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Analytical Report

Fluorochemical Characterization of Water Samples

DuPont Washington Works

Exygen Research Laboratory Report No. 008-046B (Revision 1)

Testing Laboratory

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Requester

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1 Introduction

Results are reported for the analysis of water samples received by Exygen Research (Centre Analytical Laboratories, Inc.) (Exygen) from DuPont Corporation. The Exygen study number assigned to the project is 008-046.

Specific fluorochemical characterization by liquid chromatography / tandem mass spectrometry (LC/MS/MS) was requested for all samples. A total of 11 samples were received for analysis.

The samples were prepared and analyzed by LC/MS/MS for the following list of fluorochemicals:

- Table 1: Target Analysis

Compound Name	Acronym
Ammonium Perfluorooctanoate	APFO

2 Sample Receipt

The samples were submitted in individual plastic containers, cooled with wet ice. Samples were collected on 10/11/01. Samples were received on 10/12/01. Chain-of-custody information is presented in Attachment C.

3 Holding Times

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

4 Methods - Analytical and Preparatory

4.1 LC/MS/MS

4.1.1 Sample Preparation for LC/MS/MS Analysis

Water samples were initially treated with 200 uL of 250 mg/L sodium thiosulfate solution to remove residual chlorine. Solid phase extraction (SPE) was used to prepare the samples for LC/MS/MS analysis. A forty-milliliter portion of sample was transferred to a C_{18} SPE cartridge. The cartridge was eluted with 40% methanol: water. The eluate was discarded. The cartridge was then eluted with 100% methanol. A 5 mL portion of methanol was collected for analysis by LC/MS/MS. This treatment resulted in an eight-fold concentration of the samples prior to analysis.

4.1.2 Sample Analysis by LC/MS/MS

In 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, ES/MS provides a rapid and accurate means for analyzing a wide range of organic compounds, including fluorochemicals. Electrospray is generally operated at relatively mild temperatures; molecules are ionized, fragmented, and detected. Ions characteristic of known fluorochemicals are observed and quantitated against standards.

A Hewlett-Packard HP1100 HPLC system coupled to a Micromass Ultima MS/MS was used to analyze the sample extracts. Analysis was performed using selected reaction monitoring (SRM). Samples were extracted on 10/17/01. Samples were analyzed by MS/MS between 10/17/01 and 10/19/01. Raw analytical data can be found in Attachment D.

5 Analysis

5.1 Calibration

A 6-point calibration curve was analyzed at the beginning of the analytical sequence and throughout the run for the compounds of interest. The calibration points were prepared at 0, 50, 100, 250, 500, and 1000 ng/L (ppt). The instrument response versus the concentration was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and correlation coefficient (r) and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r \geq 0.992$ ($r^2 \geq 0.985$).

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

For the results reported here, calibration criteria were met.

5.2 Blanks

Extraction blanks were prepared and analyzed with every extraction batch of samples. The extraction blanks should not have any target analytes present at or above the concentration of the low-level calibration standard. For these samples, the extraction blanks were compliant.

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

5.3 Surrogates

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

5.4 Matrix Spikes

Matrix spikes were prepared for every field sample at concentrations of 500 ng/L. Matrix spike recoveries are given in Attachment B. All samples showed matrix spike recoveries between 70 and 130%

5.5 Duplicates

All field samples were analyzed in duplicate. Results are given along with the sample results in Attachment A.

5.6 Laboratory Control Samples

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

5.7 Sample Related Comments

There are no other sample related comments associated with this sample set.

6 Data Summary

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

7 Data/Sample Retention

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

8 Attachments

8.1 Attachment A: Results

8.2 Attachment B: Matrix Spike Recoveries

8.3 Attachment C: Chain of Custody

8.4 Attachment D: LC/MS/MS Raw Analytical Data

8.5 Attachment E: Standards Data

9. Signatures

John M. Flaherty, Operations Manager — Date: 11/15/01

Kevin J Lloyd, Vice President — Date: 11/15/01

Other Lab Members Contributing to Data

Karen Smith

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Exygen Research Laboratory Report 008-046B

Revision 1

This revision was performed to report results as ammonium perfluorooctanoate (APFO) to conform to historical reporting practices. Additional quality control data was also added at the request of DuPont.

John M Flaherty — 1/15/01

John M. Flaherty — Date

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A

RJZ032551

EID185025

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Analytical Report

Analytical Results DuPont Washington Works

From Bob Ritchey
Lancaster Laboratory 8/01
ng/L

DuPont Sample Identification	APFO (ng/L)		4/01	2/01
WWK-D-Bldg-1-Main	507			
WWK-D-Bldg-1-Main (laboratory duplicate)	463			
WWK-G-Bldg-1-Main-2	471			
WWK-G-Bldg-1-Main-2 (laboratory duplicate)	463			
WWK-G-Well-332	498			
WWK-G-Well-332 (laboratory duplicate)	469			
WWK-D-Bldg-231	450			
WWK-D-Bldg-231 (laboratory duplicate)	459			
WWK-D-Bldg-293	423			
WWK-D-Bldg-293 (laboratory duplicate)	390			
WWK-D-Lubeck-Office	621	224	143	497
WWK-D-Lubeck-Office (laboratory duplicate)	605	55	65	212
WWK-G-LPWD-Well-F	288			
WWK-G-LPWD-Well-F (laboratory duplicate)	264	270	220	648
WWK-G-LPWD-Well-A	703			
WWK-G-LPWD-Well-A (laboratory duplicate)	684	236	55	638
WWK-G-LPWD-Well-B	581			
WWK-G-LPWD-Well-B (laboratory duplicate)	568			
WWK-K-FBLK-1	ND			
WWK-K-FBLK-2	ND			

Limit of Detection (LOD) for the procedure is appoximately 7.5 ng/L

Limit of Quantitation (LOQ) for the procedure is 50 ng/L

ND - Compound not detected

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

ND < LOD < NQ < LOQ

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B

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EID185027

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-D-Bldg-1-Main

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	487	844	71.4	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-G-Bldg-1-Main-2

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	453	901	89.6	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-G-Well-332

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	479	909	86.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-D-Bldg-231

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	432	967	107.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-D-Bldg-293

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	406	926	104.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-D-Lubeck Office

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	597	1080	96.6	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-G-LPWD-Well F

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	277	715	87.6	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-G-LPWD-Well A

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	675	1105	86.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: WWK-G-LPWD-Well B

Spiked Amount (ng/L): 500

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOA	558	979	84.2	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Concentrations represent amounts present as the perfluorooctanoate anion.

C

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Exygen Research

Sample Login Report

Study Number: 008-046
Protocol: NA

Exygen ID	Client Sample ID	Received	Matrix	Submitted By	Logged In By/Date	Initial Loc.	Condition of Samples	Comment
0112719	wwk-D-Bldg-1-Main	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112720	WWK-G-Bldg-1-Main-2	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112721	WWK-G-Well-332	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112722	WWK-D-Bldg-231	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112723	WWK-K-FBLK-1	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112724	WWK-K-FBLK-2	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112725	WWK-D-Bldg-293	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112726	WWK-D-Lubeck-Office	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112727	WWK-G-LPWD-Well-F	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112728	WWK-G-LPWD-Well-A	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—
0112729	WWK-G-LPWD-Well-B	10-12-01 10:30	WATER	John Flaherty	LAWRENCE O 10-12-01	COOLER 6	C-WET ICE-A	—

Verified By/Date: 10/12/01

Printed 10/12/2001

Centre Analytical Laboratories, Inc.

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Chain of Custody/Analysis Request Form

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

(1) Client: DuPont Washington Works
Route 892 South, DuPont Road
Washington WV 26181
PHONE # (304) 863-4476
FAX #

Sampler: Steve Opp

Project Manager: Andrew Hartten
Project Name:
P.O.#
CAL Quotation #:

(2) (3) (4) (5)

Sample Identification	Collection Date	Collection Time	Grab	Composite	Soil	Water (UST ACT II NPDES POTABLE RCRA SDWA CWA)	Other	Number of Containers	FC-143 (C-6)	Comments:
WWK-D-Bldg-1-Main	10/11/01	1045	X			X		1	X	
WWK-G-Bldg-1-Main-2		1045	X			X		1	X	
WWK-G-Well-332		1000	X			X		1	X	
WWK-D-Bldg-231		1015	X			X		1	X	
WWK-K-FBLK-1		1155	X			X		1	X	
WWK-K-FBLK-2		1132	X			X		1	X	
WWK-D-BLDG-293		625	X			X		1	X	
WWK-D-Lubeck-office		1105	X			X		1	X	
WWK-G-LPWD-Well-F		1152	X			X		1	X	

(7)

Relinquished by:	Date:	Time:	Received by:	Date:	Time:
Steve Opp	10/11/01	1330	[signature]	10/12/01	1030

COPIES: White and Yellow Copies accompany samples to the Lab; Pink is retained by Client.

(6) **Project Requirements:**
Rush Turnaround Time (TAT) Requested:
Results needed by: 7 day TAT
NOTE: Rush TAT is subject to Centre's approval and surcharge. Results will be provided via fax or phone.

Laboratory Report Options:
Sample results only
Add case narrative
Add quality control summary
Add calibration summary
Add raw data
Other CED

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Chain of Custody/Analysis Request Form

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

(1) Client: DuPont Washington Works
Route 88 South, DuPont Road
Washington WV 26181
PHONE # 304 863-4476
FAX #

Sampler: Steve Opp

Project Manager: Andrew Hartley

Project Name:

P.O.#

CAL Quotation #:

(2) Sample Identification | (3) Collection Date | Collection Time | Grab | Composite | (4) Matrix: Soil (UST, ACT II, NPDES) | Water (POTABLE, RCRA, SDWA, CWA) | Other | Number of Containers | (5) Analyses Requested: FC-143 (C-8) | Sample Containers | Comments:

Sample Identification	Collection Date	Collection Time	Grab	Composite	Soil	Water	Other	Number of Containers	FC-143 (C-8)	Comments
WWK-6-LPWD-well-A	6/4/01	1130	X			X		1	X	
WWK-6-LPWD-well-B	10/11/01	1140	X			X		1	X	

(7) Relinquished by:	Date:	Time:	Received by:	Date:	Time:
Stephen Opp	6/4/01	1330	[signature]	10/12/01	1030

COPIES: White and Yellow Copies accompany samples to the Lab; Pink is retained by Client.

(6) **Project Requirements:**
Rush Turnaround Time (TAT) Requested:
Results needed by: 7 TAT
NOTE: Rush TAT is subject to Centre's approval and surcharge. Results will be provided via fax or phone.

Laboratory Report Options:
Sample results only
Add case narrative
Add quality control summary
Add calibration summary
Add raw data
Other CEP

Rev 1 12/99

EID185040

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FedEx. Express USA Airbill

FedEx Tracking Number 8313 8103 0853

Recipient's Copy

1 From

Date 10/11/01

Sender's Name Steve Opp Phone 302 366-4476

Company DuPont CRG

Address Route 892 South DuPont Road

City Washington State DE ZIP 19951

2 Your Internal Billing Reference 44D6 WW 7205.191

3 To

Recipient's Name Sample Administration Phone 814 231-8882

Company Center Analytical Laboratories Inc.

Address 3048 Research Drive

To "HOLD" at FedEx location, print FedEx address.

City State PA ZIP 16801

8313 9103 0853

4a Express Package Service — Packages up to 150 lbs. Delivery commitment may be later in some areas.

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- [ ] FedEx Standard Overnight — Next business afternoon
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- [ ] FedEx 2Day — Second business day
- [ ] FedEx Express Saver — Third business day
- [ ] NEW FedEx Extra Hours — Later drop-off with next business afternoon delivery for select locations

FedEx Envelope rate not available. Minimum charge: One-pound rate

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- [ ] FedEx 1Day Freight* — Next business day
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* Call for Confirmation.

* Declared value limit $500

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- [ ] Yes — As per attached Shipper's Declaration
- [ ] Yes — Shipper's Declaration not required

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- [ ] Cargo Aircraft Only

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- [ ] Recipient
- [ ] Third Party
- [ ] Credit Card
- [ ] Cash/Check
- [ ] Obtain Recip. Acct. No.

Total Packages	Total Weight	Total Declared Value†	Total Charges
3	50	$ .00	Credit Card Auth

†Your liability is limited to $100 unless you declare a higher value. See back for details.

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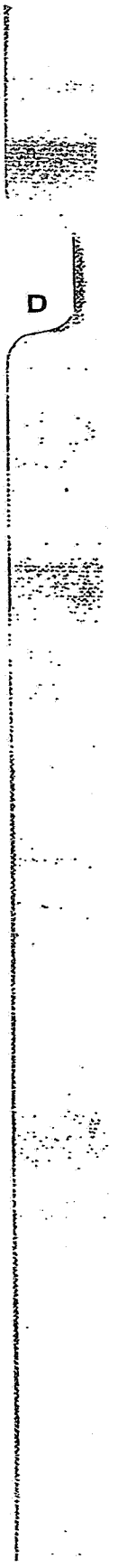

RJZ032568

EID185042

RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	50 ng/L	Set No:	101701A
Exygen Study No:	008-046	Injection Volume:	15 µL	Analyst:	Karen Risha
Analyte:	APFO	Matrix:	Water	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume:	40.0 mL	Extraction Date:	10/17/01
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	10/19/01

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC101501-0	-	CS	101701A-101	0	-	1552	-	-	-	-
XC101501-1	-	CS	101701A-102	50	-	42219	-	-	-	-
XC101501-2	-	CS	101701A-103	100	-	78392	-	-	-	-
XC101501-3	-	CS	101701A-104	250	-	172691	-	-	-	-
XC101501-4	-	CS	101701A-105	500	-	345627	-	-	-	-
XC101501-5	-	CS	101701A-106	1000	-	652107	-	-	-	-
XC101501-6	-	CK	101701A-107	250	-	153082	-	-	-	-
Methanol Wash	-	C	101701A-108	-	-	957	-	-	-	-
0106020 Control	NA	C	101701A-109	-	1	758	NQ	NQ	-	-
0106020 Spk A	NA	LCS	101701A-110	-	1	37136	47.8	49.8	50	96
0106020 Spk B	NA	LCS	101701A-111	-	1	294914	431	449	500	86
0112719 Spk C	WWK-D-Bldg-1-Main	LF	101701A-112	-	2	288866	844	879	500	72
XC101501-1	-	CS	101701A-114	50	-	34578	-	-	-	-
0112720 Spk D	WWK-G-Bld-1-Main-2	LF	101701A-115	-	2	308063	901	938	500	90
0112721 Spk E	WWK-G-Well-332	LF	101701A-117	-	2	310592	909	946	500	86
0112722 Spk F	WWK-D-Bldg-231	LF	101701A-119	-	2	330016	967	1010	500	107
XC101501-2	-	CS	101701A-121	100	-	75043	-	-	-	-
0112719	WWK-D-Bldg-1-Main	S	101701A-122	-	1	332218	487	507	-	-
0112719 Dup	WWK-D-Bldg-1-Main	S	101701A-123	-	1	304179	445	463	-	-
0112720	WWK-G-Bld-1-Main-2	S	101701A-124	-	1	309519	453	471	-	-
0112720 Dup	WWK-G-Bld-1-Main-2	S	101701A-125	-	1	304209	445	463	-	-
0112721	WWK-G-Well-332	S	101701A-126	-	1	326892	479	498	-	-
0112721 Dup	WWK-G-Well-332	S	101701A-127	-	1	307712	450	469	-	-
XC101501-3	-	CS	101701A-128	250	-	167243	-	-	-	-
0112722	WWK-D-Bldg-231	S	101701A-129	-	1	295520	432	450	-	-
0112722 Dup	WWK-D-Bldg-231	S	101701A-130	-	1	301617	441	459	-	-
0112723	WWK-K-FBLK-1	S	101701A-131	-	1	1232	NQ	NQ	-	-
0112724	WWK-K-FBLK-2	S	101701A-132	-	1	771	NQ	NQ	-	-
XC101501-4	-	CS	101701A-133	500	-	350957	-	-	-	-
XC101501-5	-	CS	101701A-134	1000	-	686516	-	-	-	-

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

Recovery (%) = [PFOA found (ng/L)-PFOA found in control (ng/L)] / amount PFOA added (ng/L) x 100

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

Standard Curve : Linear (1/x weighted)
Intercept = 4973.25
Slope = 672.537
Coef. Of Det. = 0.998381

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
NQ = Not quantifiable

Avg Resp. of the 250 ppt Standards (area): 169967
Response of the Check Standard (area): 153082
Percent Difference (%): 11.0
% difference acceptable if within ±15%

Spreadsheet prepared by: KR, 11/08/01

RJZ032569

EID185043

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

PREPARATION OF EXTRACTED CALIBRATION STANDARDS

Protocol No.: None
Method No.: *
Exygen Study No.: 008-046
Analytes: PFOA
Matrix: Type I Water^
Sample Vol: 40 mL

Sponsor Sample ID	Exygen Sample ID	Sample Description	Fort. Solution ID	Fort. Soln. Conc. (ng/mL)	Fort. Volume (µL)	Micropipet used (µL)	Fort. Level (ppt)	Final Solution ID # **	Reagents/ Materials	Lot #
NA	0106020	Type I Water^	-	-	-	-	-	XC101501-0	Methanol	41202
NA	0106020	Type I Water^	F070901-3	10	200	200	50	XC101501-1	C18 SPE	W1236B2
NA	0106020	Type I Water^	F070901-3	10	400	200	100	XC101501-2	Type I Water	NA
NA	0106020	Type I Water^	F070901-2	100	100	200	250	XC101501-3	-	-
NA	0106020	Type I Water^	F070901-2	100	200	200	500	XC101501-4	-	-
NA	0106020	Type I Water^	F070901-2	100	400	200	1000	XC101501-5	-	-
NA	0106020	Type I Water^	F101501-2	100	100	200	250	XC101501-6	Initials/Date:	X2 10-15-01
-	-	-	-	-	-	-	-	-		

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 # 18 Time: 1320 Initials/Date: X2 / 10/15/01

40 mL of each sample measured using a 50 mL graduated cylinder. Initials/Date: X2 / 10/15/01

After measuring, samples returned to refrigerator / freezer # 18 Time: 1450 Initials/Date: X2 / 10/15/01

Samples fortified: Initials/Date: ZA / 10/15/01

SPE clean-up: Initials/Date: X2 / 10/15/01

Final volume adjusted to 5 mL: Initials/Date: X2 / 10/15/01

Extracts placed in refrigerator # 13 Initials/Date: KR / 10/15/01

STANDARD EXPIRATION DATE: 10/29/01

Comments: ^This type I water has been filtered through a hypercarb filter

* Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water.

