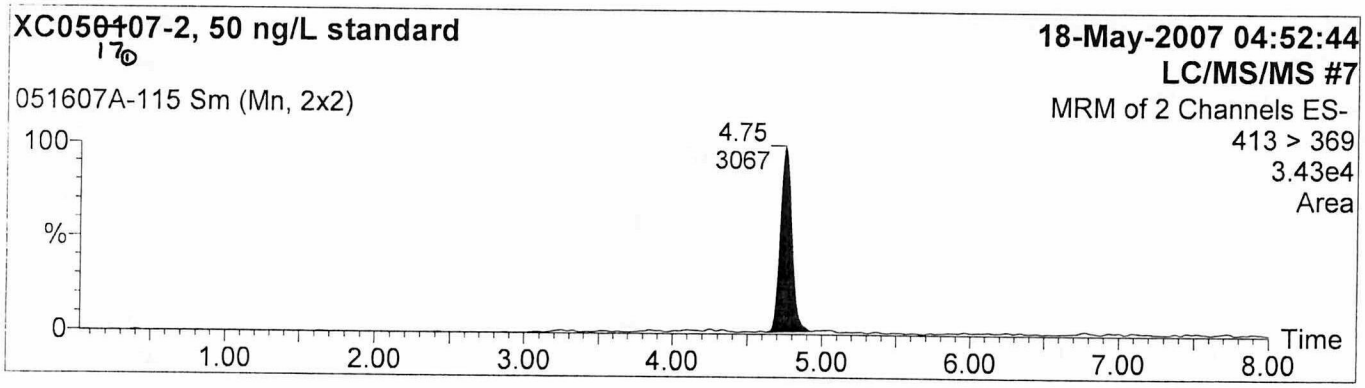
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

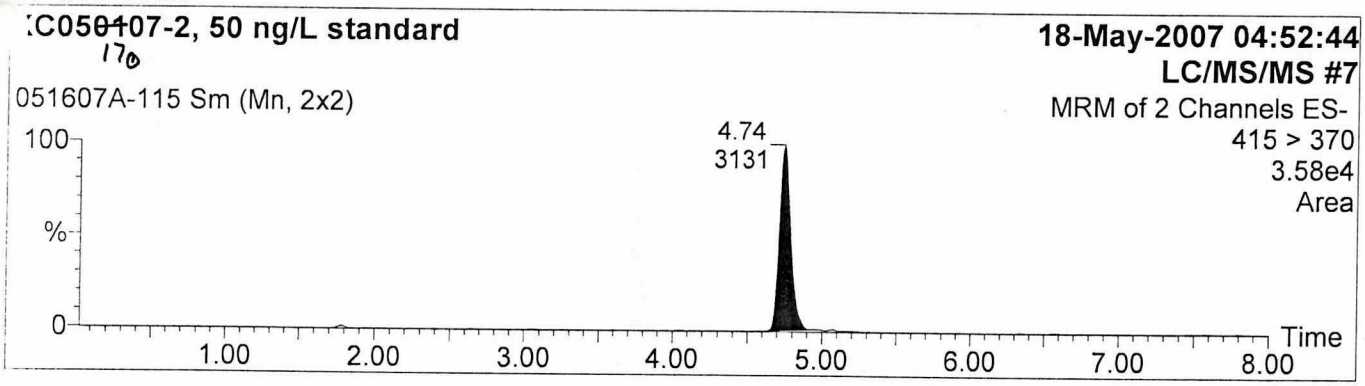
Printed: Mon May 21 09:05:19 2007

Name: 051607A-115
Text:

1: PFOA



2: 13C-PFOA



① RE mcc 5/21/07

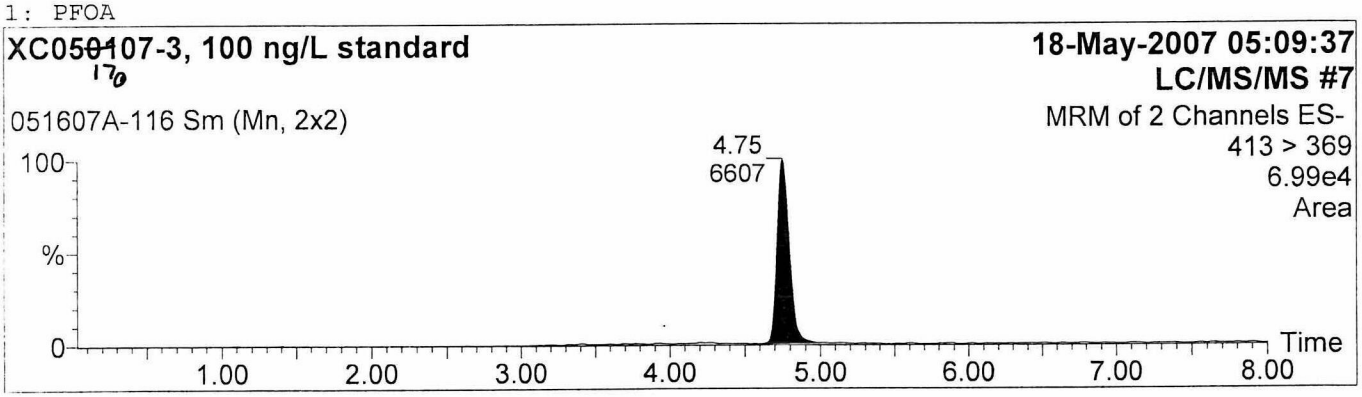
Quantify Sample Report
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

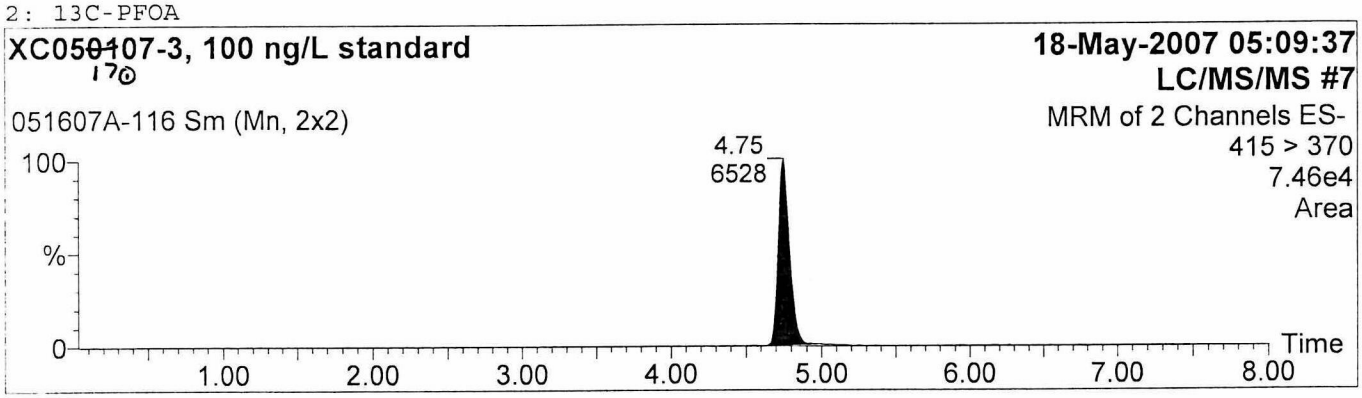
Printed: Mon May 21 09:05:19 2007

Name: 051607A-116
Text:

1: PFOA



2: 13C-PFOA



① RR mlc 5/21/07

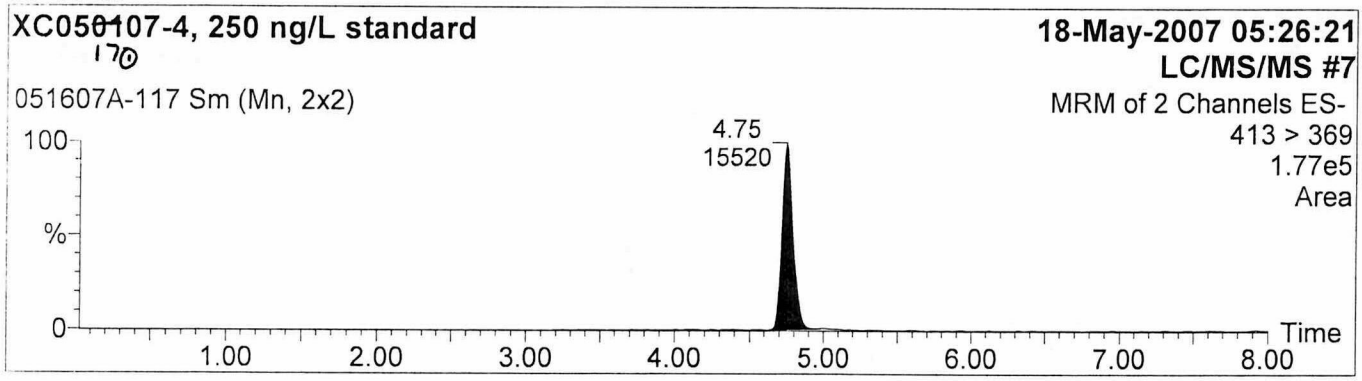
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

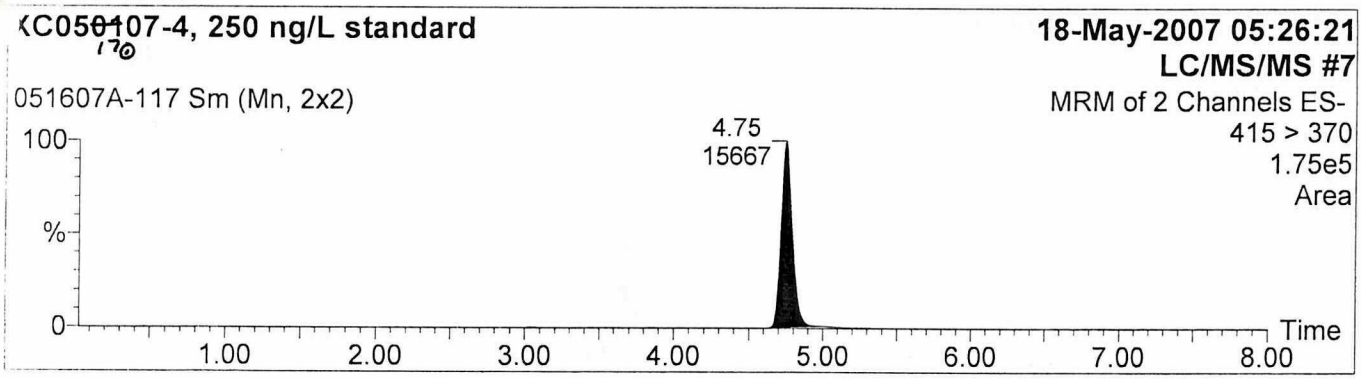
Printed: Mon May 21 09:05:19 2007

Name: 051607A-117
Text:

1: PFOA



2: 13C-PFOA



DEE MLC 5/21/07

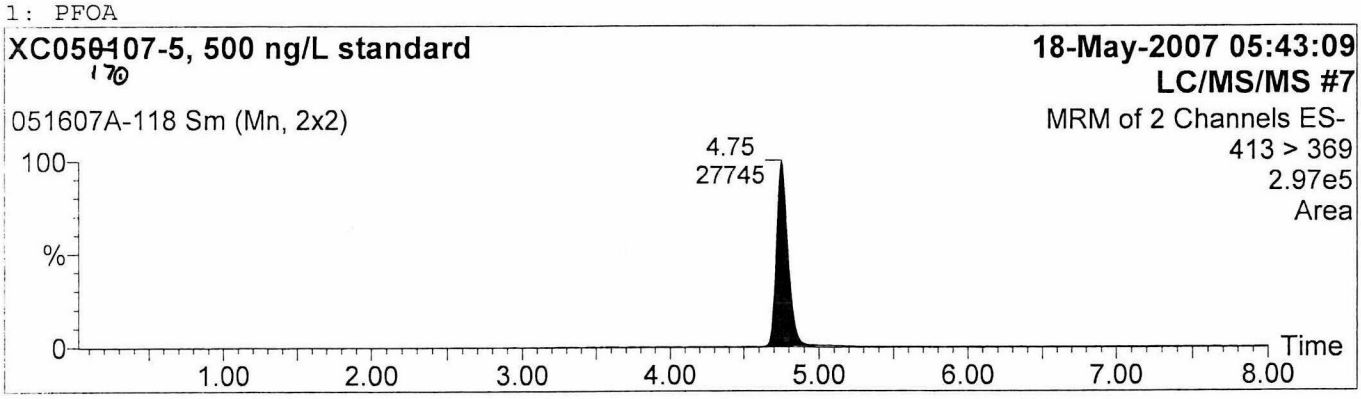
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

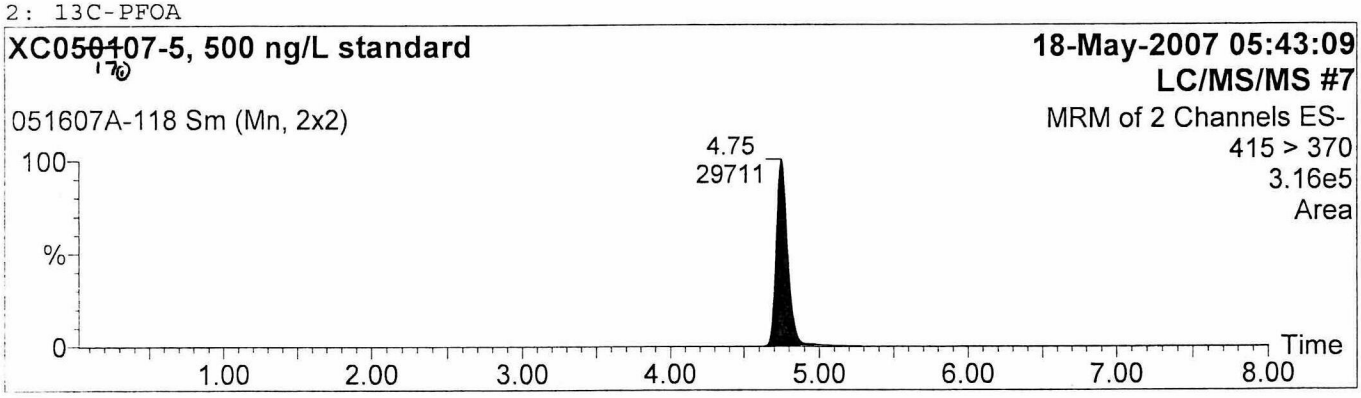
Printed: Mon May 21 09:05:19 2007

Name: 051607A-118
Text:

1: PFOA



2: 13C-PFOA



OK MLC 5/21/07

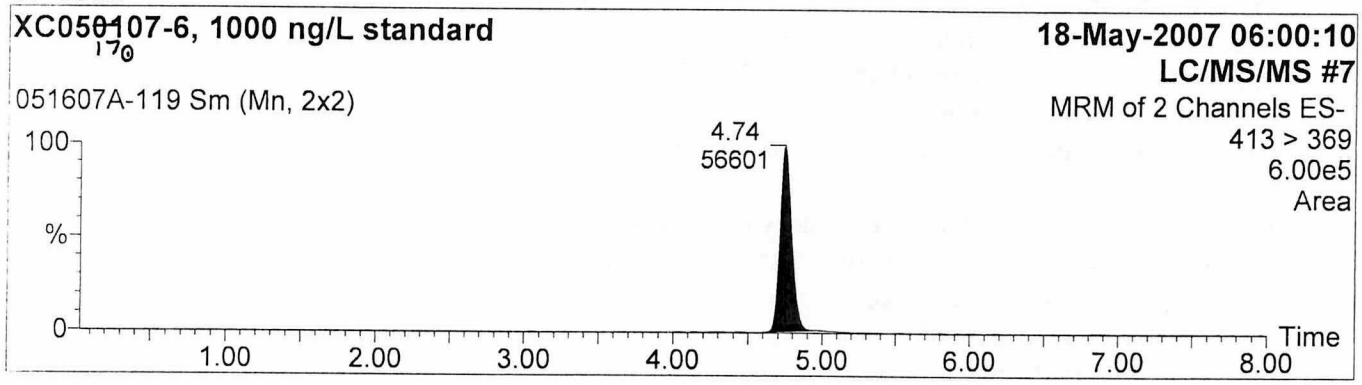
Quantify Sample Report
Study No: L11423, Set No: 051607A. Ext.Date: 05/16/07. Analyst: M.Cressley

Sample List: P:\Data\LCMSMS7\Masslynx\P593.PRO\SampleDB\051607A Dispersion Water
Last modified: Mon May 21 09:00:35 2007
Method: P:\Data\LCMSMS7\Masslynx\P593.PRO\MethDB\051507 PFOA & C13PFOA
Last modified: Tue May 15 08:29:30 2007
Job Code:

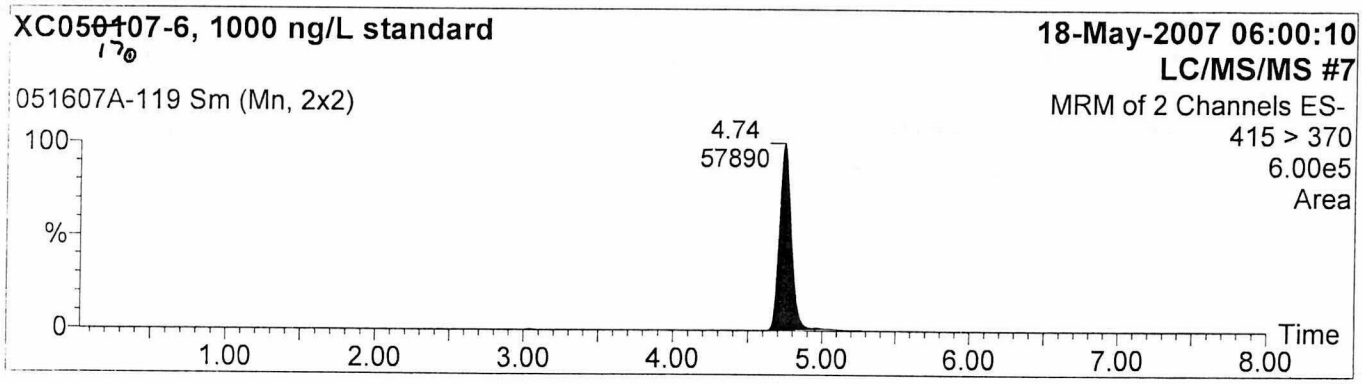
Printed: Mon May 21 09:05:19 2007

Name: 051607A-119
Text:

1: PFOA



2: 13C-PFOA



DRE MLC 5/21/07

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

Pentadecafluorooctanoic acid (Inventory #: SP6567), obtained from Aldrich (lot # 00202TC) was used to make a stock solution (ID # SS20743), which in turn, was used to make intermediate solutions (ID#'s SS20794, SS20795, SS20796, SS20797 and SS20798). These intermediate solutions are used to prepare extracted calibration standards, laboratory control samples, and matrix spike samples.

Pentadecafluorooctanoic acid (Inventory #: SP8065), obtained from Oakwood Products Inc., Fluorochem USA (lot # Y16G) was used to make a stock solution (ID # SS20744), which in turn, was used to make intermediate solutions (ID#'s SS20791, SS20792 and SS20793). 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.

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Fax: 814-231-1580

Standards - Primary

P0006567 > 6602

scription: Pentadecafluorooactaoic Acid, PFOA
pe: **Routine**
onsor Contact:
trix: Solids (Gravimetric)
talog #: 17146-8
#: 00202TC
S #: 335-67-1
vent: NONE
moved: False
ndition Recv'd: Room Temperature
ndition Stored: Room Temperature - with Standards
rrent Location: B1-105 / No Instrument / No Component
ndor: SIGMA-ALDRICH, INC.
mpany Name:
ntact:
one #:
es: Sample was stored in its original packing until a C of A and expiration date was abtained from Sigma. CEE 10/26/05

Date Received: 10/21/2005
Expires: 10/20/2007 12:00:00AM
Special: Moderately corrosive
Flammable: Non-combustible (will not burn)
Health: Highly toxic
Reactive: No significant hazard
Gross: 45.29084
Gross Units: g
Net Balance: 4.06041
Net Units: g

Aliquot - SP0006567

ddition	Component	Withdrawal	Units	Component	Purpose	Computer	Created On / By	Project
5	Balance 14		g		INITIAL		10/26/2005 11:08:04AM Edwards, Chrissy	P0001400
		0.01040	Notes: Fluorochemical Neat g	Balance 17	PREPARE	BALANCE8	11/30/2005 9:19:42AM Kravets, Brittany	
		0.104104	Notes: Prepare standards for DuPont. g	Balance 32	PREPARE	BALANCE7	12/9/2005 11:31:13AM Smith, Jennifer	P0001622
		0.10111	Notes: g	Balance 17	PREPARE	BALANCE8	12/12/2005 11:13:57AM Sheehan, Amy	
		0.0101	Notes: Prepared Standard for 008-APFO g	Balance 17	PREPARE	BALANCE8	2/7/2006 10:42:00AM Crespi, Frances	
		0.10111	Notes: Prepare Secondary Standards g	Balance 17	PREPARE	BALANCE8	4/6/2006 11:02:48AM Sheehan, Amy	P0000418
		0.01006	Notes: g	Balance 17	PREPARE	BALANCE8	4/11/2006 3:30:50PM Ingram, Devon	
		0.10112	Notes: Prepare secondary standards g	Balance 17	PREPARE	BALANCE8	5/19/2006 11:32:31AM Sheehan, Amy	P0000418

Description:	Pentadecafluorooactaoic Acid, PFOA
Type:	**Routine**
Sponsor Contact:	
Matrix:	Solids (Gravimetric)
Catalog #:	17146-8
Lot #:	00202TC
CAS #:	335-67-1
Solvent:	NONE
Removed:	False
Condition Recv'd:	Room Temperature
Condition Stored:	Room Temperature - with Standards
Current Location:	B1-105 / No Instrument / No Component
Vendor:	SIGMA-ALDRICH, INC.
Company Name:	
Contact:	
Phone #:	
Notes:	Sample was stored in its original packing until a C of A and expiration date was abtained from Sigma. CEE 10/26/05

Date Received:	10/21/2005
Expires:	10/20/2007 12:00:00AM
Special:	Moderately corrosive
Flammable:	Non-combustible (will not burn)
Health:	Highly toxic
Reactive:	No significant hazard
Gross:	45.29084
Gross Units:	g
Net Balance:	4.06041
Net Units:	g

Aliquot - SP0006567, cont'd

Addition	Component	Withdrawal	Units	Component	Purpose	Computer	Created On / By	Project
			Notes:					
		0.10093	g	Balance 17	PREPARE	BALANCE8	6/7/2006 1:06:23PM Ingram, Devon	
			Notes:	Prepare secondary standards				
		0.10112	g	Balance 17	PREPARE	BALANCE8	10/4/2006 9:47:56AM Sheehan, Amy	P0000418
			Notes:					
		0.010145	g	Balance 32	PREPARE	BALANCE7	10/10/2006 2:19:46PM Ingram, Devon	
			Notes:	Prepare secondary standards for PFOA analysis				
		0.00682	g	Balance 30	ANALYSIS	BALANCE4	1/18/2007 9:15:05AM Sheehan, Amy	P0000418
			Notes:	Used to make Sample.				
		0.07020	g	Balance 30	ANALYSIS	BALANCE4	1/18/2007 9:15:05AM Sheehan, Amy	P0000418
			Notes:	Used to make Sample				
		0.10114	g	Balance 17	PREPARE	BALANCE8	2/6/2007 2:00:54PM Crespi, Frances	P0001622
			Notes:					
		0.010114	g	Balance 32	PREPARE	BALANCE7	3/19/2007 12:01:04PM Crespi, Frances	P0002709
			Notes:					
		0.10112	g	Balance 17	PREPARE	BALANCE8	3/28/2007 1:57:31PM Sheehan, Amy	P0000418
			Notes:					

Characterization - SP0006567

Purity	Date Characterized	Date Expires	Characterized By	Created By
98.9 %	12/01/2004	10/20/2007	Sigma-Aldrich	Edwards, Chrissy

SIGMA-ALDRICH

Certificate of Analysis

Product Name	Pentadecafluorooctanoic acid, 96%
Product Number	171468
Product Brand	Aldrich
CAS Number	335-67-1
Molecular Formula	$CF_3(CF_2)_6COOH$
Molecular Weight	414.07

TEST	SPECIFICATION	LOT 00202TC RESULTS
APPEARANCE	WHITE TO OFF-WHITE POWDER	WHITE CRYSTALS
INFRARED SPECTRUM	CONFORMS TO STRUCTURE AND STANDARD AS	CONFORMS TO STRUCTURE.
TITRATION	95.5% - 104.5% (WITH NAOH)	101.6% (WITH NAOH)
GAS LIQUID CHROMATOGRAPHY	95.5% (MINIMUM)	98.9%
TITRATION	2 % H2O (MAXIMUM)	NO H2O DETECTED(WITH "KARL FISCHER" REAGENT)
QUALITY CONTROL ACCEPTANCE DATE		DECEMBER 2004

Ronnie J. Martin, Supervisor
Quality Control
Milwaukee, Wisconsin USA

SP0006567

SIGMA-ALDRICH

6000 N. Teutonia Ave.
Milwaukee, WI 53209 USA
Tel: (800) 558-9160 • (414) 438-3850
Fax: (800) 962-9591 • (414) 273-4979
e-mail: aldrich@sial.com

Date: October 26, 2005

Attn: Chrisy Edwards
Fax: 814-231-1580

In response to your request for the shelf life of Aldrich Product(s):

Aldrich Product Number: 17146-8
Description: Pentadecafluorooctanoic acid, 96%

Aldrich has not conducted specific tests for the shelf life under all conditions for this material. However, based on the history of our stock we find that this product should be stable for at least two years from the date of receipt, when stored tightly closed in the original container in which it was packaged and under the conditions listed on the label.

If we can be of any further assistance to you please feel free to contact me.

Sincerely,

Ronnie Martin
Chemist, Quality Control

SP0006567

We are committed to the Success of our Customers, Employees and Shareholders through leadership in Life Science, High Technology and Service.

SP0006567

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

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CHRISSY EDWARDS PIN-394718
EXYGEN RESEARCH
3048 RESEARCH DR
STATE COLLEGE PA 16801
USA

Customer Service 800-325-3010 ◦ http//www.sigma-aldrich.com

PAGE	1 of 1	DELIVERY#	820728515

DATE	SOLD TO ACCT.	SOLD TO NAME	PURCHASE ORDER NUMBER	REFERENCE #
10/20/2005	49435591	EXYGEN RESEARCH	6891CE10	34309482

ROUTE	PERSON TO CONTACT	PHONE NUMBER
DHL GROUND FCA SHIPPING POINT	KENDRA LONG	8142318032

STOCK NO.	LOT NO.	ORDERED	SHIPPED	BACK ORD.	DESCRIPTION
171468-5G	00202TC	1	1	0	PENTADECAFLUOROOCTANOIC ACID, 96% CNTRY OF OR: US

Received ok CEE 10/21/05

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SP0006567

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ALLENTOWN, PA 18106
610-391-9107

WEIGHT(LBS) 001

TO: CEE 10/21/05

CHRISSY EDWARDS
EXYGEN RESEARCH
3048 RESEARCH DR
STATE COLLEGE PA

REF# 820728515

PIECES 1

ZIPCODE 16801

ORIGIN ABE

SHIPMENT NO. 86700615783

SHIP DATE 10/20/05

ADDITIONAL INFORMATION
820728515 6891CE10

Track #:86700615783
Document #820728515

PHONE: 314-771-5765

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Phone: 814-272-1039
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Secondary Standard

Standard Information

ndard: **SS0020743**
tegory: Stock
scription: Pentadecafluorooctanoic Acid (PFOA) in Methanol (1000 ug/mL)

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/28/2007 2:40:34PM
Date Expires: 9/28/2007 11:05:26AM
Storage Condition: Refrigerated

eparation: Dissolve 0.10112 grams of PFOA in 100 mL of Methanol
OC: RECEIVED / B2-105 / IN0000709 / Refrigerator 33 / D0002259 / Refrigerator 33 / CC0001940 / Mar 28, 2007 2:44:02PM
LOGIN / No Location / Mar 28, 2007 2:40:33PM

Component Concentrations

Component	Concentration / Amount
Perfluorooctanoic Acid	1000.08 µg/mL
PFOA (TRIAL)	1000.08 µg/mL
eagent: RE0031862 / METHANOL HPLC / Date Expires: 03/19/2009	100 mL

Prepared From

Standard / Description	Aliquot	
SP0006567 / Pentadecafluorooactaoic Acid, PFOA / Perfluorooctanoic Acid	0.10112 g	98.9 %
SP0006567 / Pentadecafluorooactaoic Acid, PFOA / PFOA (TRIAL)	0.10112 g	98.9 %

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Secondary Standard

Standard Information

Standard: **SS0020794**
Category: Fortification
Description: Pentadecafluorooctanoic Acid (PFOA) in Methanol (100 ug/mL)

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/29/2007 12:46:32PM
Date Expires: 6/29/2007 11:05:26AM
Storage Condition: Refrigerated

Preparation: Dilute 10 mL of the 1000 ug/mL to a final volume of 100 mL in methanol.
ICOC: RECEIVED / B2-3rd Floor Lab / CC0001940 / Mar 29, 2007 12:47:26PM
LOGIN / No Location / Mar 29, 2007 12:46:32PM

Component Concentrations

Component	Concentration / Amount
Perfluorooctanoic Acid	100.008 µg/mL
PFOA (TRIAL)	100.008 µg/mL
Reagent: RE0032031 / METHANOL HPLC / Date Expires: 03/26/2009	90 mL

Prepared From

Standard / Description	Aliquot	
SS0020743 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (1000 ug/mL) / Perfluorooctanoic Acid	10 mL	1000.08 µg/mL
SS0020743 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (1000 ug/mL) / PFOA (TRIAL)	10 mL	1000.08 µg/mL

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Secondary Standard

Standard Information

ndard: **SS0020795**
tegory: Fortification
scription: Pentadecafluorooctanoic Acid (PFOA) in Methanol (10 ug/mL)

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/29/2007 12:47:34PM
Date Expires: 6/29/2007 11:05:26AM
Storage Condition: Refrigerated

paration: Dilute 10 mL of the 100 ug/mL to a final volume of 100 mL in methanol.
OC: RECEIVED / B2-3rd Floor Lab / CC0001940 / Mar 29, 2007 12:48:21PM
LOGIN / No Location / Mar 29, 2007 12:47:34PM

Component Concentrations

Component	Concentration / Amount
Perfluorooctanoic Acid	10.0008 µg/mL
PFOA (TRIAL)	10.0008 µg/mL
eagent: RE0032031 / METHANOL HPLC / Date Expires: 03/26/2009	90 mL

Prepared From

Standard / Description	Aliquot	
SS0020794 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (100 ug/mL) / Perfluorooctanoic Acid	10 mL	100.008 µg/mL
SS0020794 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (100 ug/mL) / PFOA (TRIAL)	10 mL	100.008 µg/mL

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Secondary Standard

Standard Information

Standard: **SS0020796**
Category: Fortification
Description: Pentadecafluorooctanoic Acid (PFOA) in Methanol (1.0 ug/mL)

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/29/2007 12:48:32PM
Date Expires: 6/29/2007 11:05:26AM
Storage Condition: Refrigerated