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: 008-046 Matrix: Water
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 18
Time 0910 Date 10-17-01 Initials XZ

CLIENT SAMPLE ID	EXYGEN ID NUMBER	VOLUME (mL)	FORTIFICATION (ng)
na	0106020 Control	40.0	-
na	0106020 Spk A	40.0	2.0
na	0106020 Spk B	40.0	20.0
WWK-D-Bldg-1-Main	0112719 Spk C	40.0	20.0
WWK-G-Bldg-1-Main-2	0112720 Spk D	40.0	20.0
WWK-G-Well 332	0112721 Spk E	40.0	20.0
WWK-D-Bldg-231	0112722 Spk F	40.0	20.0
WWK-D-Bldg-1-Main	0112719	40.0	-
WWK-D-Bldg-1-Main	0112719 Dup	40.0	-
WWK-G-Bldg-1-Main-2	0112720	40.0	-
WWK-G-Bldg-1-Main-2	0112720 Dup	40.0	-
WWK-G-Well 332	0112721	40.0	-
WWK-G-Well 332	0112721 Dup	40.0	-
WWK-D-Bldg-231	0112722	40.0	-
WWK-D-Bldg-231	0112722 Dup	40.0	-
WWK-K-FBLK-1	0112723	40.0	-
WWK-K-FBLK-2	0112724	40.0	-
-	-	-	-
-	-	-	-
-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
0106020 Spk A	F070901-3 (10 ng/mL)	200 µL (200 µL micropipet)	ZA 10/17/01
0106020 Spk B	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112719 Spk C	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112720 Spk D	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112721 Spk E	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112722 Spk F	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/

All samples were measured:
Time 0950. Date 10-17-01 Initials XZ

After measuring samples were returned to refrigerator No. 18
Time 1010 Date 10-17-01 Initials XZ

Comments: 200 µL of 250 mg/mL sodium thiosulfate was added to all samples before spiking. Initials/Date: XZ 10/17/01

Analysis Summary:
Data Set: 101701A Initials/Date: BC / 10/18/01
Data Set: — Initials/Date: — / —
Data Set: — Initials/Date: — / —

Set extraction/analysis data verified by: JMF Date: 10/19/01

July 26, 2001/6

RJZ032571

EID185045

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: 008-046 METHOD: $$ PROTOCOL NUMBER: NA
MATRIX: Water ANALYTES: PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	0106020 Control			–		–	–	Methanol	41202
na	0106020 Spk A			–		–	–	C18 SPE	W1236B2
na	0106020 Spk B			–		–	–	Type I Water	NA
WWK-D-Bldg-1-Main	0112719 Spk C			0.5/1.0		–	–	-	-
WWK-G-Bldg-1-Main-2	0112720 Spk D					–	–	-	-
WWK-G-Well 332	0112721 Spk E					–	–	-	-
WWK-D-Bldg-231	0112722 Spk F					–	–	-	-
WWK-D-Bldg-1-Main	0112719			–		–	–	-	-
WWK-D-Bldg-1-Main	0112719 Dup			–		–	–	-	-
WWK-G-Bldg-1-Main-2	0112720			–		–	–	*Initials/Date*	XZ 10-17-01
WWK-G-Bldg-1-Main-2	0112720 Dup			–		–	–	-	-
WWK-G-Well 332	0112721			–		–	–	-	-
WWK-G-Well 332	0112721 Dup			–		–	–	-	-
WWK-D-Bldg-231	0112722			–		–	–	-	-
WWK-D-Bldg-231	0112722 Dup			–		–	–	-	-
WWK-K-FBLK-1	0112723			–		–	–	HPLC	
WWK-K-FBLK-2	0112724			–		–	–	Methanol	41025
-	-	-	-	-	-	-	-	Ammonium Acetate	142476
-	-	-	-	-	-	-	-	Type I water	NA
-	-	-	-	-	-	-	-	-	-
*Initials/Date	-	XZ 10-17-01	XZ 10-17-01	KJC 10/18/01	KJC ① 10/18/01	–	–	*Initials/Date*	KJC 10/18/01

①SAMPLES PLACED ON AUTOSAMPLER ON 10/18/01 BUT DID NOT INJECT UNTIL 10/19/01 KJC 10/19/01

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.

*Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water

COMMENTS:

Final extracts stored in refrigerator 15 Initials: KJC Date: 10/17/01

July 19, 2001/4

EID185046

RJZ032572

Exygen RESEARCH
3058 Research Drive
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SAMPLE TRANSFER SHEET

Protocol Number: NA Exygen Study Number: 008-046

Exygen Sample #	Sponsor Sample #	Exygen Sample #	Sponsor Sample #
0112719	WWK-D-Bldg-1-Main	0112729	WWK-G-LPWD-Well-B
0112720	WWK-G-Bldg-1-Main 2	-	-
0112721	WWK-G-Well-332	-	-
0112722	WWK-D-Bldg-231	-	-
0112723	WWK-K-FBLK-1	-	-
0112724	WWK-K-FBLK-2	-	-
0112725	WWK-D-Bldg-293	-	-
0112726	WWK-D-Lubeck-Office	-	-
0112727	WWK-G-LPWD-Well-F	-	-
0112728	WWK-G-LPWD-Well-A	-	-

Requested Location: Refrig 18

KAREN 10/15/01
Requested By (Printed Name/Date)

Samples removed from: C-6 Box# 01-271 Shelf# C1
Samples removed from: — Box# — Shelf# —
Samples removed from: — Box# — Shelf# —
Samples removed from: — Box# — Shelf# —

Samples moved to R-18

✓ EL 10-15-01
Sample Custodian Initials/Date

Samples removed from R-18
Samples returned to C-6

RK 10-26-01
Sample Custodian Initials/Date

Comments:

July 12, 2001/4

RJZ032573

EID185047

Masslynx - Sample List

Sample List: C:\MASSLYNX\008-046.PRO\SampleDB\101701a.spl
Printed: Thu Oct 18 13:50:03 2001 RL 10/18/01

Exygen STUDY NO. 008-046

Page Position: (1, 1)

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc A	Conc B	Conc C	Test ID	DF	MS Method
1	1	101701A-101	---	---	XC101501-0, 0 ppt Standard	---	Standard	0	---	---	0	1	PFOA
2	2	101701A-102	---	---	XC101501-1, 50 ppt Standard	---	Standard	50	---	---	0	1	PFOA
3	3	101701A-103	---	---	XC101501-2, 100 ppt Standard	---	Standard	100	---	---	0	1	PFOA
4	4	101701A-104	---	---	XC101501-3, 250 ppt Standard	---	Standard	250	---	---	0	1	PFOA
5	5	101701A-105	---	---	XC101501-4, 500 ppt Standard	---	Standard	500	---	---	0	1	PFOA
6	6	101701A-106	---	---	XC101501-5, 1000 ppt Standard	---	Standard	1000	---	---	0	1	PFOA
7	7	101701A-107	---	---	XC101501-6, 250 ppt Check Standard	---	QC	250	---	---	0	1	PFOA
8	92	101701A-108	---	---	Methanol Wash	---	Blank	---	---	---	0	1	PFOA
9	11	101701A-109	---	---	0106020 Control	---	Blank	---	---	---	0	1	PFOA
10	12	101701A-110	---	---	0106020 Spk A, 50 ppt	---	QC	50	---	---	0	1	PFOA
11	13	101701A-111	---	---	0106020 Spk B, 500 ppt	---	QC	500	---	---	0	1	PFOA
12	14	101701A-112	---	---	0112719 Spk C, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
13	15	101701A-113	---	---	0112719 Spk C, 500 ppt	---	QC	500	---	---	0	1	PFOA
14	2	101701A-114	---	---	XC101501-1, 50 ppt Standard	---	Standard	50	---	---	0	1	PFOA
15	16	101701A-115	---	---	0112720 Spk D, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
16	17	101701A-116	---	---	0112720 Spk D, 500 ppt	---	QC	500	---	---	0	1	PFOA
17	18	101701A-117	---	---	0112721 Spk E, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
18	19	101701A-118	---	---	0112721 Spk E, 500 ppt	---	QC	500	---	---	0	1	PFOA
19	20	101701A-119	---	---	0112722 Spk F, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
20	21	101701A-120	---	---	0112722 Spk F, 500 ppt	---	QC	500	---	---	0	1	PFOA
21	3	101701A-121	---	---	XC101501-2, 100 ppt Standard	---	Standard	100	---	---	0	1	PFOA
22	22	101701A-122	---	---	0112719	---	Analyte	---	---	---	0	1	PFOA
23	23	101701A-123	---	---	0112719 Dup	---	Analyte	---	---	---	0	1	PFOA
24	24	101701A-124	---	---	0112720	---	Analyte	---	---	---	0	1	PFOA
25	25	101701A-125	---	---	0112720 Dup	---	Analyte	---	---	---	0	1	PFOA
26	26	101701A-126	---	---	0112721	---	Analyte	---	---	---	0	1	PFOA
27	27	101701A-127	---	---	0112721 Dup	---	Analyte	---	---	---	0	1	PFOA
28	4	101701A-128	---	---	XC101501-3, 250 ppt Standard	---	Standard	250	---	---	0	1	PFOA
29	28	101701A-129	---	---	0112722	---	Analyte	---	---	---	0	1	PFOA
30	29	101701A-130	---	---	0112722 Dup	---	Analyte	---	---	---	0	1	PFOA
31	30	101701A-131	---	---	0112723	---	Analyte	---	---	---	0	1	PFOA
32	31	101701A-132	---	---	0112724	---	Analyte	---	---	---	0	1	PFOA
33	5	101701A-133	---	---	XC101501-4, 500 ppt Standard	---	Standard	500	---	---	0	1	PFOA
34	6	101701A-134	---	---	XC101501-5, 1000 ppt Standard	---	Standard	1000	---	---	0	1	PFOA

Exygen STUDY NO. 008-046

Sample List: C:\MASSLYNX\008-046.PRO\SampleDB\101701a.spl
Printed: Thu Oct 18 13:50:03 2001 KJL 10/18/01

	HPLC Method	MS Tune File	Inj.Volume
1	WATER	Water	15
2	WATER	Water	15
3	WATER	Water	15
4	WATER	Water	15
5	WATER	Water	15
6	WATER	Water	15
7	WATER	Water	15
8	WATER	Water	15
9	WATER	Water	15
10	WATER	Water	15
11	WATER	Water	15
12	WATER	Water	15
13	WATER	Water	15
14	WATER	Water	15
15	WATER	Water	15
16	WATER	Water	15
17	WATER	Water	15
18	WATER	Water	15
19	WATER	Water	15
20	WATER	Water	15
21	WATER	Water	15
22	WATER	Water	15
23	WATER	Water	15
24	WATER	Water	15
25	WATER	Water	15
26	WATER	Water	15
27	WATER	Water	15
28	WATER	Water	15
29	WATER	Water	15
30	WATER	Water	15
31	WATER	Water	15
32	WATER	Water	15
33	WATER	Water	15
34	WATER	Water	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA | Exygen Study No: 008-046

Instrument: Micromass Quattro Ultima (LC/MS/MS Unit #6) 7 (RE) KR 11/08/01

ELECTROSPRAY ION SOURCE:

Capillary: 3.0 kV	Hexapole 2: 0 V
Hexapole 1: 0 V	Source Block Temp.: 100°C
Aperture 1: 0 V	Desolvation Temp.: 300°C

ANALYZER:

LM Res 1: 10.5 V	LM Res 2: 13.0 V
HM Res 1: 10.5 V	HM Res 2: 13.0 V
IEnergy 1: 1.0 V	IEnergy 2: 2.0 V
Entrance: -2 V	Multiplier: 650 V
Exit: 2 V	

GAS FLOWS AND PRESSURE:

Desolvation N_2 Flow Rate: ~650 L/hr
Nebuliser N_2 Flow Rate: ~150 L/hr
Gas Cell Pressure: ~0.003 mbar

Computer: COMPAQ Professional Workstation AP200

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

HPLC Equipment: Hewlett Packard (HP) Series 1100

HP Binary Pump	HP 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: 35A)
(JONESCHROMATOGRAPHY: Part No. FK5962E)

Column Temperature: 35°C
Mobile Phase (A) : 2 mM Ammonium Acetate in Type I Water
Mobile Phase (B) : Methanol

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

Total run time = 14 min

Injected Volume: 15 µL

Ions monitored :

Analyte	Parent ion	Daughter ion	Dwell (secs)	Coll Energy (eV)	Cone (V)
PFOA	413	369	0.2	10	50

Analyst: Karen Risha
Exygen Research
3058 Research Drive, State College, PA 16801 KR 10/18/01
Phone: (814) 272-1039 FAX: (814) 231-1580

All Handwritten Peak ID's by: KR 10/19/01

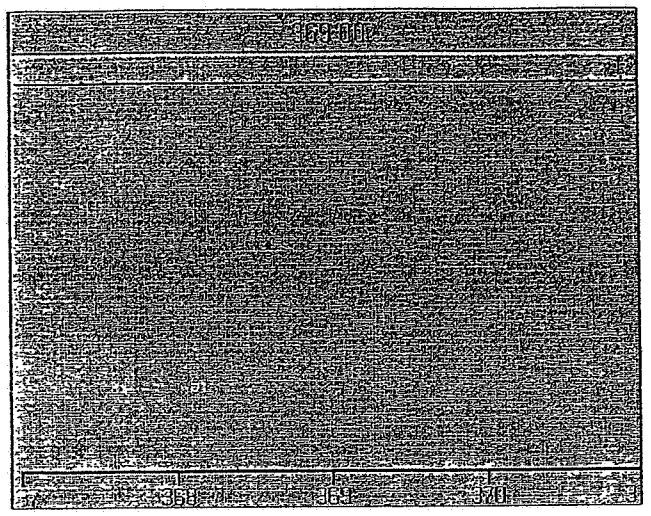
NOTE: THE NEXT 3 PAGES ARE MASSLYNX PRINTOUTS FROM THE SYSTEM AND CAN BE USED TO VERIFY VALUES LISTED ON THIS PAGE. KR 11/09/01

Tuning Method Report

Method: C:\MASSLYNX\008-046.PRO\ACQUDB\WATER

Printed: Fri Nov 09 09:37:01 2001

KJR 11/09/01



Dau 413.00

SOURCE (ESP-)	Set	Rdbk	Analyser	Set	Rdbk
Capillary	3.00	-2.93	LM Res 1	10.5	
Cone	50	-20	HM Res 1	10.5	
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	100	Exit	2	11
Desolvation Temp.	300	301	LM Res 2	13.0	
			HM Res 2	13.0	
			IEnergy 2	2.0	
			Multiplier	650	-644

Pressures	Rdbk	Gas Flows	Rdbk
Analyser Vacuum	OFF	Cone Gas	136.5
Gas Cell	2.6e-3	Desolvation	660.4

RJZ032577

EID185051

Scanning Method Report

Method: C:\MASSLYNX\008-046.PRO\ACQUDB\PFOA
Last Modified: Mon Oct 15 12:37:26 2001

Printed: Fri Nov 09 09:41:26 2001 KEF 11/09/01

Solvent Delay (mins) : 1.00

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

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 8.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	369.00	0.20	10	50

Method File: C:\MASSLYNX\008-046.PRO\ACQUDB\water
Last Modified: Friday, November 09, 2001 09:38:47

Printed: Friday, November 09, 2001 09:42:01 KJC 11/09/01

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)	14.0
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 9 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

HP1100 LC Pump External Event Timetable

The Timetable contains 4 entries which are :

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

HP1100 Autosampler Initial Conditions

Draw Speed	200.0
Eject Speed (µl/min)	200
Draw Position (mm)	0.10
Stop Time (mins)	14.00
Injection Volume(µl)	15.0
Vial Number	6

Exygen™ RESEARCH
Precise Research.
Proven Results.