Preparation: Dilute 10 mL of the 10 ug/mL to a final volume of 100 mL in methanol.
ICOC: RECEIVED / B2-3rd Floor Lab / CC0001940 / Mar 29, 2007 12:49:21PM
LOGIN / No Location / Mar 29, 2007 12:48:31PM

Component Concentrations

Component	Concentration / Amount
Perfluorooctanoic Acid	1.00008 µg/mL
PFOA (TRIAL)	1.00008 µg/mL
Reagent: RE0032031 / METHANOL HPLC / Date Expires: 03/26/2009	90 mL

Prepared From

Standard / Description	Aliquot	
SS0020795 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (10 ug/mL) / Perfluorooctanoic Acid	10 mL	10.0008 µg/mL
SS0020795 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (10 ug/mL) / PFOA (TRIAL)	10 mL	10.0008 µg/mL

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Secondary Standard

Standard Information

andard: **SS0020797**
ategory: Fortification
escription: Pentadecafluorooctanoic Acid (PFOA) in Methanol (0.1 ug/mL)

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/29/2007 12:49:33PM
Date Expires: 6/29/2007 11:05:26AM
Storage Condition: Refrigerated

eparation: Dilute 10 mL of the 1.0 ug/mL to a final volume of 100 mL in methanol.
OC: RECEIVED / B2-3rd Floor Lab / CC0001940 / Mar 29, 2007 12:50:29PM
LOGIN / No Location / Mar 29, 2007 12:49:33PM

Component Concentrations

Component	Concentration / Amount	
Perfluorooctanoic Acid	0.100008	µg/mL
PFOA (TRIAL)	0.100008	µg/mL
Reagent: RE0031706 / OmniSolv Water / Date Expires: 12/31/2007	90	mL

Prepared From

Standard / Description	Aliquot	
SS0020796 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (1.0 ug/mL) / Perfluorooctanoic Acid	10 mL	1.00008 µg/mL
SS0020796 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (1.0 ug/mL) / PFOA (TRIAL)	10 mL	1.00008 µg/mL

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Secondary Standard

Standard Information

Standard: **SS0020798**
Category: Fortification
Description: Pentadecafluorooctanoic Acid (PFOA) in Methanol (0.01 ug/mL)

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/29/2007 12:50:42PM
Date Expires: 6/29/2007 11:05:26AM
Storage Condition: Refrigerated

Preparation: Dilute 10 mL of the 0.1 ug/mL to a final volume of 100 mL in methanol.
ICOC: RECEIVED / B2-3rd Floor Lab / CC0001940 / Mar 29, 2007 12:51:42PM
LOGIN / No Location / Mar 29, 2007 12:50:42PM

Component Concentrations

Component	Concentration / Amount
Perfluorooctanoic Acid	0.0100008 µg/mL
PFOA (TRIAL)	0.0100008 µg/mL
Reagent: RE0032031 / METHANOL HPLC / Date Expires: 03/26/2009	90 mL

Prepared From

Standard / Description	Aliquot	
SS0020797 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (0.1 ug/mL) / Perfluorooctanoic Acid	10 mL	0.100008 µg/mL
SS0020797 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (0.1 ug/mL) / PFOA (TRIAL)	10 mL	0.100008 µg/mL

Exygen RESEARCH

3058 Research Drive
State College, PA 16801

Phone: [illegible]4-272-1039
Fax: 814-231-1580

Standards - Primary

P0008065 > 8102

scription: Pentadecafluorooctanoic Acid (C8 Acid - PFOA)
pe: **Routine**
onsor Contact:
trix: Solids (Gravimetric)
talog #: 1319
#: Y16G
S #: 335-67-1
vent: NONE
moved: False
ndition Recv'd: Room Temperature
ndition Stored: Refrigerated - with Standards
rrent Location: B1-105 / Walkin Cooler 9 (IN0000678) / No Component
ndor: OAKWOOD PRODUCTS, INC.
mpany Name:
ntact:
one #:
es:

Date Received: 09/08/2006
Expires: 9/8/2008 12:00:00AM
Special: Unknown
Flammable: Slightly combustible (above 200F)
Health: Highly toxic
Reactive: Unknown
Gross: 16.88299
Gross Units: g
Net Balance: 2.75505
Net Units: g

Aliquot - SP0008065

ddition	Component	Withdrawal	Units	Component	Purpose	Computer	Created On / By	Project
5			g		INITIAL		9/8/2006 10:58:07AM Gallant, Krista	P0002561
		1.02041	Notes: g	Balance 14	ANALYSIS	BALANCE5	9/8/2006 11:28:37AM Gallant, Krista	P0002561
		0.10205	Notes: Stocks for fluorochemicals. g	Balance 17	PREPARE	BALANCE8	10/4/2006 10:07:35AM Sheehan, Amy	P0000418
		1.02044	Notes: g	Balance 17	PREPARE	BALANCE8	3/1/2007 11:05:32AM Edwards, Chrissy	P0002561
		0.10205	Notes: Stocks for Fluorochemicals g	Balance 17	PREPARE	BALANCE8	3/28/2007 2:31:42PM Sheehan, Amy	P0000418
			Notes:					

Characterization - SP0008065

Purity	Date Characterized	Date Expires	Characterized By	Created By
98 %	09/08/2006	09/08/2008	Certificate of Analysis from Oakwood Products, Inc.	Gallant, Krista

OAKWOOD PRODUCTS, INC.
1741 OLD DUNBAR ROAD
WEST COLUMBIA, SC 29172
Phone: 803-739-8800 FAX: 803-739-6957

72752

O R D E R

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

SHIP TO:
Exygen Research
Attn: CHRISSY EDWARDS
3048 Research Drive
State College, PA 16801
PO #8349CE10
(814) 231-8032

CUST NO.	SALESMAN NO.	P. O. NUMBER	SHIPPING INSTRUCTIONS	COL	PPD	SHIP DATE	TERMS	INVOICE DATE
10926		8349CE10	OVT	X		09/05/06	net 30	09/05/06

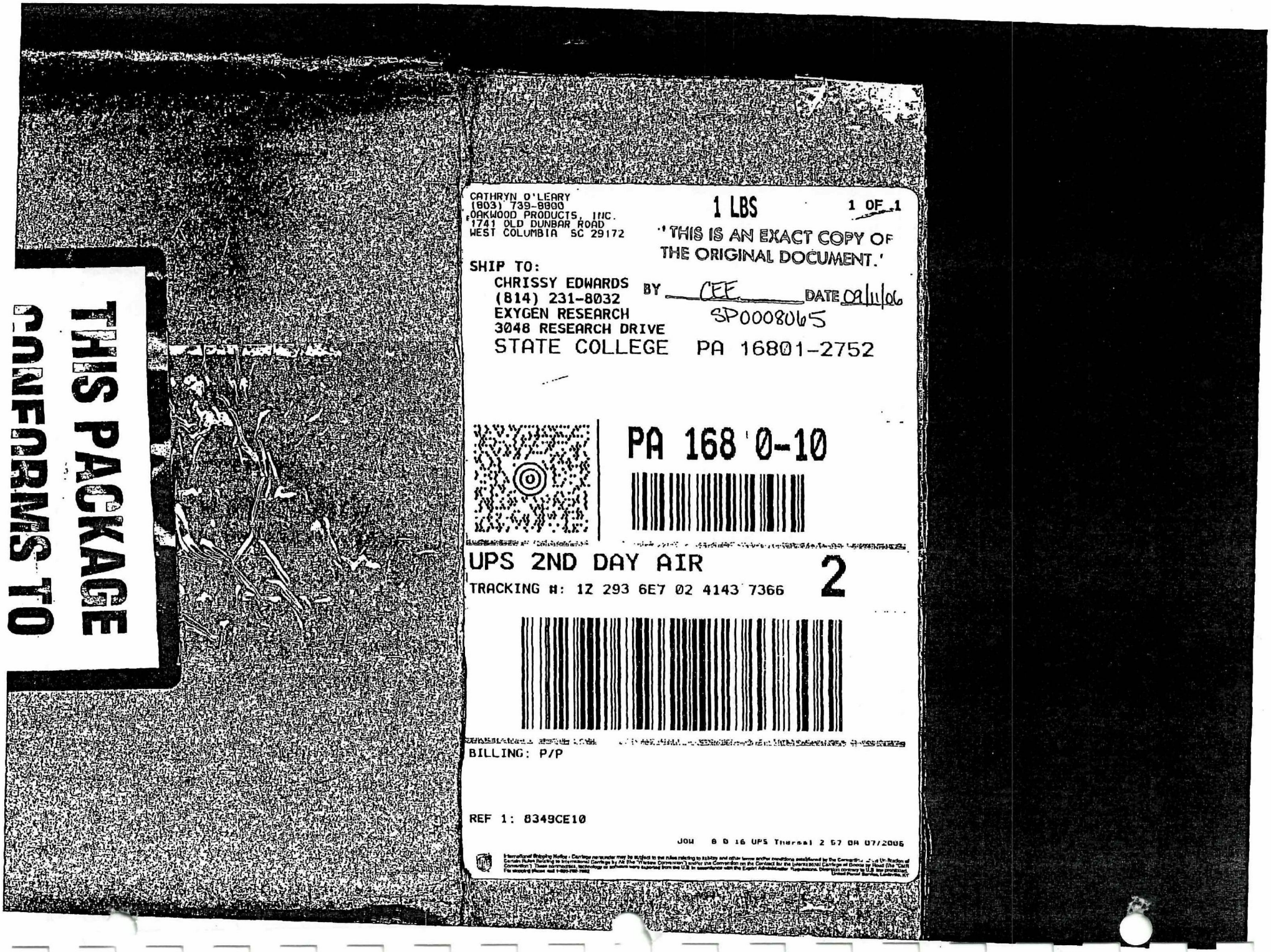
QUANTITY ORDERED	QUANTITY SHIPPED	QUAN. B.O.	ITEM NUMBER	DESCRIPTION
1	1		001269-25G	Heptafluorobutyric aci
1	1		003302-25G	n-Perfluoropentanoic a
24	24		008504	Perfluorohexanoic acid
1	1		002261-5G	Perfluoroheptanoic aci
1	1		001319-5G	Pentadecafluorooctanoi
1	1		002263-25G	Heptadecafluorononanoi
1	1		002264-5G	Nonadecafluorodecanoic
1	1		002265-5G	Perfluoroundecanoic ac
1	1		002266-5G	Perfluorododecanoic ac

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT."

BY CEE DATE 09/11/06

SP0008065

PACKING LIST


THIS PACKAGE CONFORMS TO
CATHRYN O'LEARY
(803) 739-8800
OAKWOOD PRODUCTS, INC.
1741 OLD DUNBAR ROAD
WEST COLUMBIA SC 29172
1 LBS
1 OF 1
'THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT.'
SHIP TO:
CHRISSY EDWARDS
(814) 231-8032
EXYGEN RESEARCH
3048 RESEARCH DRIVE
STATE COLLEGE PA 16801-2752
BY CEE DATE 02/11/06
SP0008065
PA 168 0-10
UPS 2ND DAY AIR
2
TRACKING #: 1Z 293 6E7 02 4143 7366
BILLING: P/P
REF 1: 8349CE10
JOH 8 D 16 UPS Thermal 2 07 08 07/2006

RECEIVED TIME SEP. 8. 9:51AM PRINT TIME SEP. 8. 9:56AM

Oakwood Products, Inc.
1741 Old Dunbar Road
West Columbia, SC 29172
Phone (803) 739-8800
Fax (803) 739-6957

CERTIFICATE OF ANALYSIS

Date: 18-May-05

Material: Pentadecafluorooctanoic acid

Cat.No.: 1319

Cas No.: [335-67-1]

Lot No.: Y16G

Assay: 98+% by NaOH Titration

We have stored this compound for years at room temperature without encountering problems with stability.

Appearance: White solid

Melting Point: 52-56°C

Yumei Yang
QC Manager

SP0008065

Exygen® RESEARCH

3058 Research Drive
State College, PA 16801

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

Secondary Standard

Standard Information

Standard: **SS0020744**
Category: Stock
Description: Pentadecafluorooctanoic Acid (PFOA) in Methanol (1000 ug/mL)

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/28/2007 2:44:18PM
Date Expires: ~~4/4~~ 4/23/2007 11:05:26AM (W.D) AMB 04/19/07
Storage Condition: Refrigerated

Preparation: Dissolve 0.10205 grams of PFOA in 100 mL of Methanol
ICOC: RECEIVED / B2-105 / IN0000709 / Refrigerator 33 / D0002259 / Refrigerator 33 / CC0001940 / Mar 28, 2007 2:46:43PM
LOGIN / No Location / Mar 28, 2007 2:44:17PM

Component Concentrations

Component	Concentration / Amount
PFOA (TRIAL)	1000.09 µg/mL
Reagent: RE0031862 / METHANOL HPLC / Date Expires: 03/19/2009	100 mL

Prepared From

Standard / Description	Aliquot	
SP0008065 / Pentadecafluorooctanoic Acid (C8 Acid - PFOA) / PFOA (TRIAL)	0.10205 g	98 %

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Fax: 814-231-1580

Secondary Standard

Standard Information

andard: **SS0020791**
tegory: Fortification
scription: Pentadecafluorooctanoic Acid (PFOA) in Methanol (100 ug/mL) ALT

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/29/2007 12:43:05PM
Date Expires: 6/29/2007 10:29:38AM
Storage Condition: Refrigerated

eparation: Dilute 10 mL of the 1000 ug/mL to a final volume of 100 mL in methanol.
OC: RECEIVED / B2-3rd Floor Lab / CC0001940 / Mar 29, 2007 12:44:11PM
LOGIN / No Location / Mar 29, 2007 12:43:05PM

Component Concentrations

Component	Concentration / Amount	Prepared From: Standard / Description	Aliquot	
PFOA (TRIAL)	100.009 µg/mL			
Reagent: RE0032031 / METHANOL HPLC / Date Expires: 03/26/2009	90 mL	SS0020744 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (1000 ug/mL) / PFOA (TRIAL)	10 mL	1000.09 µg/mL

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Fax: 814-231-1580

Secondary Standard

Standard Information

Standard: **SS0020792**
Category: Fortification
Description: Pentadecafluorooctanoic Acid (PFOA) in Methanol (1 ug/mL) ALT

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/29/2007 12:44:20PM
Date Expires: 6/29/2007 10:29:38AM
Storage Condition: Refrigerated

Preparation: Dilute 1 mL of the 100 ug/mL to a final volume of 100 mL in methanol.
ICOC: RECEIVED / B2-3rd Floor Lab / CC0001940 / Mar 29, 2007 12:45:12PM
LOGIN / No Location / Mar 29, 2007 12:44:20PM

Component Concentrations

Component	Concentration / Amount
PFOA (TRIAL)	1.00009 µg/mL
Reagent: RE0032031 / METHANOL HPLC / Date Expires: 03/26/2009	90 mL

Prepared From

Standard / Description	Aliquot	
SS0020791 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (100 ug/mL) ALT / PFOA (TRIAL)	1 mL	100.009 µg/mL

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State College, PA 16801

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

Secondary Standard

Standard Information

andard: **SS0020793**
tegory: Fortification
scription: Pentadecafluorooctanoic Acid (PFOA) in Methanol (0.1 ug/mL) ALT

Solvent: Methanol
Quantity: 100 mL
Date Prepared: 3/29/2007 12:45:22PM
Date Expires: 6/29/2007 10:29:38AM
Storage Condition: Refrigerated

eparation: Dilute 10 mL of the 1 ug/mL to a final volume of 100 mL in methanol.
OC: RECEIVED / B2-3rd Floor Lab / CC0001940 / Mar 29, 2007 12:46:19PM
LOGIN / No Location / Mar 29, 2007 12:45:21PM

Component Concentrations

Component	Concentration / Amount
PFOA (TRIAL)	0.100009 µg/mL
Reagent: RE0032031 / METHANOL HPLC / Date Expires: 03/26/2009	90 mL

Prepared From

Standard / Description	Aliquot	
SS0020792 / Pentadecafluorooctanoic Acid (PFOA) in Methanol (1 ug/mL) ALT / PFOA (TRIAL)	10 mL	1.00009 µg/mL

Login

10X 1000X

DELTEK #: 1395,0001

Samples

Sample ID	Container	Matrix	Fraction	Sample	Date Sample	
L0011423-0001	C0250357	LIQUID	Impinger solution	SG-A-001 Impinger DI	04/26/2007	
L0011423-0002	C0250358	LIQUID	Impinger solution	SG-A-002 NaOH	04/26/2007	
L0011423-0003	C0250359	LIQUID	Impinger solution	SG-A-003 Buffer	5/07/2007	
L0011423-0004	C0250360	LIQUID	Impinger solution	SG-A-004 MeOH/filter	5/07/2007	
L0011423-0005	C0250361	LIQUID	Impinger solution	SG-A-005 DI	5/07/2007	
L0011423-0006	C0250362	LIQUID	Impinger solution	SG-A-006 NaOH	04/26/2007	05/07/2007
L0011423-0007	C0250363	LIQUID	Impinger solution	SG-A-007 Buffer	04/26/2007	05/07/2007
L0011423-0008	C0250364	LIQUID	Impinger solution	SG-A-008 MeOH/filter	04/26/2007	05/07/2007
L0011423-0009	C0250365	LIQUID	Impinger solution	SG-A-009 DI	04/26/2007	05/07/2007
L0011423-0010	C0250366	LIQUID	Impinger solution	SG-A-010 NaOH	04/26/2007	05/07/2007
L0011423-0011	C0250367	LIQUID	Impinger solution	SG-A-011 Buffer	04/26/2007	05/07/2007
L0011423-0012	C0250368	LIQUID	Impinger solution	SG-A-012 MeOH/filter	04/26/2007	05/07/2007
L0011423-0013	C0250369	LIQUID	Impinger solution	SG-A-013 DI	04/26/2007	05/07/2007
L0011423-0014	C0250370	LIQUID	Impinger solution	SG-A-014 NaOH	04/26/2007	05/07/2007
L0011423-0015	C0250371	LIQUID	Impinger solution	SG-A-015 Buffer	04/26/2007	05/07/2007
L0011423-0016	C0250372	LIQUID	Impinger solution	SG-A-016 MeOH/filter	04/26/2007	05/07/2007

★ Sonicate Sample Containers before you start Extractions!

Login

L0011423-0017	C0250373	LIQUID	Impinger solution	SG-A-017 DI	04/26/2007	05/07/2007
L0011423-0018	C0250374	LIQUID	Impinger solution	SG-A-018 NaOH	04/26/2007	05/07/2007
L0011423-0019	C0250375	LIQUID	Impinger solution	SG-A-019 Buffer	04/26/2007	05/07/2007
L0011423-0020	C0250376	LIQUID	Impinger solution	SG-A-020 MeOH/filter	04/26/2007	05/07/2007
L0011423-0021	C0250377	LIQUID	Dispersion	SG-L-001	04/26/2007	05/07/2007
L0011423-0022	C0250378	LIQUID	Dispersion	SG-L-002	04/26/2007	05/07/2007

Login Reviewed By: _____ Date/Time: _____

Solvay

SS0019495

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

Login

Login Group: L0011423

Login #:	11534	Conform COC Sample:	True
Project:	P0003133	Conform COC:	True
Company Name:	Barr Engineering	Conform Sample:	True
Submitted By:	Tim Russell	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	04/27/2007		
Due Date:	05/07/2007		
Login Initiated*:	04/27/2007		

* Dates entered into "Login Initiated" field prior to 1/5/06 reflect dates of receipt. The field was formerly called "Received Date"

Received By: Ammerman, Mark
Spread Sample:
Label:
Exygen SD/PI: Risha, Karen
Project Title/Type: Analysis of Samples for PFOA/APFO by LCMSMS / ROUTINE
Login Notes:

Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By
PK0013603		Received Date: 4/27/07 9:00 Package & Contents Uncompromised		Wet Ice 2.0	RECEIVED Ammerman, Mark

Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0250357	139.30 g		500 ml Clear Plastic Narrow	NONE		
C0250358	151.60 g		500 ml Clear Plastic Narrow	NONE		
C0250359	158.10 g		500 ml Clear Plastic Narrow	NONE		
C0250360	121.40 g		500 ml Clear Plastic Narrow	NONE		
C0250361	155.20 g		500 ml Clear Plastic Narrow	NONE		
C0250362	156.10 g		500 ml Clear Plastic Narrow	NONE		
C0250363	156.60 g		500 ml Clear Plastic Narrow	NONE		
C0250364	129.50 g		500 ml Clear Plastic Narrow	NONE		
C0250365	158.00 g		500 ml Clear Plastic Narrow	NONE		
C0250366	158.40 g		500 ml Clear Plastic Narrow	NONE		
C0250367	156.70 g		500 ml Clear Plastic Narrow	NONE		
C0250368	305.40 g		500 ml Clear Plastic Narrow	NONE		
C0250369	154.80 g		500 ml Clear Plastic Narrow	NONE		

Login

Login Group: L0011423

Login #:	11534	Conform COC Sample:	True
Project:	P0003133	Conform COC:	True
Company Name:	Barr Engineering	Conform Sample:	True
Submitted By:	Tim Russell	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	04/27/2007		
Due Date:	05/07/2007		
Login Initiated*:	04/27/2007		