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

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

KJ 11/09/01

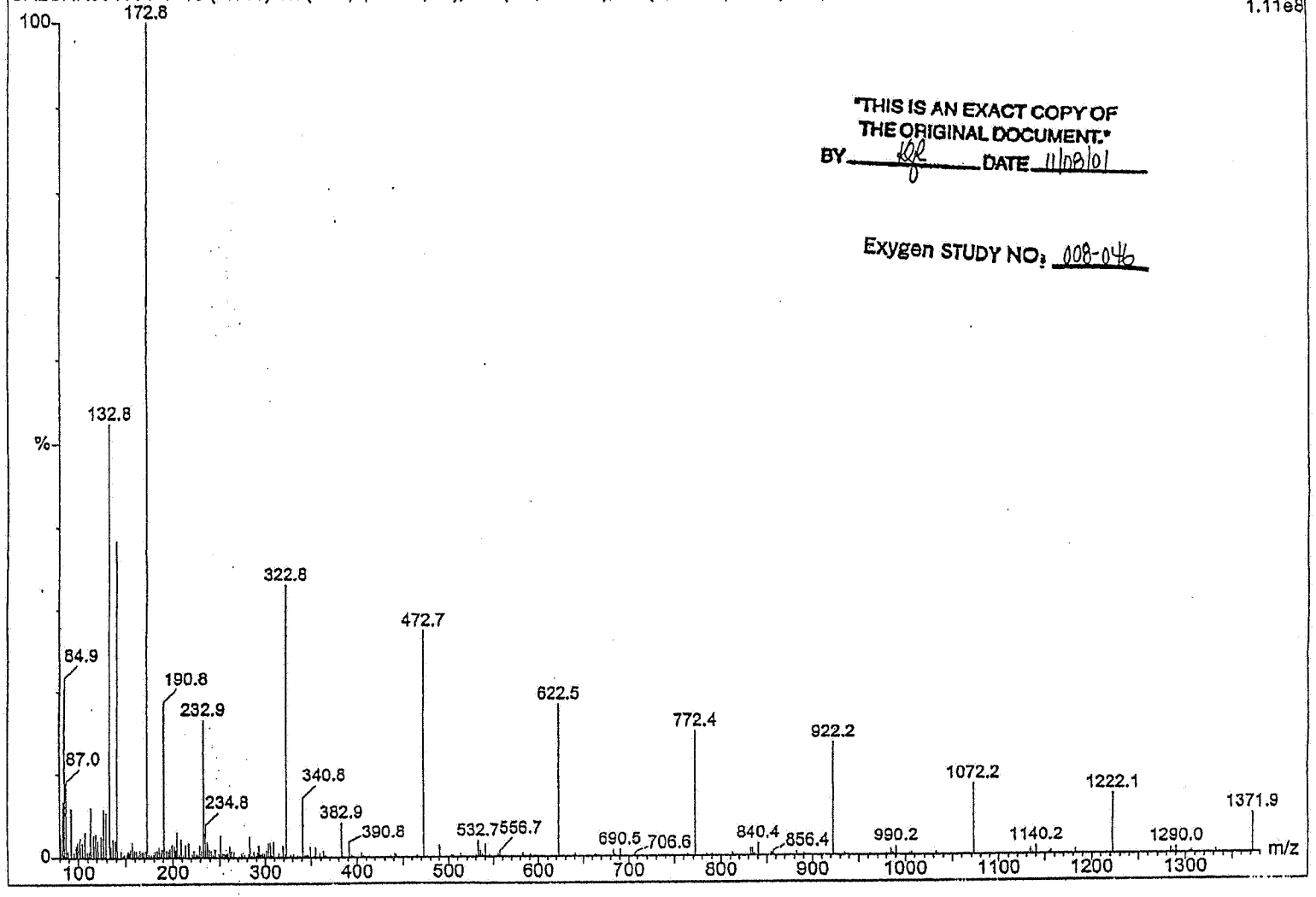
3058 Research Drive
State College, PA 16801, USA
T: 814.272.1039
F: 814.231.1580
exygen.com

Calibration Check of NaIRbCs090800 on MS1 using NaIRbCs tuning solution

KGL 09/10/01

LC/MS/MS #7
10-Sep-2001 08:53:03
Scan ES+
1.11e8

CALCHK091001-1 13 (0.811) Cn (Cen,2, 80.00, Ht); Sm (Mn, 2x2.00); Sb (1,40.00); Cm (1:16)



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BY KGL DATE 11/08/01

Exygen STUDY NO: 008-046

EID185055

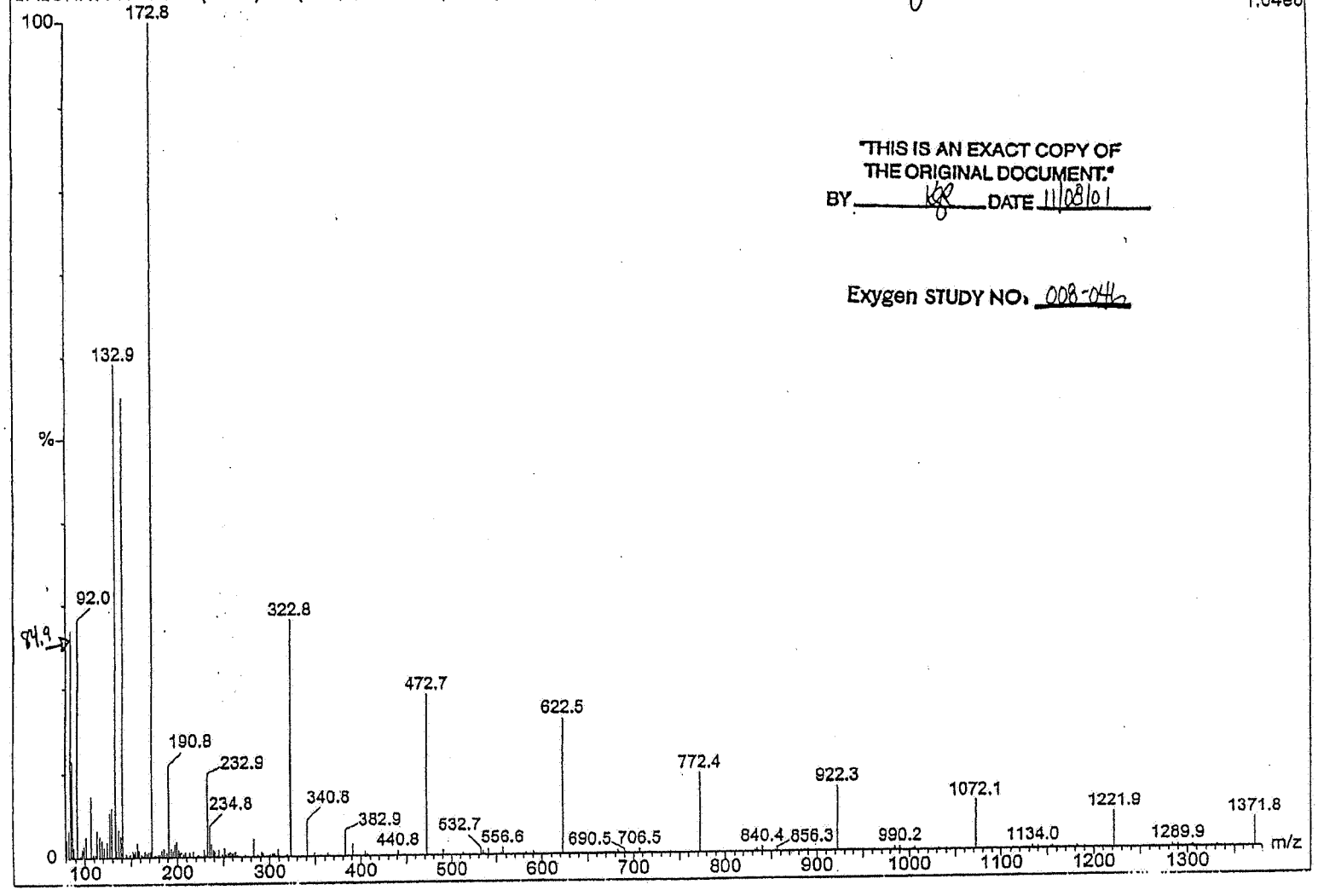
RJZ032581

Calibration Check of NaIRbCs090800 on MS2 using NaIRbCs tuning solution

CALCHK091001-2 9 (0.569) Cn (Cen,2, 80.00, Ht); Sm (Mn, 2x2.00); Sb (1,40.00); Cm (1:17)

KJR 09/10/01

LC/MS/MS #7
10-Sep-2001 08:55:20
MS2 ES+
1.04e8



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BY KJR DATE 11/08/01

Exygen STUDY NO. 008-046

EID185056

RJZ032582

Initial Calibration Curve

Exygen Study No.: 008-046
Set No: 101701A
Extraction Date: 10/17/01
Analyzed on: 10/19/01

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
XC101501-0	0	1552
XC101501-1	50	42219
XC101501-2	100	78392
XC101501-3	250	172691
XC101501-4	500	345627
XC101501-5	1000	652107

Coefficient of Determination: 0.99916321

KJL 11/09/01

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.99958152
R Square	0.99916321
Adjusted R Squa	0.99895402
Standard Error	7974.56702
Observations	6

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	3.03736E+11	3.04E+11	4776.1882	2.6265E-07
Residual	4	254374876.7	63593719		
Total	5	3.0399E+11			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	10229.6406	4406.260677	2.321615	0.0809931	-2004.12568	22463.4068	-2004.1257	22463.4068
X Variable 1	648.005346	9.376437708	69.10997	2.627E-07	621.972127	674.038564	621.972127	674.038564

RJZ032583

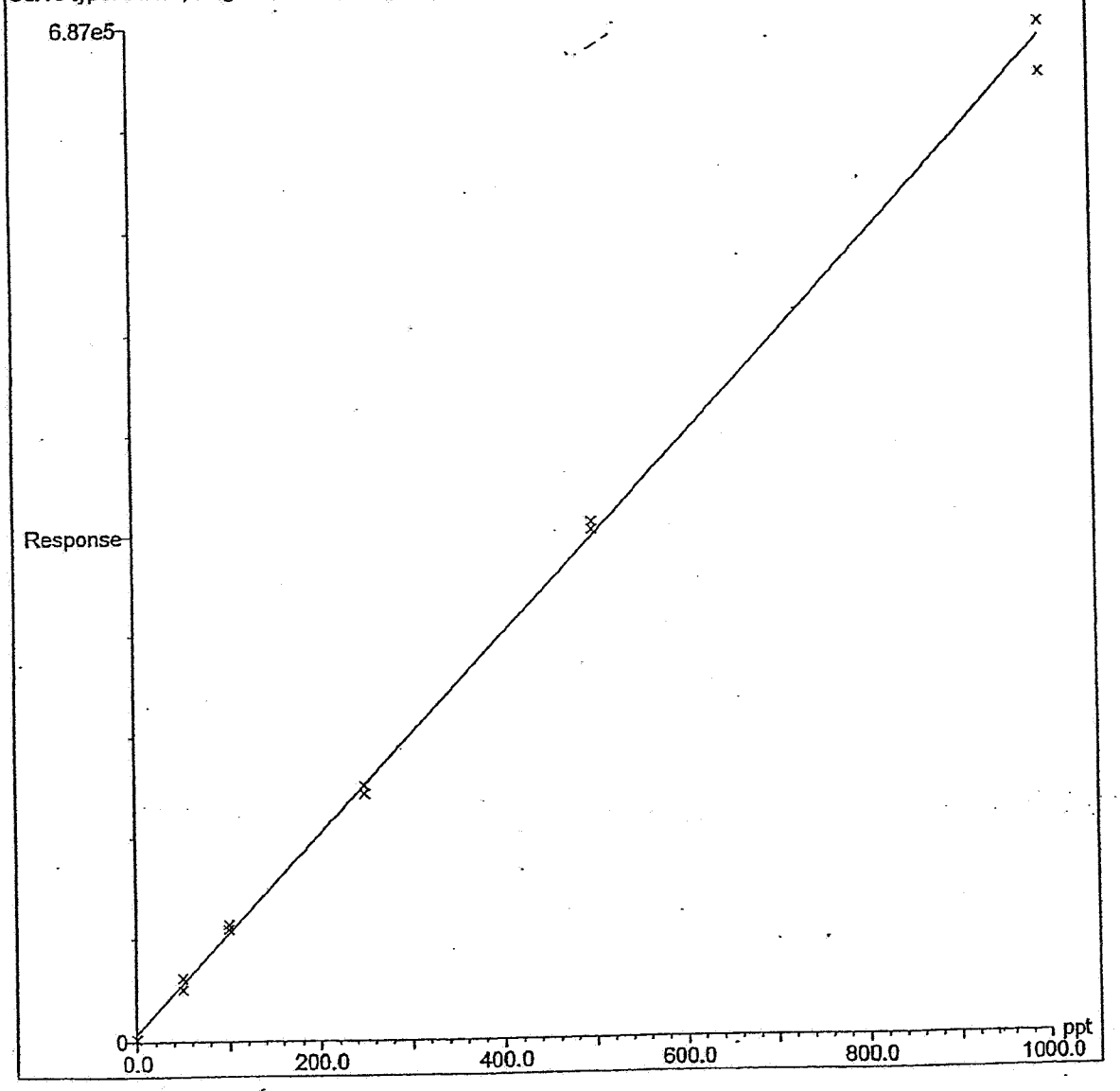
EID185057

Quantify Calibration Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Calibration: P:\Data\LCMSMS6\Masslynx\008-046.PRO\CurveDB\101701A
Last modified: Fri Oct 19 16:30:35 2001
Printed: Fri Oct 19 16:33:11 2001

KJR 10/19/01

Compound 1 name: PFOA
Coefficient of Determination: 0.998381
Calibration curve: 672.537 * x + 4973.25
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Exygen Research

Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

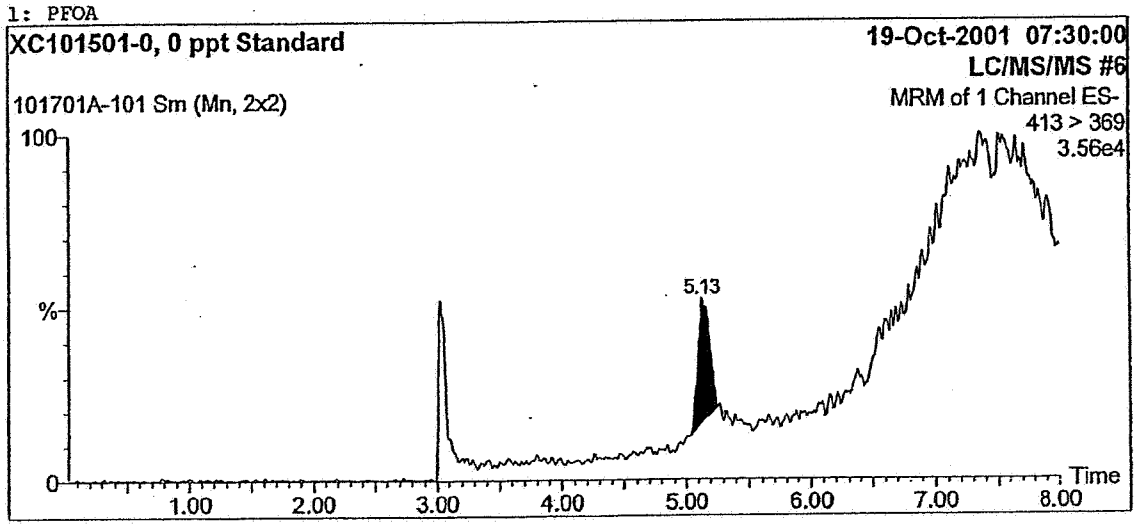
Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Initials KR
Date 10/19/01
Run# 101701A-101 To 101701A-134

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-101
Text:

1: PFOA



Exygen Research

RJZ032585

EID185059

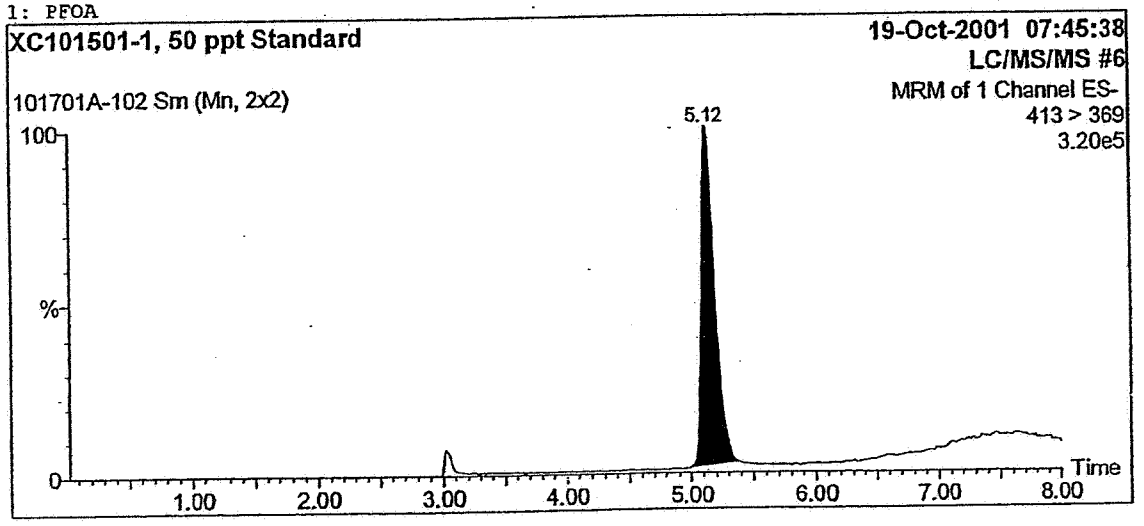
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-102
Text:

1: PFOA



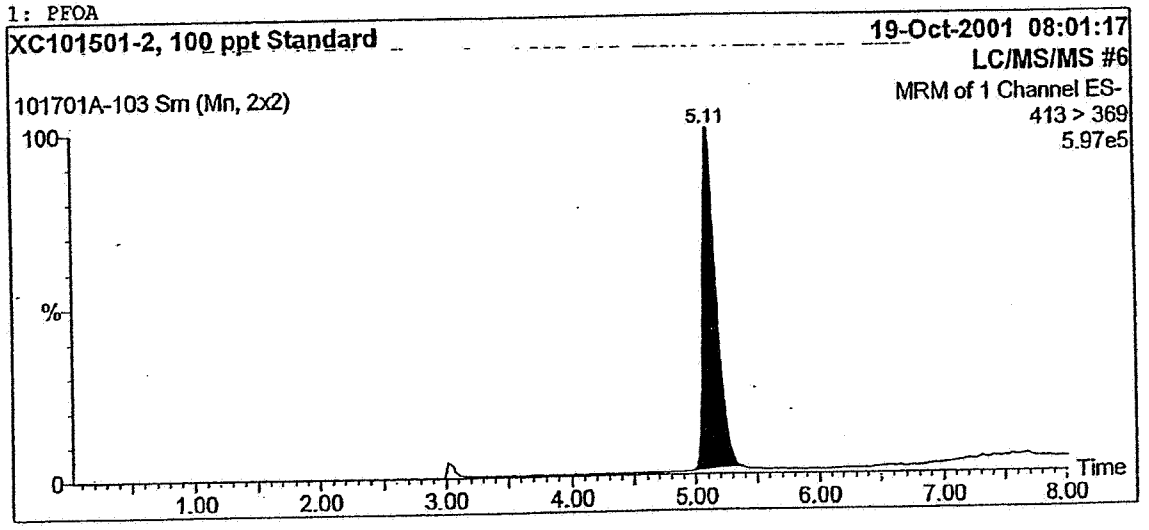
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-103
Text:

1: PFOA



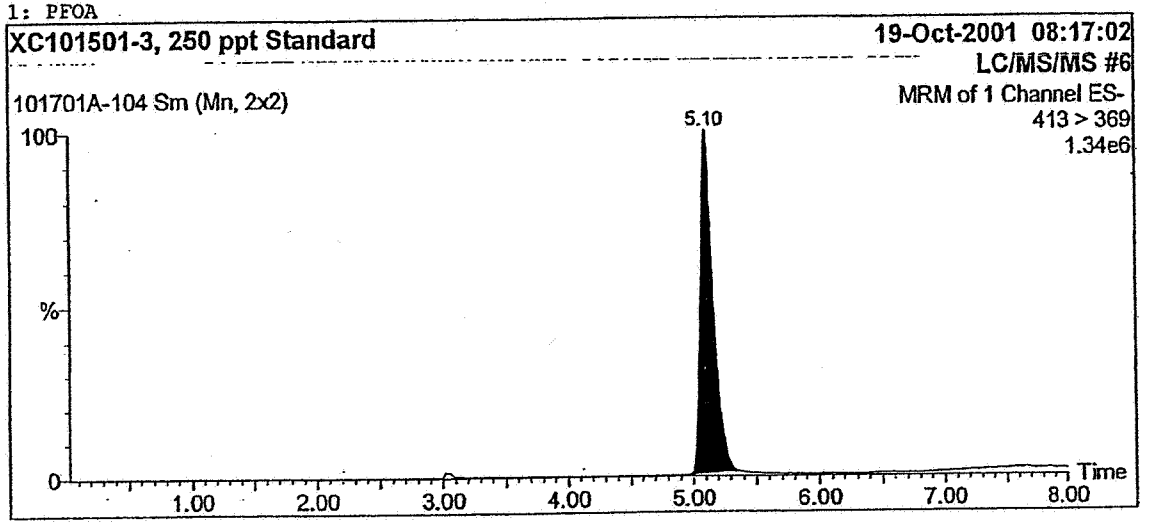
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-104
Text:

1: PFOA



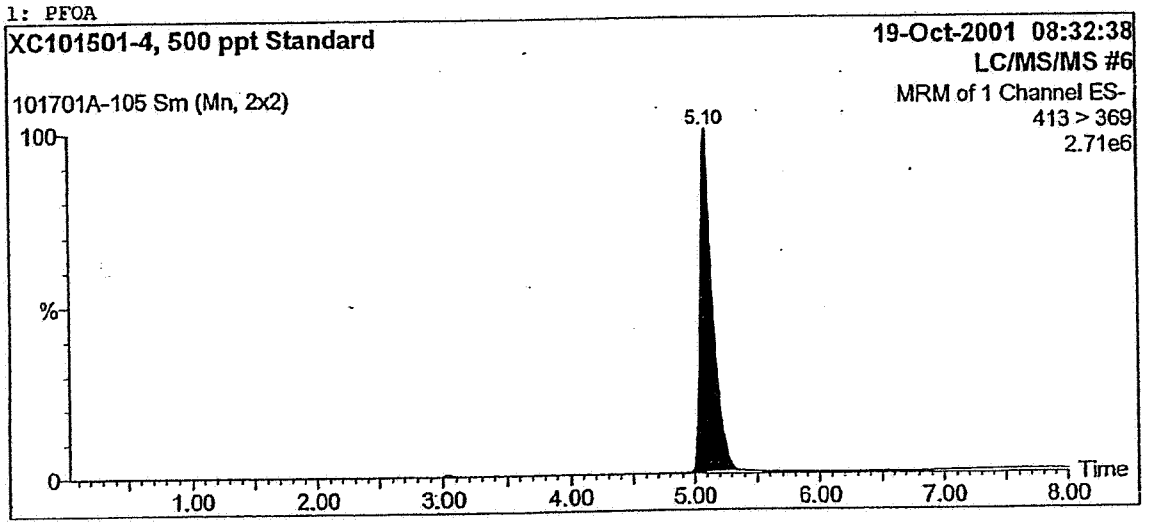
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-105
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

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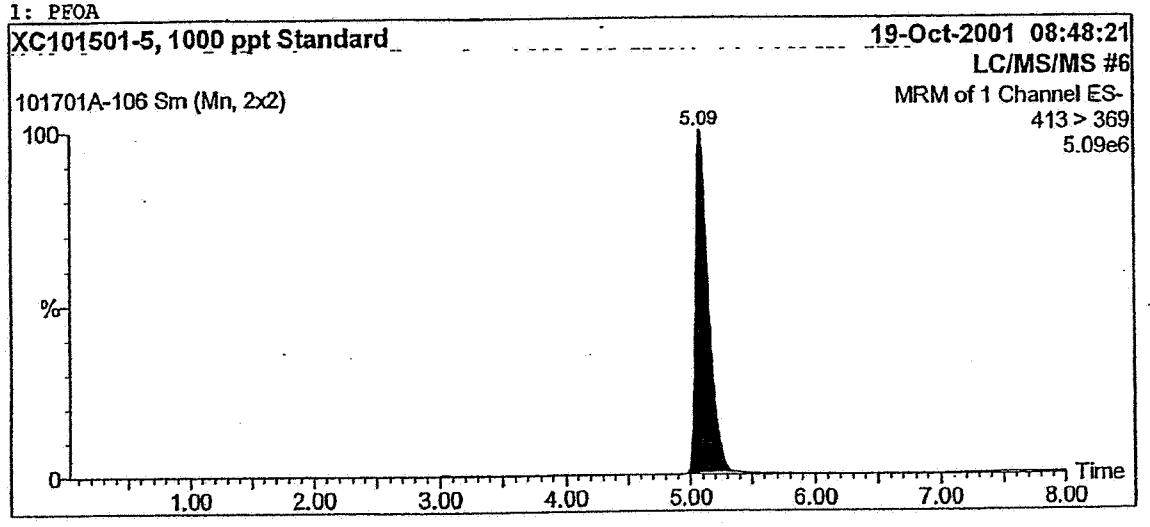
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-106
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

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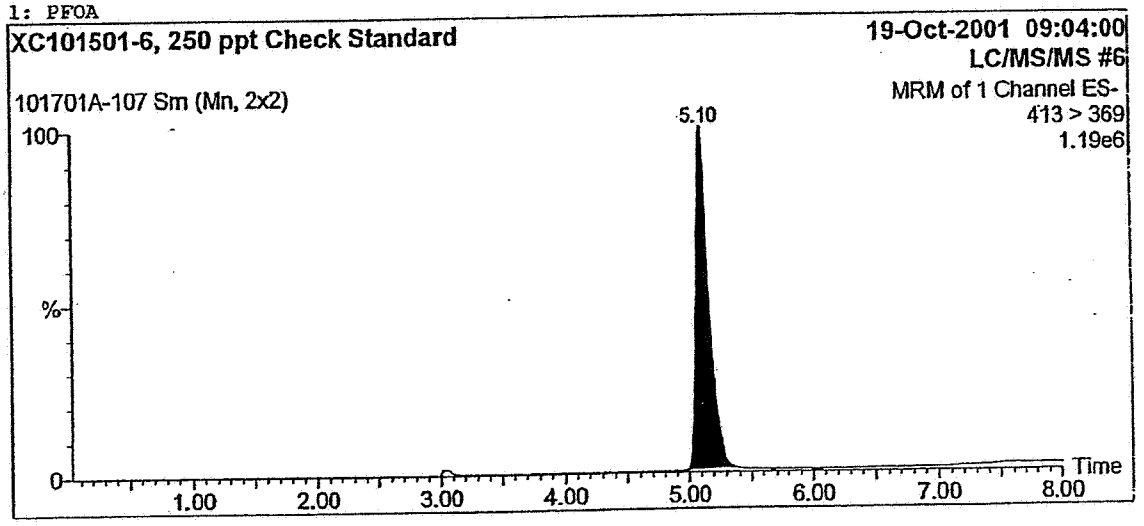
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-107
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
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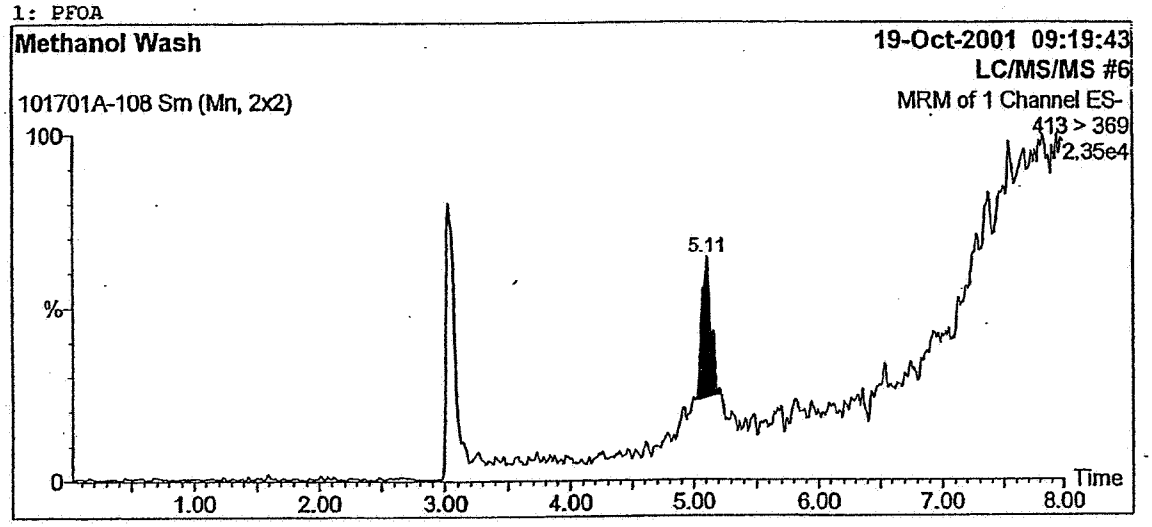
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-108
Text:

1: PFOA



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Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

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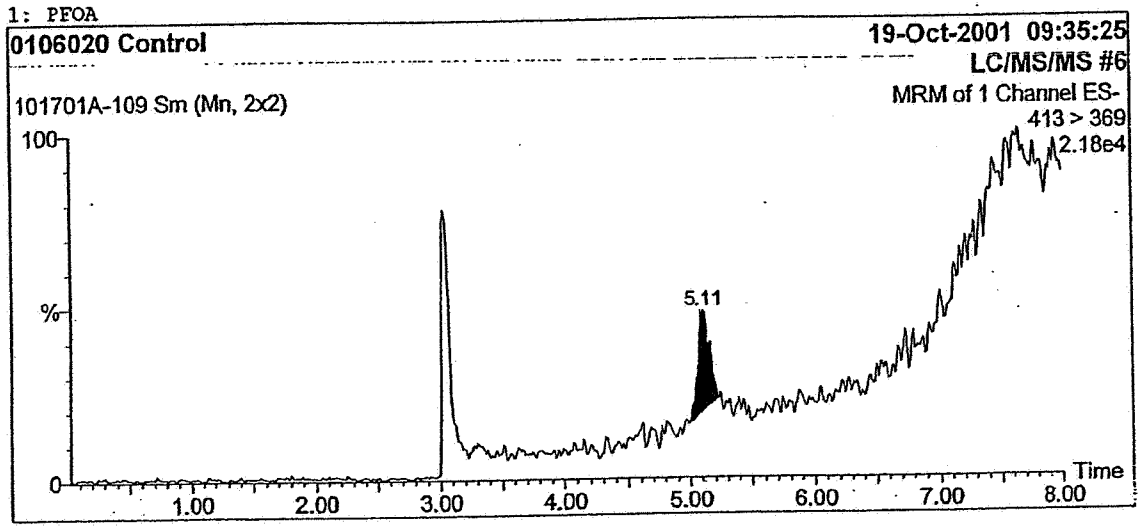
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-109
Text:

1: PFOA



Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
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Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-110
Text:

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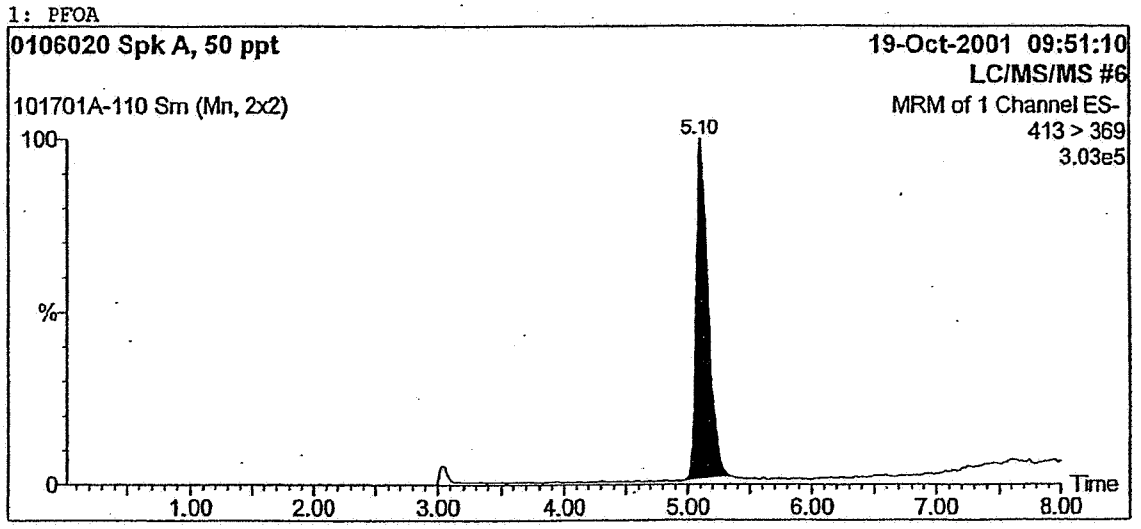

Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

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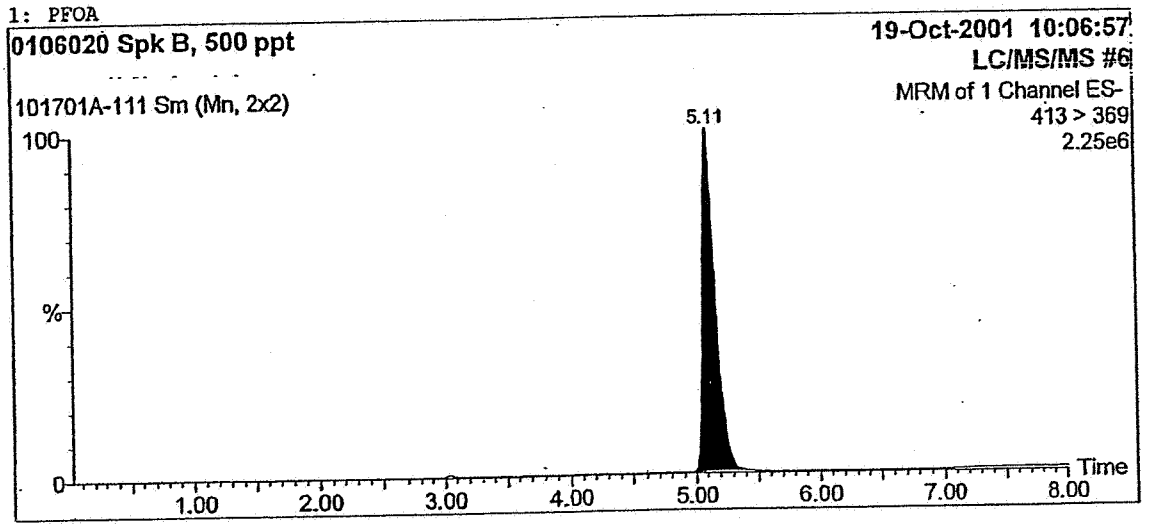
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-111
Text:

1: PFOA



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Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

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Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-112
Text:

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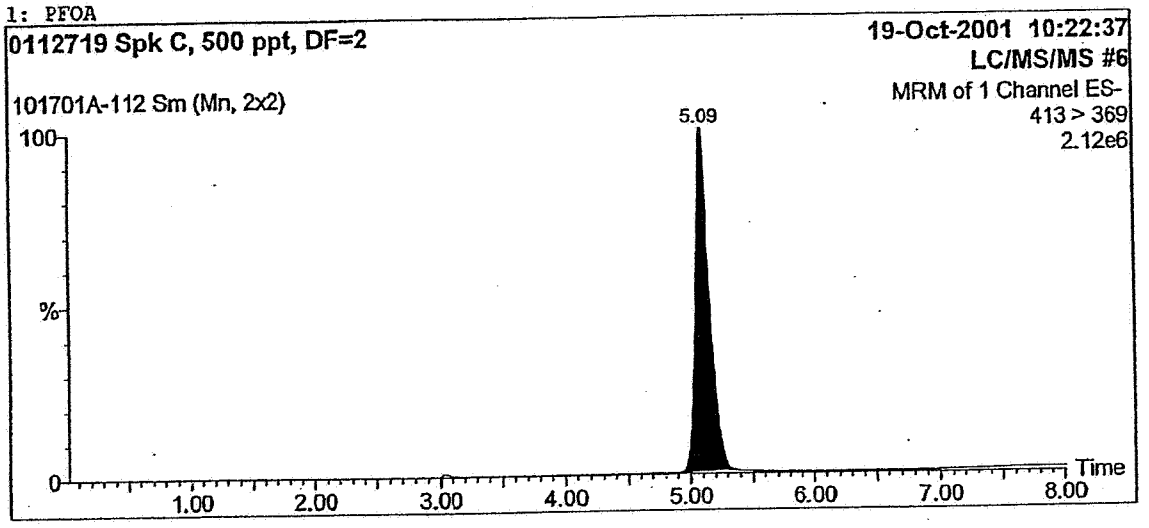

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Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
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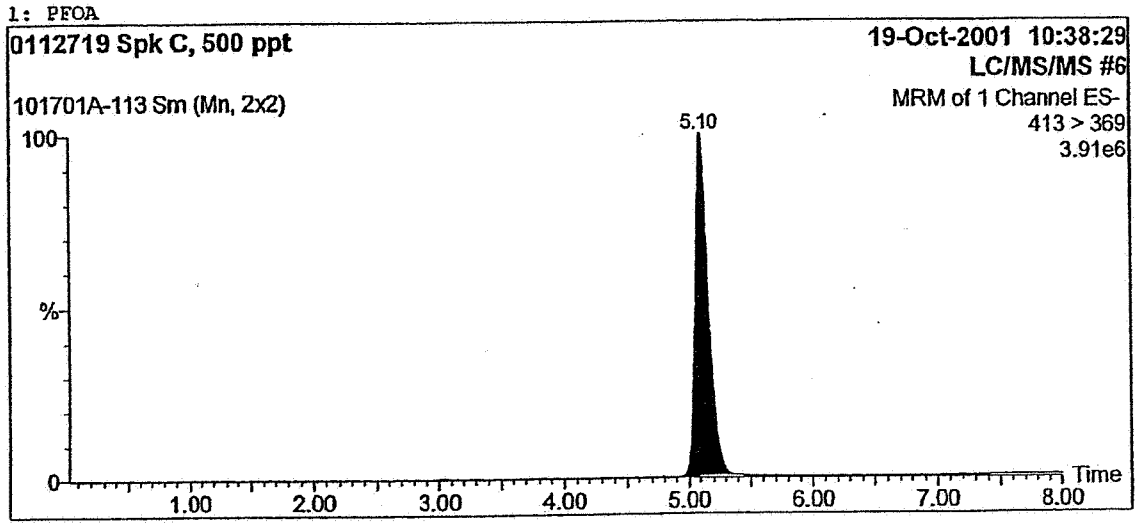
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-113
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

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Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-114
Text:

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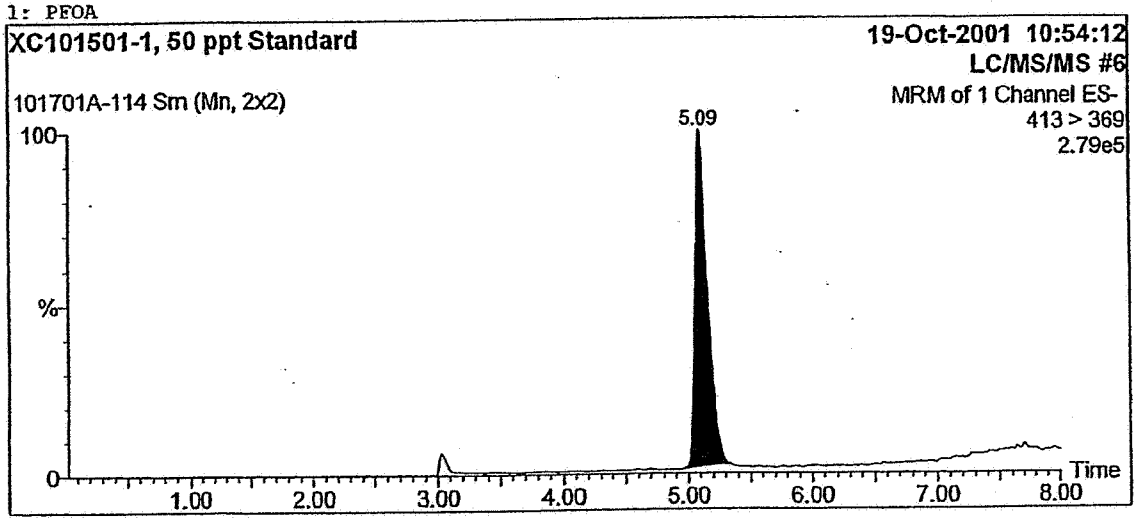


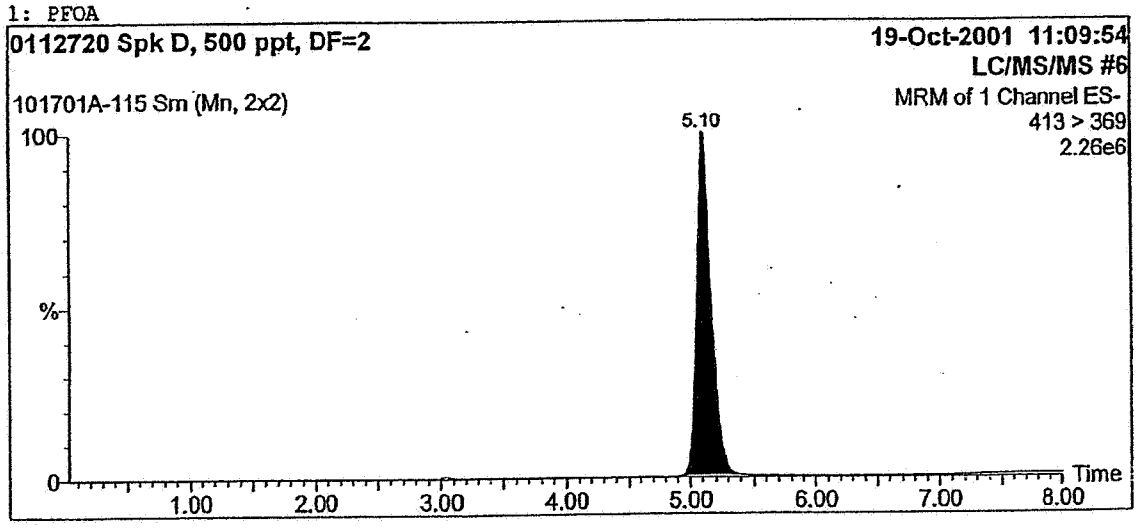
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-115
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
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Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-116
Text:

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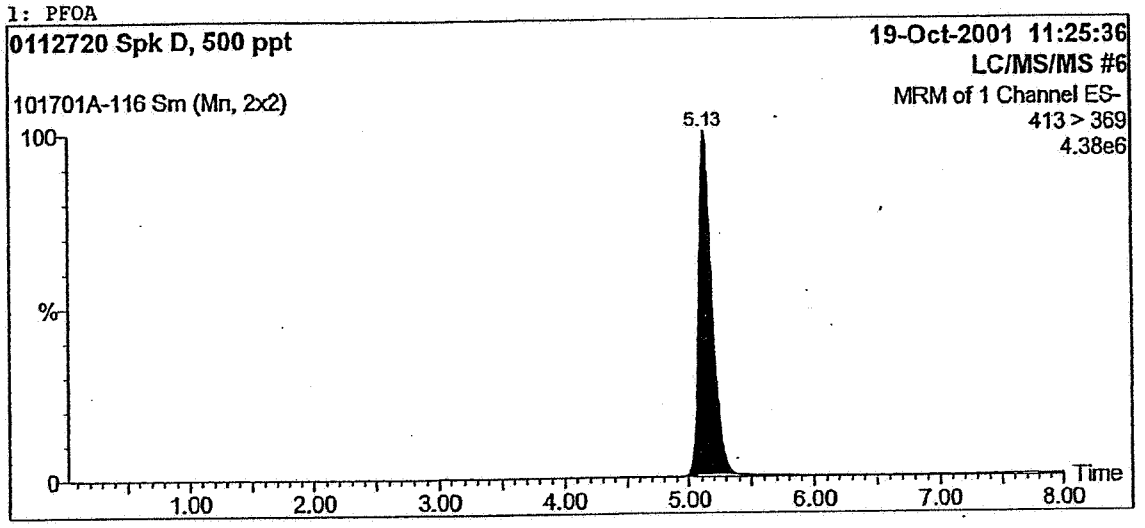

Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
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Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-117
Text:

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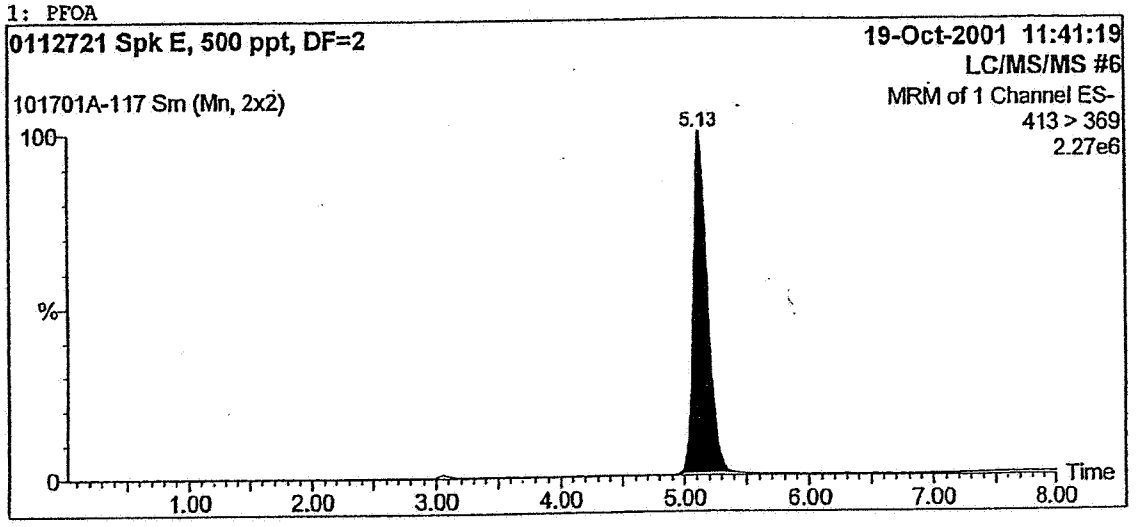

Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
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Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-118
Text:

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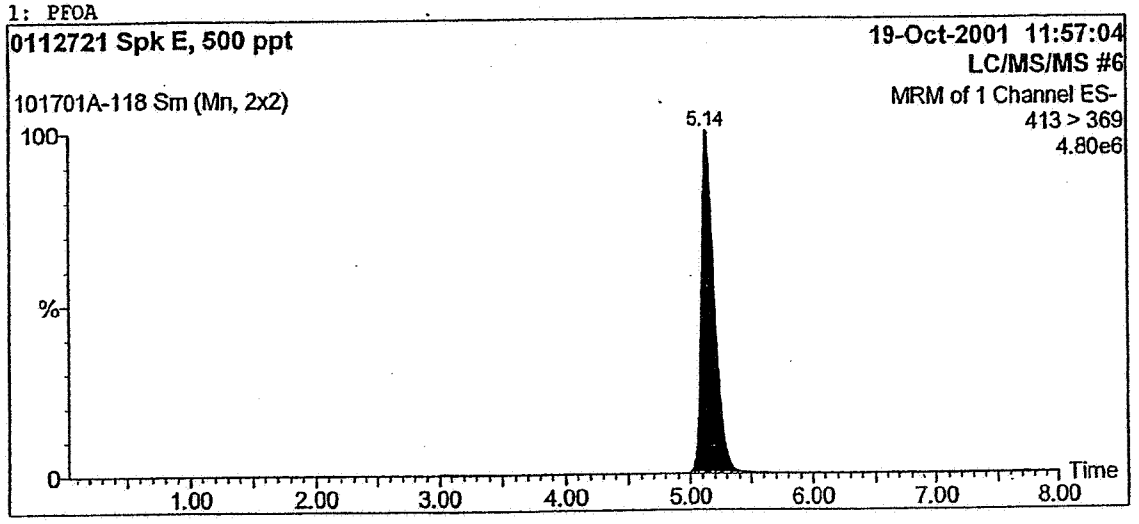

Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-119
Text:

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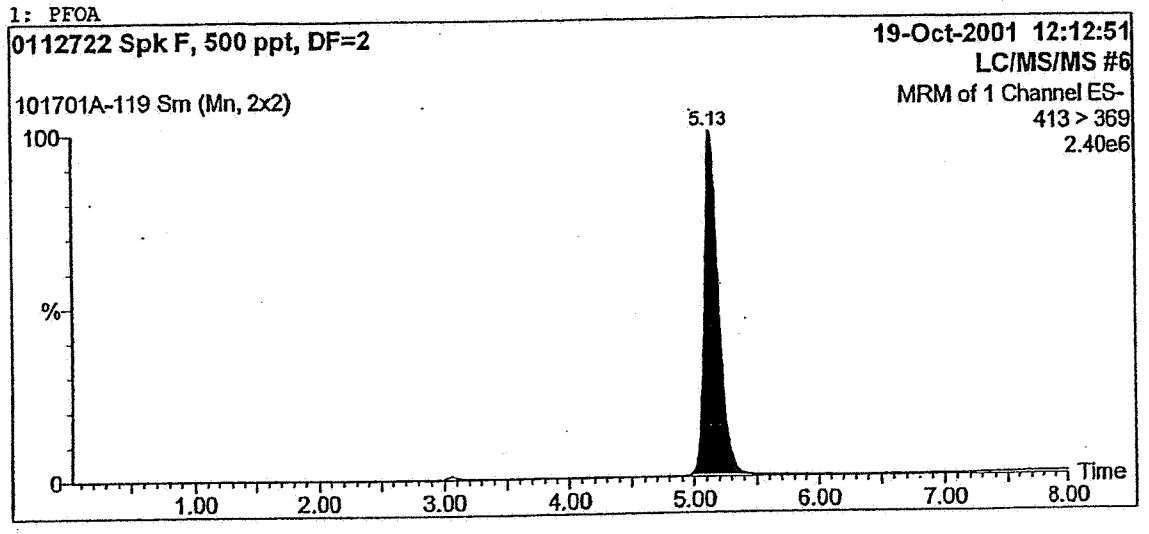

Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

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Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-120
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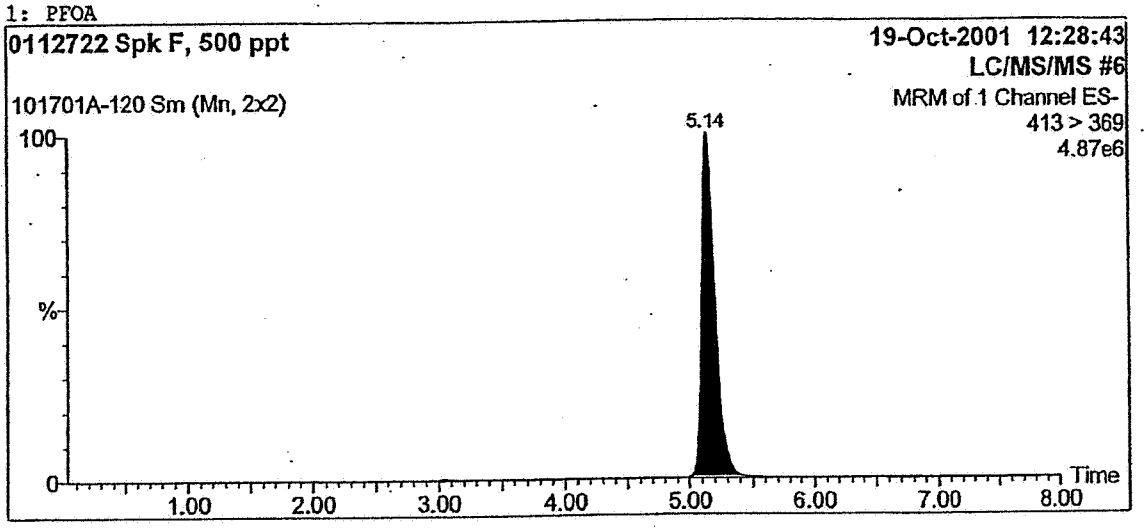

Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
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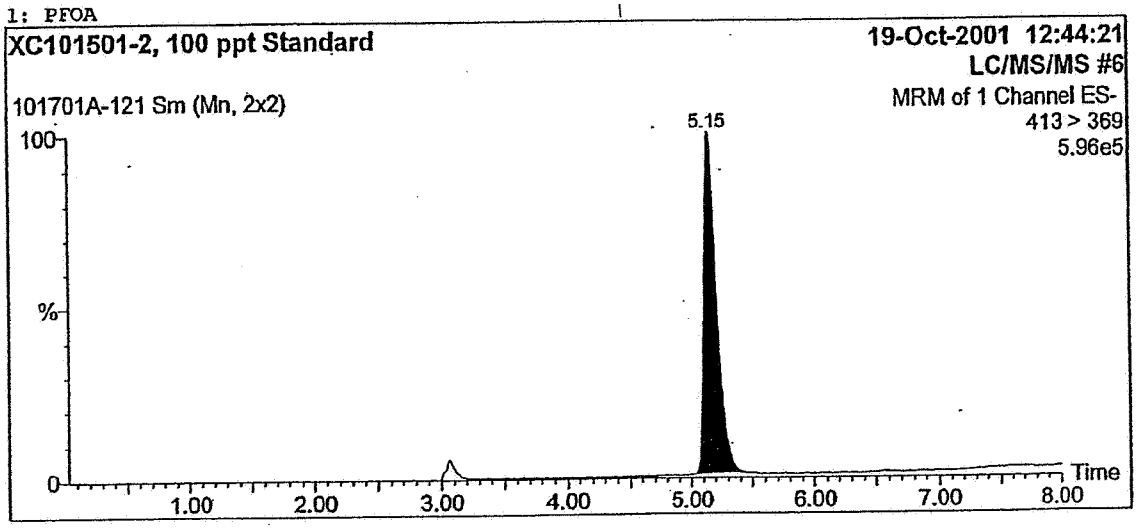
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-121
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
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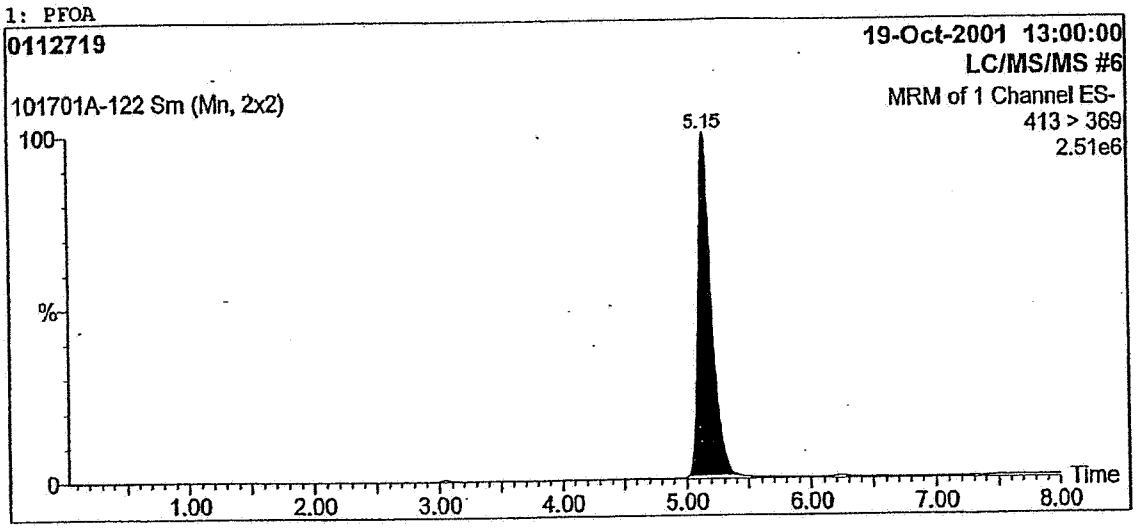
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-122
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
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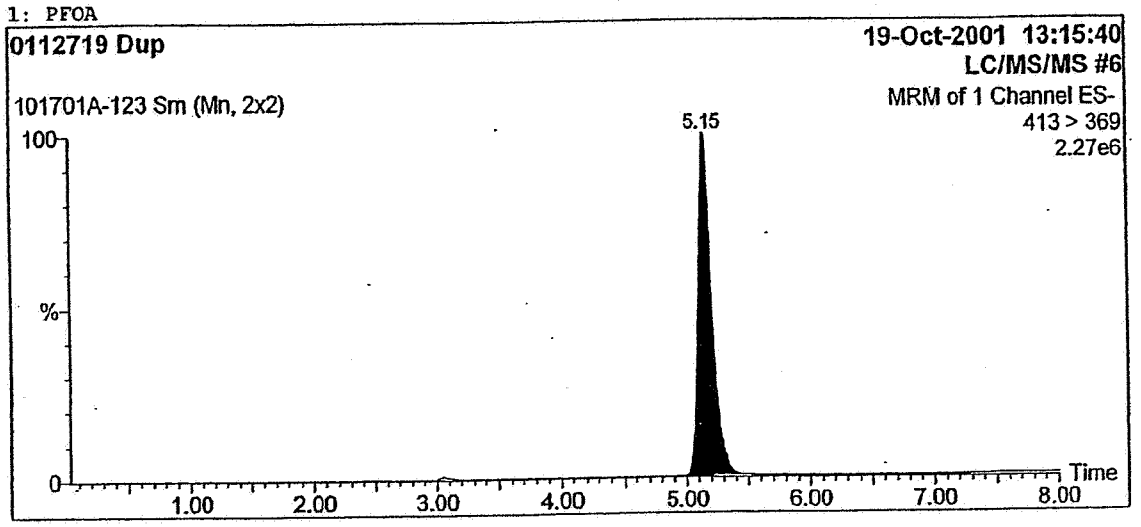
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-123
Text:

1: PFOA



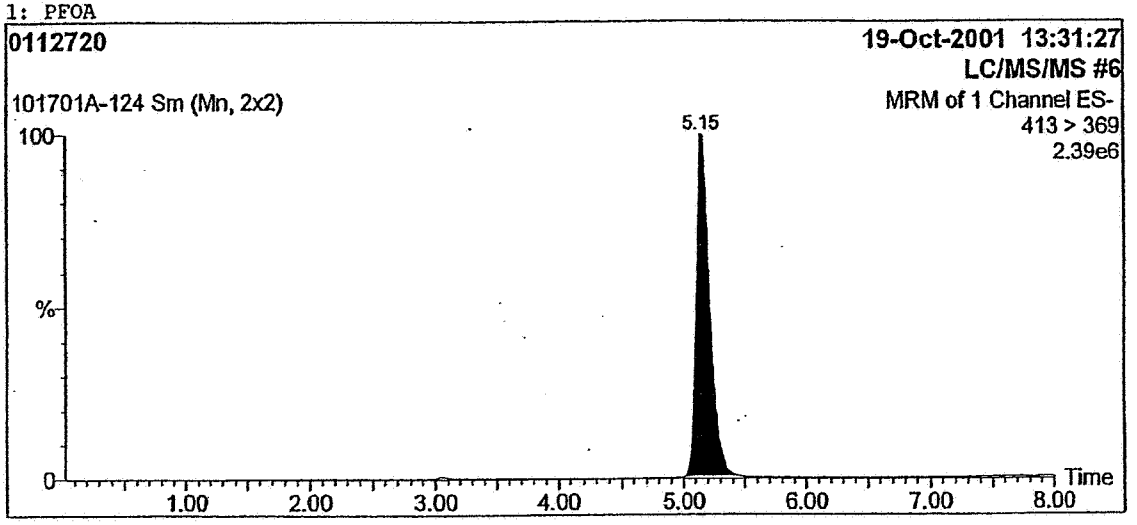
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-124
Text:

1: PFOA



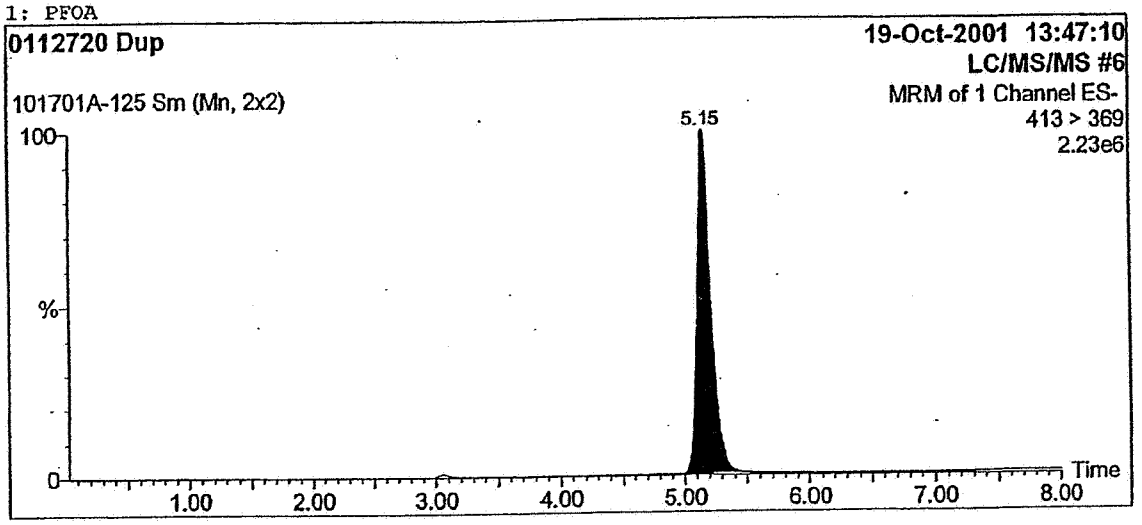
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-125
Text:

1: PFOA



Exygen Research

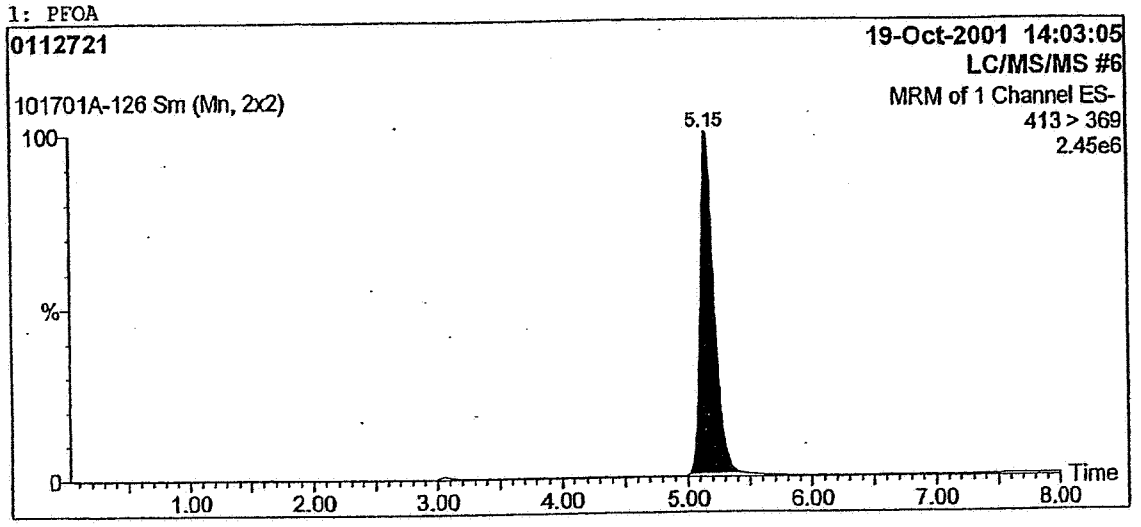
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-126
Text:

1: PFOA



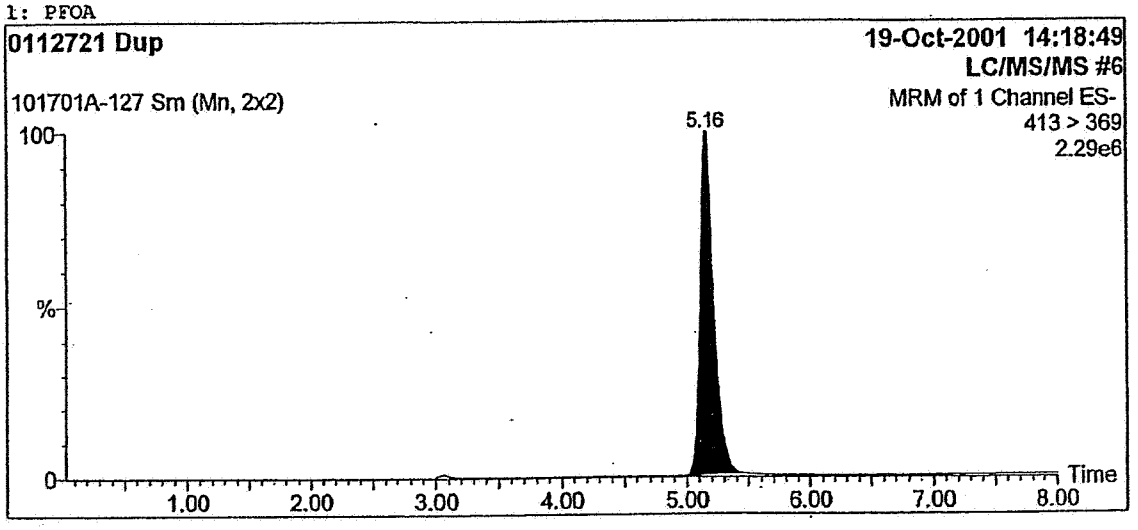
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-127
Text:

1: PFOA



Exygen Research

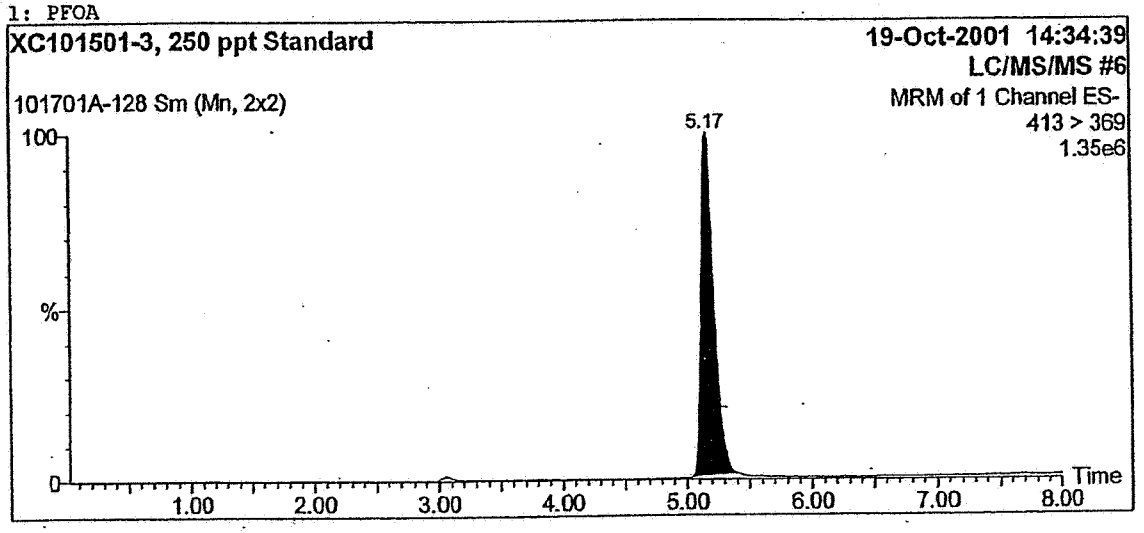
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-128
Text:

1: PFOA



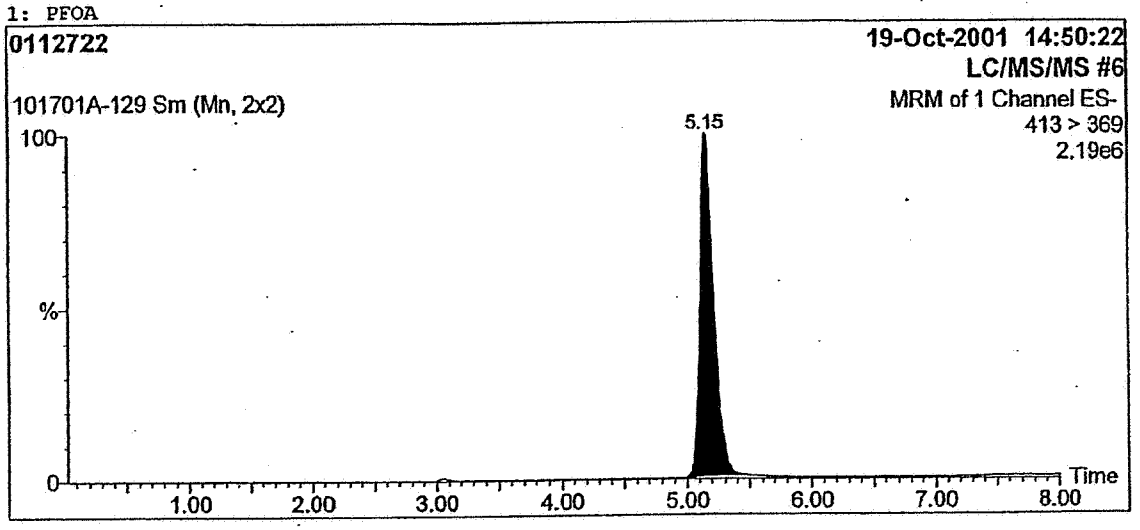
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-129
Text:

1: PFOA



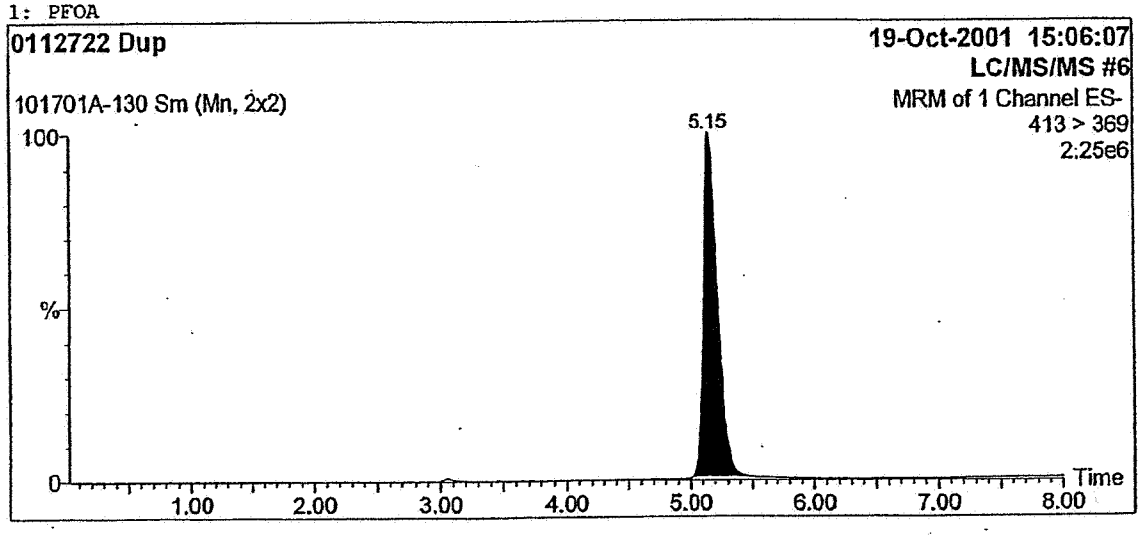
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-130
Text:

1: PFOA



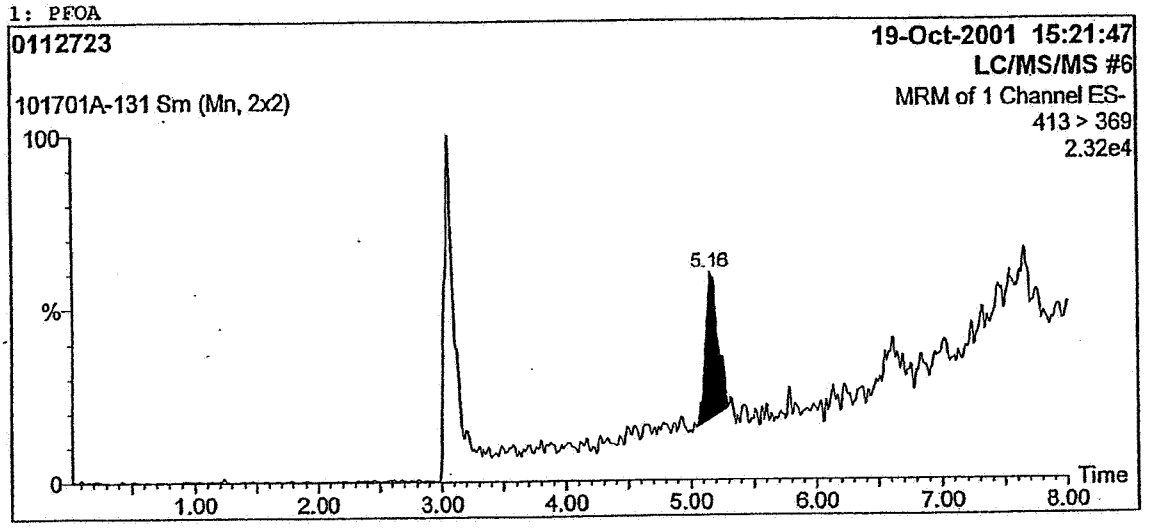
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-131
Text:

1: PFOA



Exygen Research

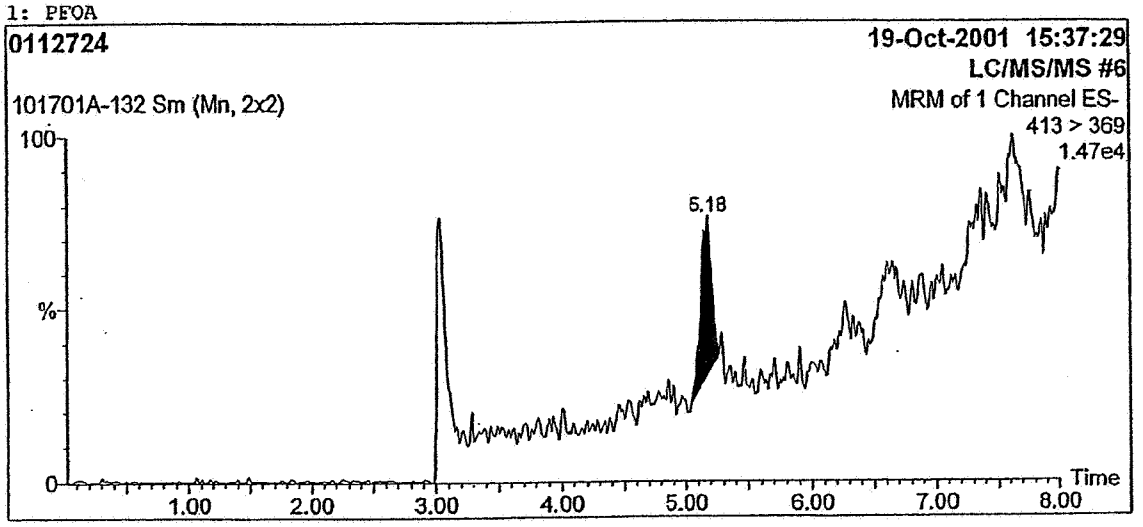
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-132
Text:

1: PFOA



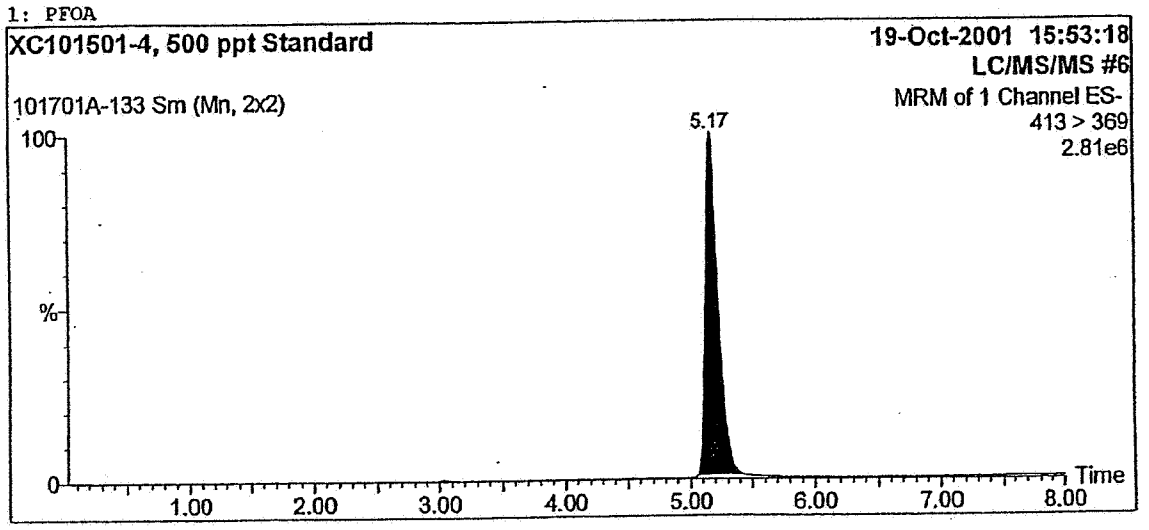
Quantify Sample Report
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-133
Text:

1: PFOA



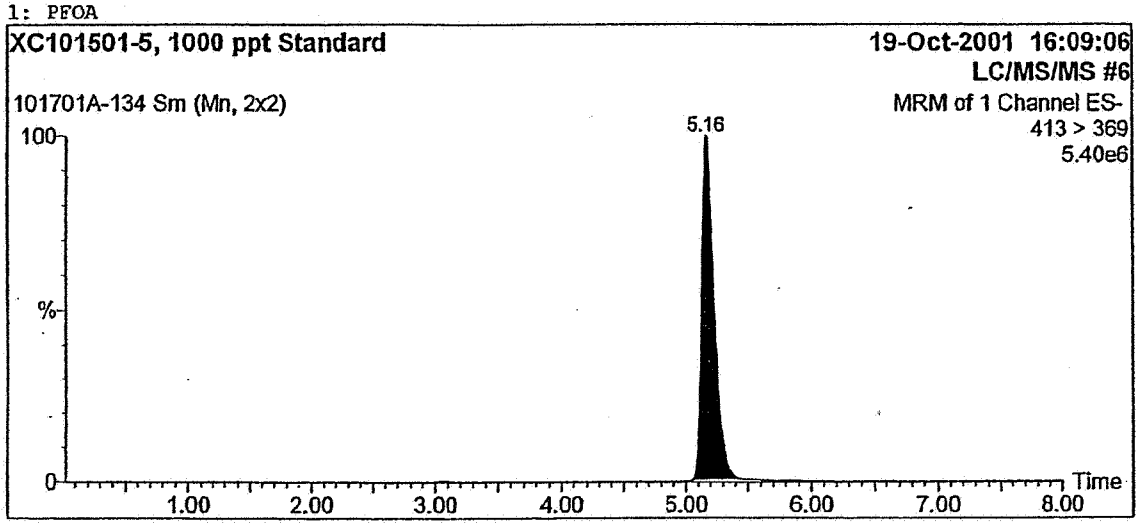
Study No.: 008-046, Set No: 101701A, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS6\Masslynx\008-046.PRO\SampleDB\101701a
Last modified: Fri Oct 19 16:25:32 2001
Method: P:\Data\LCMSMS6\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Fri Oct 19 16:19:26 2001
Job Code:

Printed: Fri Oct 19 16:33:13 2001

Name: 101701A-134
Text:

1: PFOA



RAW DATA REPORT

Sponsor Study No:	NA	Limit of Quantitation:	50 ng/L	Set No:	101701B
Exygen Study No:	008-046	Injection Volume:	15 µL	Analyst:	Karen Risha
Analyte:	APFO	Matrix:	Water	Instrument Type:	LC/MS/MS Unit # 7
Ions Monitored:	413 -> 369	Sample Volume:	40.0 mL	Extraction Date:	10/17/01
Site:	NA	Final Volume:	5.0 mL	Analyzed on:	10/18/01

Exygen ID	Sponsor ID	Sample Code	Run No.	Std. Conc. (ng/L)	Dilution Factor	Peak Area	PFOA Found (ng/L)	APFO Found (ng/L)	Amount PFOA Added (ng/L)	Recovery (%)
XC101501-0	-	CS	101701B-201	0	-	ND	-	-	-	-
XC101501-1	-	CS	101701B-202	50	-	2269	-	-	-	-
XC101501-2	-	CS	101701B-203	100	-	4485	-	-	-	-
XC101501-3	-	CS	101701B-204	250	-	9753	-	-	-	-
XC101501-4	-	CS	101701B-205	500	-	20662	-	-	-	-
XC101501-5	-	CS	101701B-206	1000	-	40877	-	-	-	-
XC101501-6	-	CK	101701B-207	250	-	9594	-	-	-	-
Methanol Wash	-	C	101701B-208	-	-	ND	-	-	-	-
0106020 Control	NA	C	101701B-209	-	1	ND	ND	ND	-	-
0106020 Spk A	NA	LCS	101701B-210	-	1	1896	42.5	44.2	50	85
0106020 Spk B	NA	LCS	101701B-211	-	1	19553	506	526	500	101
0112725 Spk C	WWK-D-Bldg-293	LF	101701B-213	-	1	35585	926	964	500	104
0112726 Spk D	WWK-D-Lubeck-Office	LF	101701B-214	-	2	20859	1080	1120	500	97
XC101501-1	-	CS	101701B-216	50	-	2212	-	-	-	-
0112727 Spk E	WWK-G-LPWD-Well-F	LF	101701B-218	-	1	27533	715	744	500	88
0112728 Spk F	WWK-G-LPWD-Well-A	LF	101701B-219	-	2	21348	1110	1150	500	87
0112729 Spk G	WWK-G-LPWD-Well-B	LF	101701B-222	-	1	37615	979	1020	500	84
XC101501-2	-	CS	101701B-223	100	-	3863	-	-	-	-
0112725	WWK-D-Bldg-293	S	101701B-224	-	1	15758	406	423	-	-
0112725 Dup	WWK-D-Bldg-293	S	101701B-225	-	1	14574	375	390	-	-
0112726	WWK-D-Lubeck-Office	S	101701B-226	-	1	23022	597	621	-	-
0112726 Dup	WWK-D-Lubeck-Office	S	101701B-227	-	1	22433	581	605	-	-
0112727	WWK-G-LPWD-Well-F	S	101701B-228	-	1	10823	277	288	-	-
0112727 Dup	WWK-G-LPWD-Well-F	S	101701B-229	-	1	9954	254	264	-	-
XC101501-3	-	CS	101701B-230	250	-	8481	-	-	-	-
0112728	WWK-G-LPWD-Well-A	S	101701B-231	-	1	26028	675	703	-	-
0112728 Dup	WWK-G-LPWD-Well-A	S	101701B-232	-	1	25325	657	684	-	-
0112729	WWK-G-LPWD-Well-B	S	101701B-233	-	1	21537	558	581	-	-
0112729 Dup	WWK-G-LPWD-Well-B	S	101701B-234	-	1	21074	546	568	-	-
XC101501-4	-	CS	101701B-235	500	-	18440	-	-	-	-
XC101501-5	-	CS	101701B-236	1000	-	36595	-	-	-	-

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

Recovery (%) = [PFOA found (ng/L)-PFOA found in control (ng/L)] / amount PFOA added (ng/L) x 100

Standard Curve: Linear (1/x weighted)
Intercept = 276.530
Slope = 38.1239
Coef. Of Det. = 0.992303

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
NQ = Not quantifiable

Avg Resp. of the 250 ppt Standards (area): 9117
Response of the Check Standard (area): 9594
Percent Difference (%): -5.2
% difference acceptable if within ±15%

Spreadsheet prepared by: KR / 11/09/01

RJZ032619

EID185093

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: 008-046 Matrix: Water
Sponsor Study/Protocol No: NA
The samples listed below were removed from refrigerator No. 18
Time 1410 Date 10-17-01 Initials XZ

CLIENT SAMPLE ID	EXYGEN ID NUMBER	VOLUME (mL)	FORTIFICATION (ng)
na	0106020 Control	40.0	-
na	0106020 Spk A	40.0	2.0
na	0106020 Spk B	40.0	20.0
WWK-D-Bldg-293	0112725 Spk C	40.0	20.0
WWK-D-Lubeck-Office	0112726 Spk D	40.0	20.0
WWK-G-LPWD-Well-F	0112727 Spk E	40.0	20.0
WWK-G-LPWD-Well-A	0112728 Spk F	40.0	20.0
WWK-G-LPWD-Well-B	0112729 Spk G	40.0	20.0
WWK-D-Bldg-293	0112725	40.0	-
WWK-D-Bldg-293	0112725 Dup	40.0	-
WWK-D-Lubeck-Office	0112726	40.0	-
WWK-D-Lubeck-Office	0112726 Dup	40.0	-
WWK-G-LPWD-Well-F	0112727	40.0	-
WWK-G-LPWD-Well-F	0112727 Dup	40.0	-
WWK-G-LPWD-Well-A	0112728	40.0	-
WWK-G-LPWD-Well-A	0112728 Dup	40.0	-
WWK-G-LPWD-Well-B	0112729	40.0	-
WWK-G-LPWD-Well-B	0112729 Dup	40.0	-
-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
0106020 Spk A	F070901-3 (10 ng/mL)	200 µL (200 µL micropipet)	ZA 10/17/01
0106020 Spk B	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112725 Spk C	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112726 Spk D	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112727 Spk E	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112728 Spk F	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/
0112729 Spk G	F070901-2 (100 ng/mL)	200 µL (200 µL micropipet)	/

All samples were measured:
Time 1440 Date 10-17-01 Initials XZ

After measuring samples were returned to refrigerator No. 18
Time 1445 Date 10-17-01 Initials XZ

Comments: 200 µL of 250 mg/mL sodium thiosulfate was added to all samples before spiking. Initials/Date: XZ 10/17/01

Analysis Summary:
Data Set: 101701B Initials/Date: KGR / 10/18/01
Data Set: — Initials/Date: — / —
Data Set: — Initials/Date: — / —

Set extraction/analysis data verified by: JDF 10/19/01 Date: 10/19/01

July 26, 2001/6

RJZ032620

EID185094

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: 008-046 METHOD: $$ PROTOCOL NUMBER: NA
MATRIX: Water ANALYTES: PFOA

Client ID	Exygen ID	STEP 1	STEP 2	Dilutions (mL/mL)	STEP 3	Dilutions (mL/mL)	STEP 4	Reagents/ Materials	Lot #
na	0106020 Control			—		—	—	Methanol	41202
na	0106020 Spk A			—		—	—	C18 SPE	W1236B2
na	0106020 Spk B			—		—	—	Type I Water	NA
WWK-D-Bldg-293	0112725 Spk C			0.5/1.0		—	—	-	-
WWK-D-Lubeck-Office	0112726 Spk D					—	—	-	-
WWK-G-LPWD-Well-F	0112727 Spk E					—	—	-	-
WWK-G-LPWD-Well-A	0112728 Spk F					—	—	-	-
WWK-G-LPWD-Well-B	0112729 Spk G					—	—	-	-
WWK-D-Bldg-293	0112725			—		—	—	-	-
WWK-D-Bldg-293	0112725 Dup			—		—	—	*Initials/Date*	X2 10-17-01
WWK-D-Lubeck-Office	0112726			—		—	—	-	-
WWK-D-Lubeck-Office	0112726 Dup			—		—	—	-	-
WWK-G-LPWD-Well-F	0112727			—		—	—	-	-
WWK-G-LPWD-Well-F	0112727 Dup			—		—	—	-	-
WWK-G-LPWD-Well-A	0112728			—		—	—	-	-
WWK-G-LPWD-Well-A	0112728 Dup			—		—	—	HPLC	
WWK-G-LPWD-Well-B	0112729			—		—	—	Methanol	41025
WWK-G-LPWD-Well-B	0112729 Dup			—		—	—	Ammonium Acetate	TA2476
-	-	-	-	-	-	-	-	Type I water	NA
*Initials/Date	-	X2 10-17-01	X2 10-17-01	KJL 10/18/01	KJL 10/18/01	—	—	*Initials/Date*	KJL 10/18/01

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.