* Dates entered into "Login Initiated" field prior to 1/5/06 reflect dates of receipt. The field was formerly called "Received Date"

Received By: Ammerman, Mark
Spread Sample:
Label:
Exygen SD/PI: Risha, Karen
Project Title/Type: Analysis of Samples for PFOA/APFO by LCMSMS / ROUTINE
Login Notes:

Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By
PK0013603		Received Date: 4/27/07 9:00 Package & Contents Uncompromised		Wet Ice 2.0	RECEIVED Ammerman, Mark

Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0250370	160.40 g		500 ml Clear Plastic Narrow	NONE		
C0250371	158.10 g		500 ml Clear Plastic Narrow	NONE		
C0250372	341.10 g		500 ml Clear Plastic Narrow	NONE		
C0250373	156.40 g		500 ml Clear Plastic Narrow	NONE		
C0250374	160.00 g		500 ml Clear Plastic Narrow	NONE		
C0250375	155.90 g		500 ml Clear Plastic Narrow	NONE		
C0250376	368.30 g		500 ml Clear Plastic Narrow	NONE		
C0250377	380.10 g		500 ml Clear Plastic Narrow	NONE		
C0250378	387.30 g		500 ml Clear Plastic Narrow	NONE		

Appendix D

Calibration Data

BARR

EPA M5 POST TEST METER CALIBRATION

Project Name: Saint Gobain
Module Number: C-5
Date: 3-May-07
Tech: NRS

Nominal sample conditions
Delta H: 1.100 W.C.
Vacuum: 2.0 in. Hg

Project No.: 29/06-004-001
Barometric Pressure: 29.26
Previous Y : 1.0010
Previous Delta H : 1.8381

Orifice Diff Presure dH	Wet Test Volume, Cubic Feet	Dry Gas Meter Temp, F Inlet	Dry Gas Meter Temp, F Outlet	Wet Test Meter Temp, F	Dry Gas Volume, Cubic Feet	Elapsed Time of Cal. Point		Meter Coefficient Y	Orifice Coefficient dH@
Nominal 1.100	Initial 1920.00	Initial 75	Initial 75	Initial 73.5	Initial 199.870				
Actual 1.10	Final 1931.00	Final 75	Final 75	Final 73.5	Final 210.870	Minutes 19.0	SEC 44.65		
	Total 11.00	Average 75.0	Average 75.0	Average 73.5	Total 11.000	Minutes 19.74		1.0000	2.0426
		75.0 Tm							
Nominal 1.100	Initial 1933.00	Initial 77	Initial 76	Initial 73.5	Initial 212.875				
Actual 1.10	Final 1938.00	Final 77	Final 76	Final 73.5	Final 217.885	Minutes 9.0	SEC 0.56		
0	Total 5.00	Average 77.0	Average 76.0	Average 73.5	Total 5.010	Minutes 9.01		1.0008	2.0546
		76.5 Tm							
Nominal 1.100	Initial 1948.00	Initial 78	Initial 76	Initial 73.5	Initial 227.915				
Actual 1.10	Final 1953.00	Final 79	Final 76	Final 73.5	Final 232.930	Minutes 8.0	SEC 59.36		
	Total 5.00	Average 78.5	Average 76.0	Average 73.5	Total 5.015	Minutes 8.99		1.0012	2.0455
		77.3 Tm							
						Meter Average		1.0007	2.0476

Pre cal + 5%	1.0511
Pre cal - 5%	0.9510
Within 5% Y/N	YES

BARR

THERMOCOUPLE CALIBRATION

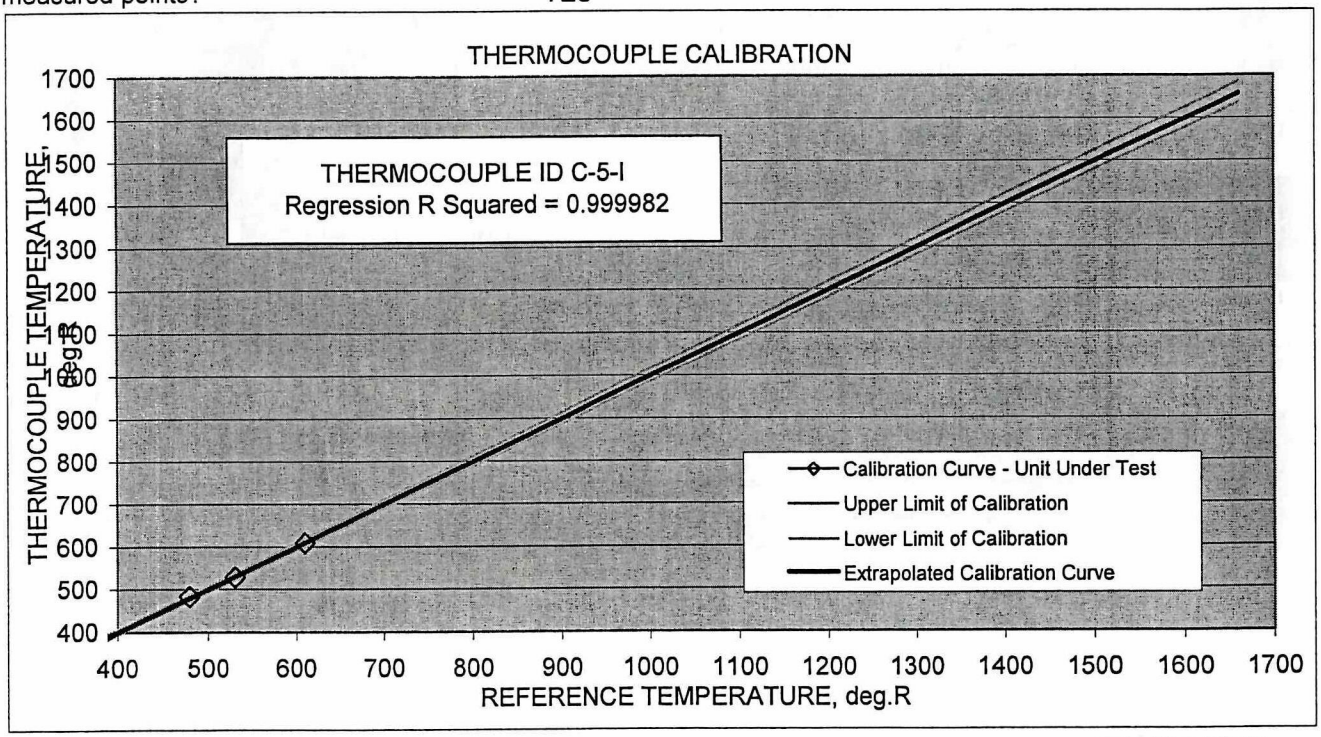
THERMOCOUPLE ID **C-5-I**

Cal Date: **1/15/2007** **Meter In or Out**

CALIBRATION TECHNICIAN: **TYL**

REFERENCE STANDARDS	TRACEABILITY	DATE	LABORATORY
Hart Scientific 9103-A s/n A1B289	Report No. A1B0718	12/15/2005	Hart Scientific
Hart Scientific 9140 s/n A1B086	Report No. A1B2024	12/15/2005	Hart Scientific

Temperature Calibration Points	20	70	150
Reference Deg F (To)	20	70	150
Probe Temp (deg F)	23	68	149
Reference Temp (deg R) deg F + 460	480	530	610
Probe Temp (deg R), deg F + 460	483	528	609
Difference (degrees)	-3	2	1
% Diff Abs. T	0.6%	0.4%	0.2%
Is difference less than 1.5% at all measured points?	YES		



Are extrapolated limits less than 1.5%? YES

FAHRENHEIT CALIBRATION RANGE
-20 1200

If not acceptable, describe corrective action:

Tchnician signature

QA signature

BARR

THERMOCOUPLE CALIBRATION

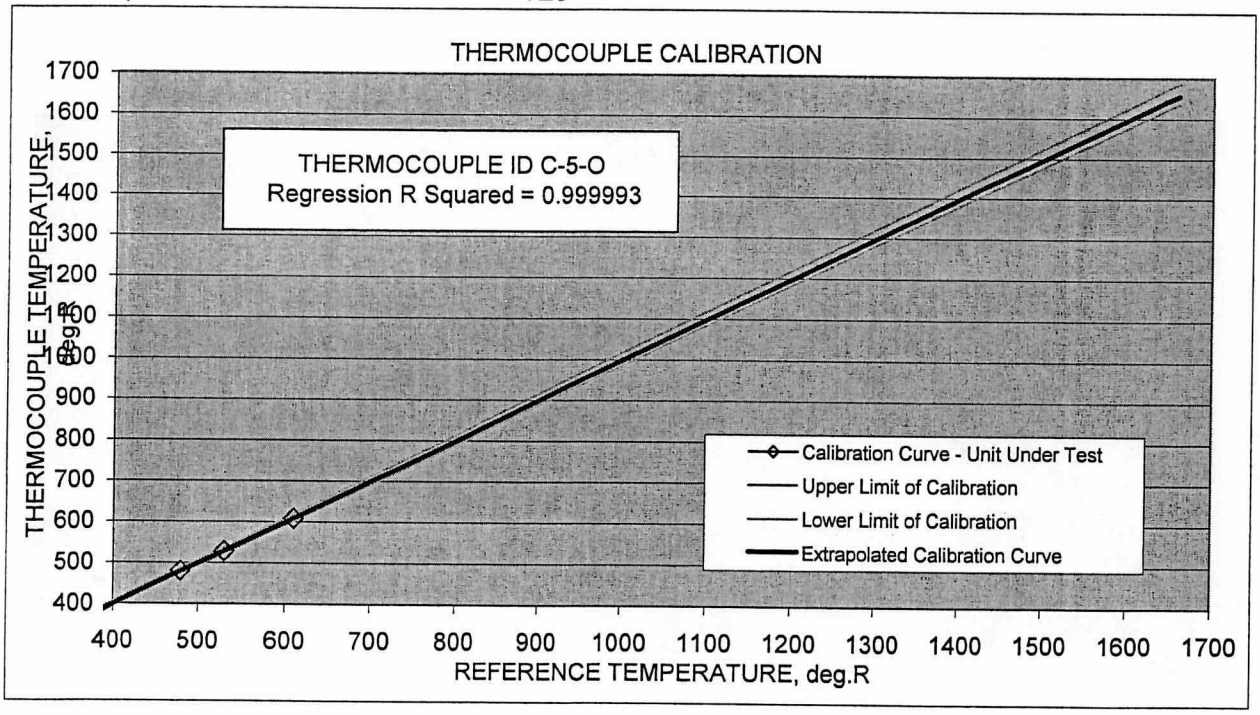
THERMOCOUPLE ID C-5-O

Cal Date: 1/15/2007 Meter In or Out

CALIBRATION TECHNICIAN: TYL

REFERENCE STANDARDS	TRACEABILITY	DATE	LABORATORY
Hart Scientific 9103-A s/n A1B289	Report No. A1B0718	12/15/2005	Hart Scientific
Hart Scientific 9140 s/n A1B086	Report No. A1B2024	12/15/2005	Hart Scientific

Temperature Calibration Points	20	70	150
Reference Deg F (To)	20	70	150
Probe Temp (deg F)	20	68	148
Reference Temp (deg R) deg F + 460	480	530	610
Probe Temp (deg R), deg F + 460	480	528	608
Difference (degrees)	0	2	2
% Diff Abs. T	0.0%	0.4%	0.3%
Is difference less than 1.5% at all measured points?	YES		



Are extrapolated limits less than 1.5%? YES

FAHRENHEIT CALIBRATION RANGE
-20 1200

If not acceptable, describe corrective action:

Tchnician signature

QA signature

THERMOCOUPLE CALIBRATION

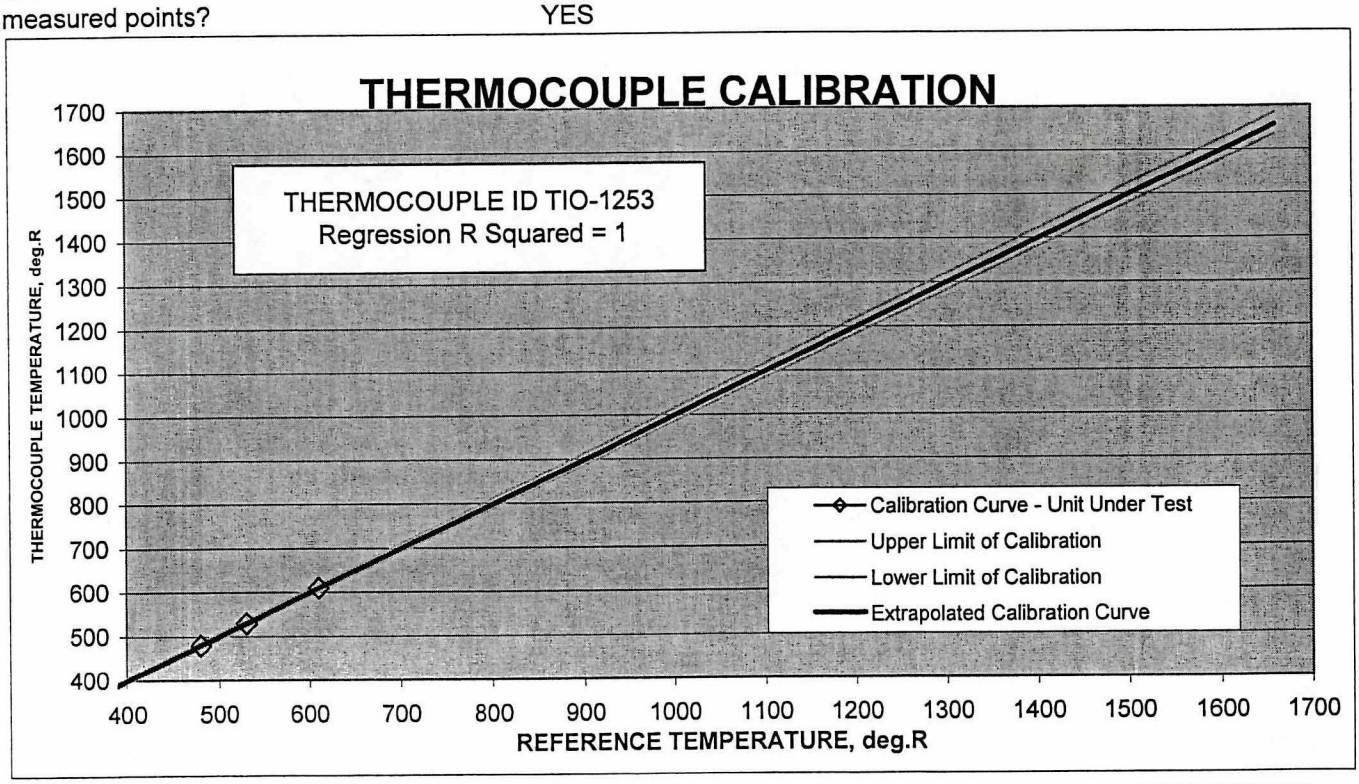
THERMOCOUPLE ID TIO-1253

Cal Date: 1/20/2007 **Impinger Outlet**

CALIBRATION TECHNICIAN: NRS

REFERENCE STANDARDS	TRACEABILITY	DATE	LABORATORY
Hart Scientific 9103-A s/n A1B289	Report No. A1B0718	12/15/2005	Hart Scientific
Hart Scientific 9140 s/n A1B086	Report No. A1B2024	12/15/2005	Hart Scientific

Temperature Calibration Points	20	70	150
Reference Deg F (To)	20	70	150
Probe Temp (deg F)	19	69	149
Reference Temp (deg R) deg F + 460	480	530	610
Probe Temp (deg R), deg F + 460	479	529	609
Difference (degrees)	1	1	1
% Diff Abs. T	0.2%	0.2%	0.2%
Is difference less than 1.5% at all measured points?	YES		



Are extrapolated limits less than 1.5%? YES

FAHRENHEIT CALIBRATION RANGE

-20 1200

If not acceptable, describe corrective action:

Tchnician signature

QA signature

BARR

UMBILICAL CALIBRATION

Umbilical ID: UM-300-1 Date: 1/20/2007

Pyrometer Reference: CL300-K-1000F Technician: NRS

TC 1 - Stack Temp

Reference (°F)	Pyrometer ° F Reading	Pass/Fail
1000	1002	Pass
900	901	Pass
800	801	Pass
700	702	Pass
600	601	Pass
500	499	Pass
400	399	Pass
300	300	Pass
200	200	Pass
150	151	Pass
100	100	Pass
50	49	Pass
0	-1	Pass
-50	-58	Fail

TC 2 n- Probe Temp

Reference (°F)	Pyrometer ° F Reading	Pass/Fail
500	501	Pass
400	400	Pass
300	300	Pass
200	200	Pass
150	151	Pass
100	99	Pass
50	49	Pass

TC 3 Oven/Filter Temp

Reference (°F)	Pyrometer ° F Reading	Pass/Fail
500	499	Pass
400	399	Pass
300	300	Pass
200	200	Pass
150	151	Pass
100	100	Pass
50	50	Pass

TC 4 - TIO-1253

Reference (°F)	Pyrometer ° F Reading	Pass/Fail
150	151	Pass
100	100	Pass
50	50	Pass
0	-1	Pass
-50	-57	Fail

TC 5 - Auxillary

Reference (°F)	Pyrometer ° F Reading	Pass/Fail
1000	1000	Pass
900	900	Pass
800	800	Pass
700	701	Pass
600	601	Pass
500	499	Pass
400	399	Pass
300	300	Pass
200	200	Pass
150	151	Pass
100	100	Pass
50	50	Pass
0	-1	Pass
-50	-57	Fail

Pyrometer used: D-8

Pitot Line Leak Check

Side	Press. In H2O	Vac. In. H2O	Leak(Y/N)
Positive	6.0		N
Negative		5.0	N

	(Y/N)
Are all thermocouple leads functioning properly?	Y
Are pitot lines blocked?	N

Pass/Fail based on +/- 0.75% of Renkin value

Technician signiture:

QA signiture:

BARR

S-Type Pitot Tube
Geometry Check

Pitot Tube Number: 12

Length: 4'

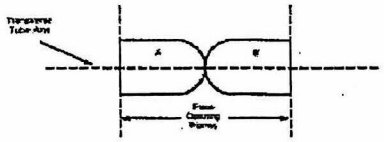
Function: M-5 Probe / Free

Inspection Date: 1-24-07

Technician: TYL

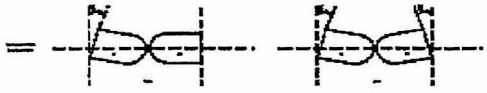
1. Are face openings perpendicular to tube axis?

☒ YES (go to 2) ☐ NO (go to 1a)



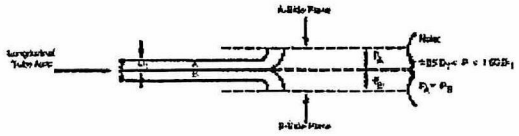
1a. If NO, is angle less than 10°?

☐ YES (go to 2) ☐ NO (discontinue use)



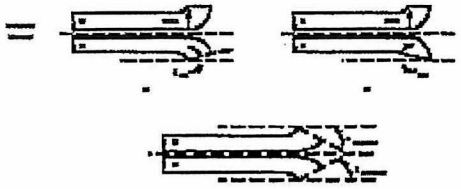
2. Are face openings parallel to longitudinal axis?

☐ YES (go to 3) ☒ NO (go to 2a)



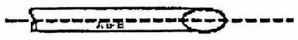
2a. If NO, is angle less than 5°?

☒ YES (go to 3) ☐ NO (discontinue use)



3. Are legs of equal length?

☐ YES (go to 4) ☒ NO (go to 3a)

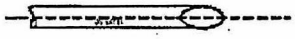


3a. If NO, is difference less than 1/8 inch?

☒ YES (go to 4) ☐ NO (discontinue use)

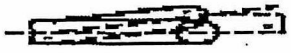
4. Are center-lines of legs coincident?

☒ YES (go to 5) ☐ NO (go to 4a)



4a. If NO, are center-lines of face openings less than 1/32 inch?

☐ YES (go to 5) ☐ NO (discontinue use)



5. Does this pitot tube pass all of the above criteria? ☒ YES ☐ NO

Technician Signature: [signature]

Reviewed by: [signature]

BARR

S-Type Pitot Tube
Geometry Check

Pitot Tube Number: 7-4

Length: 7'

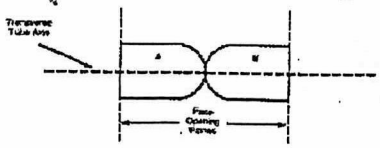
Function: ~~M-5 Probe~~ / Free

Inspection Date: 1-24-07

Technician: TYL

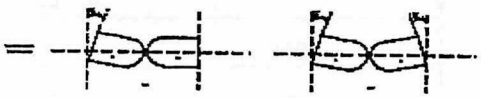
1. Are face openings perpendicular to tube axis?

☑ YES (go to 2) ☐ NO (go to 1a)



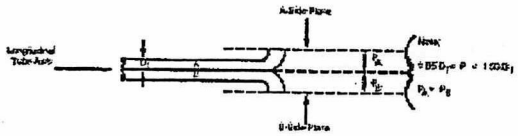
1a. If NO, is angle less than 10°?

☐ YES (go to 2) ☐ NO (discontinue use)



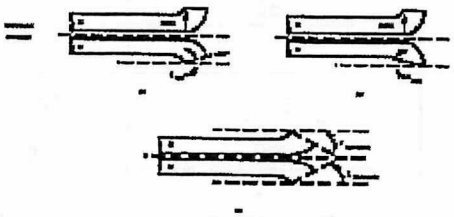
2. Are face openings parallel to longitudinal axis?

☐ YES (go to 3) ☒ NO (go to 2a)



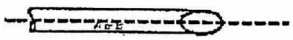
2a. If NO, is angle less than 5°?

☒ YES (go to 3) ☐ NO (discontinue use)



3. Are legs of equal length?

☒ YES (go to 4) ☐ NO (go to 3a)

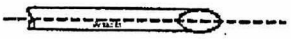


3a. If NO, is difference less than 1/8 inch?

☐ YES (go to 4) ☐ NO (discontinue use)

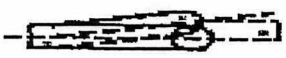
4. Are center-lines of legs coincident?

☒ YES (go to 5) ☐ NO (go to 4a)



4a. If NO, are center-lines of face openings less than 1/32 inch?

☐ YES (go to 5) ☐ NO (discontinue use)



5. Does this pitot tube pass all of the above criteria?

☒ YES ☐ NO

Technician Signature: Tom Lien

Reviewed by: [signature]

BARR

PYROMETER CALIBRATION

Pyrometer Number: C-5 Date: 1/15/2007

Pyrometer Reference: CL-300-1000F Technician: TYL

Reference (°F)	Reference (°C)	Pyrometer ° F	
		Reading	Pass/Fail
1000	538	1000	Pass
950	510	950	Pass
900	482	899	Pass
850	454	850	Pass
800	427	800	Pass
750	399	751	Pass
700	371	700	Pass
650	343	651	Pass
600	316	599	Pass
550	288	548	Pass
500	260	497	Pass
450	232	447	Pass
400	204	398	Pass
350	177	349	Pass
300	149	300	Pass
250	121	251	Pass
200	93	200	Pass
150	67	151	Pass
100	38	98	Pass
50	10	50	Pass
0	-18	1	Pass
-50	-46	-48	Pass

Pass/Fail based on +/- 0.75% of Renkin value

Technician signiture:

QA signiture: Richard Beer

BARR

Manometer Calibration Sheet

Manometer Number	Alnor M-8	Leak Check:	
Date of Calibration	1/9/2007	Negative	0.0 @3"
Technician	CCL	Positive	0.0 @3"

	Oil Manometer	Digital Manometer	Pass/Fail
Positive	0.06	0.06	Pass
	0.2	0.20	Pass
	0.5	0.49	Pass
	0.8	0.79	Pass
	1	0.99	Pass
	1.5	1.50	Pass
	2	2.00	Pass
	2.5	2.50	Pass
	3	3.00	Pass
	3.5	3.50	Pass
Negative	0.06	0.06	Pass
	0.2	0.20	Pass
	0.5	0.49	Pass
	0.8	0.78	Pass
	1	0.97	Pass
	1.5	1.50	Pass
	2	1.97	Pass
	2.5	2.49	Pass
	3	2.99	Pass
	3.5	3.50	Pass

Pass/Fail based on +/- 5% of set value

Technician signiture: Christopher C. Lee

QA signiture:

Appendix E

Stack Test Plan

TEST PLAN FOR POLYMER CLOTH COATING FACILITY

SAINT GOBAIN PERFORMANCE PLASTICS CORPORATION
MERRIMACK, NEW HAMPSHIRE

Date test plan created/revised: March 23, 2007

Scheduled test date(s): April 25, 2007

PART I. GENERAL INFORMATION

Name and address of emission facility: Saint Gobain Performance Plastics Corporation
701 Daniel Webster Highway
Merrimack, NH 03054

Facility contact: Bruce Briand (603) 424-9000, x2341
Health, Safety & Environmental Manager
Fax (603) 424-9012

Air Emission Facility ID Number: 3301100165

Reason for the test: Determine ammonium perfluorooctanoate (APFO) stack emission rate for air dispersion modeling input.

Testing Company and Contact: Barr Engineering Company (952) 832-2630
Tim Russell Fax (952) 832-2601
Vice President, Chemical Engineer trussell@barr.com
4700 W. 77th Street
Minneapolis, MN 55435

PART II. TESTING REQUIREMENTS

The table below provides a summary of the pollutants to be tested and test methods.

Source Description	Limitation Basis of Pollutant Tested	Pollutant Tested and Applicable Emission Limit	Specific Methods/Procedures Citation
MA Coating Tower Exhaust	none	Ammonium Perfluorooctanoate (APFO)	EPA Methods 1-4, Modified EPA Method 5 40 CFR Part 60, Appendix A

PART III. OPERATING CONDITIONS

The table and paragraph below identifies the process equipment to be tested, the operating parameters to be monitored and reported, and the rationale for testing.

Process Equipment Description for Units to be Tested	MA Coating Tower
Process Equipment Parameter Monitoring During Performance Test	Dispersion identification, dispersion application rate, web width, line speed, coating pass number. Information will be recorded during the test and provided separate from the test report under confidential cover.
Control Equipment Description	The source is uncontrolled
Control Equipment Operating Parameter During Performance Test	Not applicable

The production coating tower chosen for sampling is Tower MA, an intermediate width tower that has an above average run time percentage, compared to all towers in Merrimack. The products that are processed on this line are representative of those that are run on all coating towers in Merrimack. This line has been tested on two prior occasions and we feel would be the most relevant in terms of comparing current, APFO light emission rates to the products formerly processed. The same product families would be selected for the upcoming test as were tested previously.

PART IV. TEST AND ANALYTICAL METHODS

The following is a description of the test methods, number of test runs, length of test runs, and sampling rate for each pollutant

A. EPA Method 1 for the location of sampling ports and points. One determination per test location.

B. EPA Method 2 for stack gas velocity and volumetric flow rate. One determination per EPA Method 5 test run.

C. EPA Method 3 to determine stack gas molecular weight. One determination per EPA Method 5 test run.

D. EPA Method 4 for determination of moisture content in stack gas. One determination per EPA Method 5 test run.

E. EPA Method 5 Modified to collect APFO. The sample train will consist of three impingers followed by an unheated glass fiber filter. The three impingers will be charged in order with 100

ml each of de-ionized water, 0.1*N* sodium hydroxide, and 0.01*N* sodium borate buffer. The filter will be collected and placed in a sample bottle with a methanol rinse of all components of the sample train. Each of the four sample fractions will be recovered and analyzed separately for the determination of total APFO mass. The modification of Method 5 follows test protocols previously accepted by the USEPA.

F. Sampling times will be three runs of 60 minute duration.

G. Exygen Research of State College, PA will perform the laboratory analysis of the air impinger and dispersion coating samples using high pressure liquid chromatography and tandem mass spectrometry (LC/MS/MS) by analytical methods they have developed for the measurement of APFO.

PART V. CALIBRATION METHODS AND PRETEST PREPARATIONS

A. The EPA Method 5 Equipment will be calibrated prior to the test in accordance with the requirements in 40 CFR Part 60, Appendix A, EPA Quality Assurance Handbook for Air Pollutant Measurement Systems: Volume III Stationary Sources Specific Methods and Barr Standard Operating Procedures. Calibration data will be available onsite for review. Samples of calibration and field sample data sheets are provided as an attachment to this document.

B. Pretest preparations include cleaning and methanol rinse of all sample train glassware components and the preparation of sampling reagents in addition to that standard to the mobilization of particulate measurement and support equipment. Special care is made to eliminate all components of the sample train that may contain fluoropolymer constituents in order to avoid trace contamination of the samples from those components. Examples include avoiding the use of Teflon™ compression fittings or ferrules, sealing tape or sample jar liners, and fluoropolymer encapsulated o-ring seals.

PART VI. QUALITY ASSURANCE AND LABORATORY STANDARD OPERATING PROCEDURES

A. MPI Research, State College Facility, (previously Exygen Research) has developed standard operating procedures for the laboratory analysis of APFO in air impinger and liquid samples using analytical method number ExM-079-007C entitled "Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Air Process (Stack) Samples by LC/MS/MS for SPI Processors Mass Balance Study." Dispersion samples

will be analyzed using analytical method number ExM-079-007A entitled "Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Water by LC/MS/MS for SPI Processors Mass Balance Study." The SOPs are considered to be proprietary business information by Exygen and can be shared upon execution of a confidentiality agreement.

B. A complete review of the analytical laboratory recoveries, duplicates, and raw data by a member of Barr's Data Quality Assurance staff will be performed and presented in the test report.

PART VII. SAFETY

Emergency procedures are outlined in Saint Gobain's "Call to Action Safety Awareness Program" which can be reviewed upon arrival at the facility. Facility emergencies notifications are made to the facility main front desk at 603-424-900 in the event that medical, fire or other emergency response is needed.

PART VIII. TEST SCHEDULE

Week of April 23-27, 2007

April 23 Monday	April 24 Tuesday	April 25 Wednesday	April 26 Thursday	April 27 Friday
		Safety Meeting Pretest Coordination Meeting Equipment Mobilization	Perform Test (0700-1400) Recover Samples (1400-1500) Demobilize (1500-1700)	Contingency Test Day

PART VII. TEST REPORT

One complete test report (bound copy) will be submitted within 60 days of the test performance date.

Submittal Address: Mr. Jim Black
New Hampshire Department of Environmental Services
29 Hazen Drive
P.O. Box 95
Concord, NH 03302-0095

Appendix F

Project Participants

Project Participants

Saint Gobain Performance Plastics Corporation

Edward J. Canning – Manager, Health, Safety and Environment - Corporate

Bruce Briand - Manager, Health, Safety and Environment - Specialty Coated Fabrics

Dan Methot – Plant/Project Engineer

Ruth A. (Sam) Jamke – Research Associate

Ray Pieczarka – Facility Manager

Kirk Johnston – Production Manager

Keith Margenau – Maintenance Supervisor

Barr Engineering Company

Tim Russell – Vice President/Chemical Engineer

Richard Berg – Project Manager/Senior Air Quality Technician

Chris Lee – Stack Testing Technician

Ward Swanson – Senior Data Quality Specialist

New Hampshire Department of Environmental Services

Jack C. Glenn – Air Pollution Engineer