*Initials and date under each step indicates the personnel that performed this step.
$$ Method of Analysis for the Determination of Perfluorooctanoic Acid (PFOA) in Water

COMMENTS:

Final extracts stored in refrigerator 15 Initials: KJL Date: 10/17/01

July 19, 2001/4

Masslynx - Sample List

Sample List: C:\MASSLYNX\008-046.PRO\SampleDB\101701B.SPL
Printed: Thu Oct 18 07:07:13 2001 KJL 10/18/01

Exygen STUDY NO. 008-046

	Vial	File Name	LIMS ID	Client ID	Sample Description	Matrix	Sample Type	Conc (ppt)	Conc B	Conc C	Test ID	DF	MS Method
1	1	101701B-201	---	---	XC101501-0, 0 ppt standard	---	Blank	0	---	---	0	1	PFOA
2	2	101701B-202	---	---	XC101501-1, 50 ppt standard	---	Standard	50	---	---	0	1	PFOA
3	3	101701B-203	---	---	XC101501-2, 100 ppt standard	---	Standard	100	---	---	0	1	PFOA
4	4	101701B-204	---	---	XC101501-3, 250 ppt standard	---	Standard	250	---	---	0	1	PFOA
5	5	101701B-205	---	---	XC101501-4, 500 ppt standard	---	Standard	500	---	---	0	1	PFOA
6	6	101701B-206	---	---	XC101501-5, 1000 ppt standard	---	Standard	1000	---	---	0	1	PFOA
7	7	101701B-207	---	---	XC101501-6, 250 ppt check standard	---	QC	250	---	---	0	1	PFOA
8	92	101701B-208	---	---	Methanol Wash	---	Blank	---	---	---	0	1	PFOA
9	41	101701B-209	---	---	0106020 Control	---	Blank	---	---	---	0	1	PFOA
10	42	101701B-210	---	---	0106020 Spk A, 50 ppt	---	QC	50	---	---	0	1	PFOA
11	43	101701B-211	---	---	0106020 Spk B, 500 ppt	---	QC	500	---	---	0	1	PFOA
12	44	101701B-212	---	---	0112725 Spk C, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
13	45	101701B-213	---	---	0112725 Spk C, 500 ppt	---	QC	500	---	---	0	1	PFOA
14	46	101701B-214	---	---	0112726 Spk D, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
15	47	101701B-215	---	---	0112726 Spk D, 500 ppt	---	QC	500	---	---	0	1	PFOA
16	2	101701B-216	---	---	XC101501-1, 50 ppt standard	---	Standard	50	---	---	0	1	PFOA
17	48	101701B-217	---	---	0112727 Spk E, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
18	49	101701B-218	---	---	0112727 Spk E, 500 ppt	---	QC	500	---	---	0	1	PFOA
19	50	101701B-219	---	---	0112728 Spk F, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
20	51	101701B-220	---	---	0112728 Spk F, 500 ppt	---	QC	500	---	---	0	1	PFOA
21	52	101701B-221	---	---	0112729 Spk G, 500 ppt, DF=2	---	QC	250	---	---	0	2	PFOA
22	53	101701B-222	---	---	0112729 Spk G, 500 ppt	---	QC	500	---	---	0	1	PFOA
23	3	101701B-223	---	---	XC101501-2, 100 ppt standard	---	Standard	100	---	---	0	1	PFOA
24	54	101701B-224	---	---	0112725	---	Analyte	---	---	---	0	1	PFOA
25	55	101701B-225	---	---	0112725 Dup	---	Analyte	---	---	---	0	1	PFOA
26	56	101701B-226	---	---	0112726	---	Analyte	---	---	---	0	1	PFOA
27	57	101701B-227	---	---	0112726 Dup	---	Analyte	---	---	---	0	1	PFOA
28	58	101701B-228	---	---	0112727	---	Analyte	---	---	---	0	1	PFOA
29	59	101701B-229	---	---	0112727 Dup	---	Analyte	---	---	---	0	1	PFOA
30	4	101701B-230	---	---	XC101501-3, 250 ppt standard	---	Standard	250	---	---	0	1	PFOA
31	60	101701B-231	---	---	0112728	---	Analyte	---	---	---	0	1	PFOA
32	61	101701B-232	---	---	0112728 Dup	---	Analyte	---	---	---	0	1	PFOA
33	62	101701B-233	---	---	0112729	---	Analyte	---	---	---	0	1	PFOA
34	63	101701B-234	---	---	0112729 Dup	---	Analyte	---	---	---	0	1	PFOA
35	5	101701B-235	---	---	XC101501-4, 500 ppt standard	---	Standard	500	---	---	0	1	PFOA
36	6	101701B-236	---	---	XC101501-5, 1000 ppt standard	---	Standard	1000	---	---	0	1	PFOA

Sample List: C:\MASSLYNX\008-046.PRO\SampleDB\101701B.SPL
Printed: Thu Oct 18 07:07:13 2001 EJL 10/18/01

Exygen STUDY NO. 008-046

	HPLC Method	MS Tune File	Inj Volume (µL)
1	water	PFOA WATER	15
2	water	PFOA WATER	15
3	water	PFOA WATER	15
4	water	PFOA WATER	15
5	water	PFOA WATER	15
6	water	PFOA WATER	15
7	water	PFOA WATER	15
8	water	PFOA WATER	15
9	water	PFOA WATER	15
10	water	PFOA WATER	15
11	water	PFOA WATER	15
12	water	PFOA WATER	15
13	water	PFOA WATER	15
14	water	PFOA WATER	15
15	water	PFOA WATER	15
16	water	PFOA WATER	15
17	water	PFOA WATER	15
18	water	PFOA WATER	15
19	water	PFOA WATER	15
20	water	PFOA WATER	15
21	water	PFOA WATER	15
22	water	PFOA WATER	15
23	water	PFOA WATER	15
24	water	PFOA WATER	15
25	water	PFOA WATER	15
26	water	PFOA WATER	15
27	water	PFOA WATER	15
28	water	PFOA WATER	15
29	water	PFOA WATER	15
30	water	PFOA WATER	15
31	water	PFOA WATER	15
32	water	PFOA WATER	15
33	water	PFOA WATER	15
34	water	PFOA WATER	15
35	water	PFOA WATER	15
36	water	PFOA WATER	15

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Sponsor Protocol No: NA | Exygen Study No: 008-046

Instrument: Micromass Quattro Ultima (LC/MS/MS Unit #7)

ELECTROSPRAY ION SOURCE:

Capillary: 3.0 kV	Hexapole 2: 0 V
Hexapole 1: 0 V	Source Block Temp.: 100°C
Aperture 1: 0 V	Desolvation Temp.: 300°C

ANALYZER:

LM Res 1: 10.5 V	LM Res 2: 13.0 V
HM Res 1: 10.5 V	HM Res 2: 13.0 V
IEnergy 1: 1.0 V	IEnergy 2: 2.0 V
Entrance: -2 V	Multiplier: 700 V
Exit: 2 V	

GAS FLOWS AND PRESSURE:

Desolvation N_2 Flow Rate: ~650 L/hr
Nebuliser N_2 Flow Rate: ~150 L/hr
Gas Cell Pressure: ~0.003 mbar

Computer: COMPAQ Professional Workstation AP200

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

HPLC Equipment: Hewlett Packard (HP) Series 1100

HP 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: 37A)
(JONESCHROMATOGRAPHY: Part No. FK5962E)

Column Temperature: 35°C
Mobile Phase (A) : 2 mM Ammonium Acetate in Type I Water
Mobile Phase (B) : Methanol

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

Total run time = 14 min

Injected Volume: 15 µL

Ions monitored :

Analyte	Parent ion	Daughter ion	Dwell (secs)	Coll Energy (eV)	Cone (V)
PFOA	413	369	0.2	10	50

Analyst: Karen Risha
Exygen Research
3058 Research Drive, State College, PA 16801 KRR 10/18/01
Phone: (814) 272-1039 FAX: (814) 231-1580

All Handwritten Peak ID's by: KRR 10/19/01

NOTE: THE NEXT 3 PAGES ARE MASSLYNX PRINTOUTS FROM THE SYSTEM AND CAN BE USED TO VERIFY VALUES LISTED ON THIS PAGE. KRR 11/09/01

Scanning Method Report

Method: C:\MASSLYNX\008-046.PRO\ACQUDB\PFOA
Last Modified: Mon Oct 15 12:37:26 2001

Printed: Fri Nov 09 09:41:26 2001

KJL 11/09/01

Solvent Delay (mins) : 1.00

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

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 8.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	369.00	0.20	10	50

Method File: C:\MASSLYNX\008-046.PRO\ACQUDB\water
Last Modified: Friday, November 09, 2001 09:38:47

Printed: Friday, November 09, 2001 09:42:01 KJL 11/09/01

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)	14.0
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 9 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

HP1100 LC Pump External Event Timetable

The Timetable contains 4 entries which are :

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

HP1100 Autosampler Initial Conditions

Draw Speed	200.0
Eject Speed (µl/min)	200
Draw Position (mm)	0.10
Stop Time (mins)	14.00
Injection Volume(µl)	15.0
Vial Number	6

Exygen RESEARCH
Precise Research.
Proven Results.

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

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

KJC 11/09/01

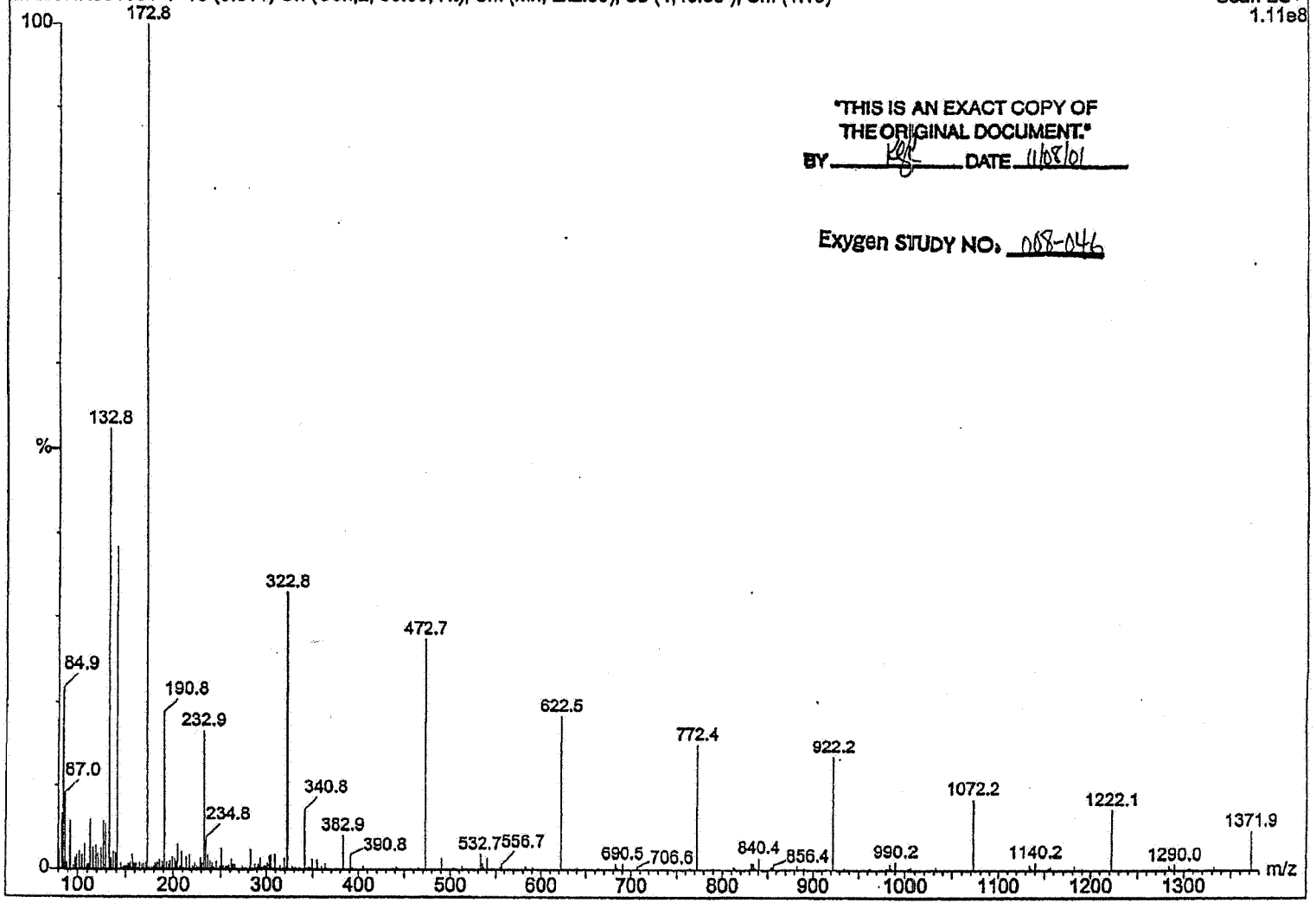
3058 Research Drive
State College, PA 16801, USA
T: 814.272.1039
F: 814.231.1580
exygen.com

Calibration Check of NaIRbCs090800 on MS1 using NaIRbCs tuning solution

KJ 09/10/01

LC/MS/MS #7
10-Sep-2001 08:53:03
Scan ES+
1.11e8

CALCHK091001-1 13 (0.811) Cn (Cen,2, 80.00, Ht); Sm (Mn, 2x2.00); Sb (1,40.00); Cm (1:16)



"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT."
BY KJ DATE 11/08/01

Exygen STUDY NO. 008-046

EID185102

RJZ032628

Calibration Check of NaIRbCs090800 on MS2 using NaIRbCs tuning solution

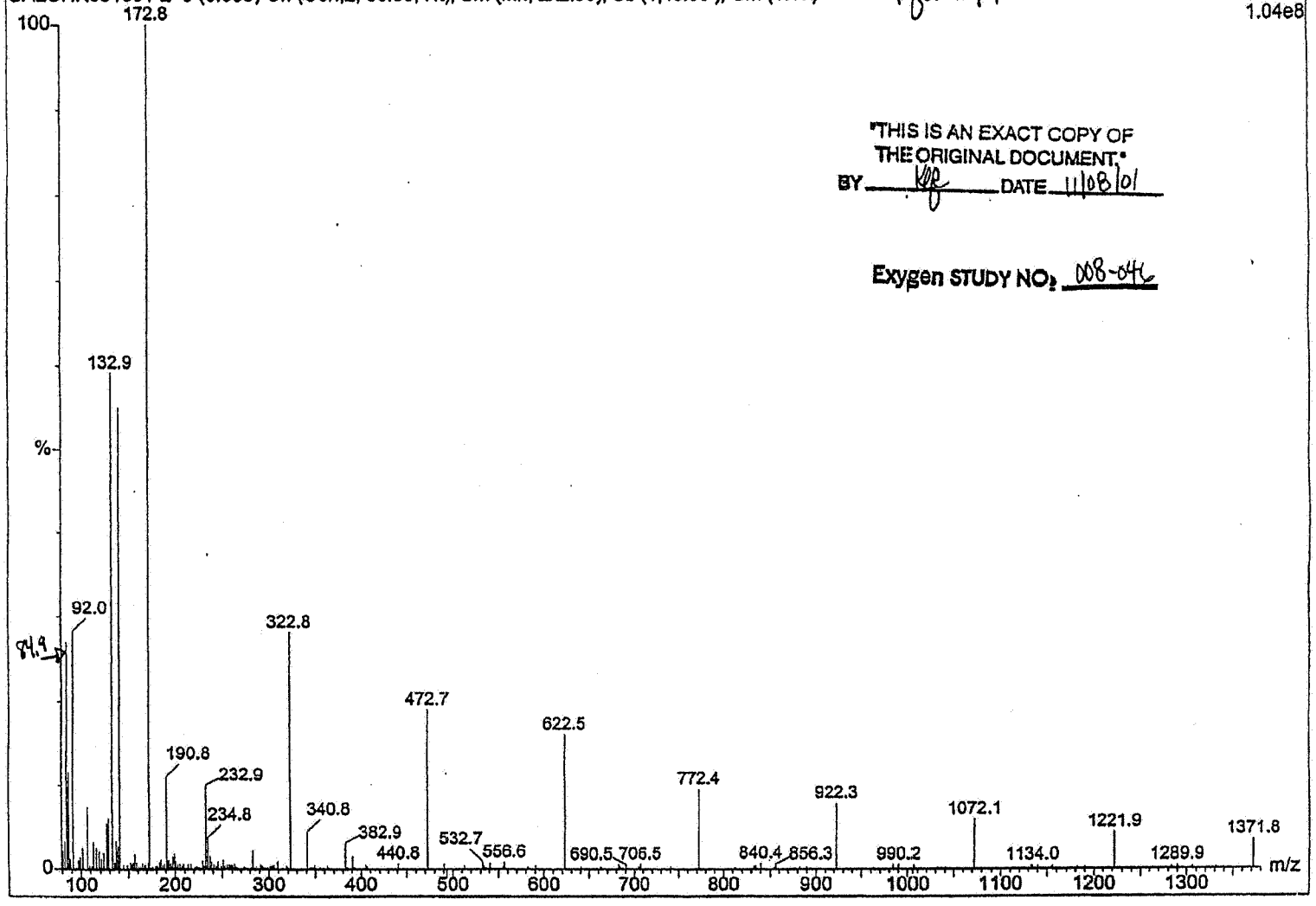
LC/MS/MS #7
10-Sep-2001 08:55:20
MS2 ES+
1.04e8

CALCHK091001-2 9 (0.569) Cn (Cen,2, 80.00, Ht); Sm (Mn, 2x2.00); Sb (1,40.00); Cm (1:17)

KGR 09/10/01

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT."
BY KGR DATE 11/08/01

Exygen STUDY NO. 008-044



EID185103

RJZ032629

Initial Calibration Curve

Exygen Study No.: 008-046
Set No: 101701B
Extraction Date: 10/17/01
Analyzed on: 10/18/01

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
XC101501-0	0	0
XC101501-1	50	2269
XC101501-2	100	4485
XC101501-3	250	9753
XC101501-4	500	20662
XC101501-5	1000	40877

Coefficient of Determination: 0.99963071

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.99981534
R Square	0.99963071
Adjusted R Squa	0.99953839
Standard Error	333.267525
Observations	6

KGF 11/09/01

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1202591346	1.2E+09	10827.597	5.1147E-08
Residual	4	444268.9724	111067.2		
Total	5	1203035615			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	95.7073733	184.143363	0.519744	0.6306791	-415.557624	606.972371	-415.55762	606.972371
X Variable 1	40.7746083	0.391853524	104.0557	5.115E-08	39.6866462	41.8625703	39.6866462	41.8625703

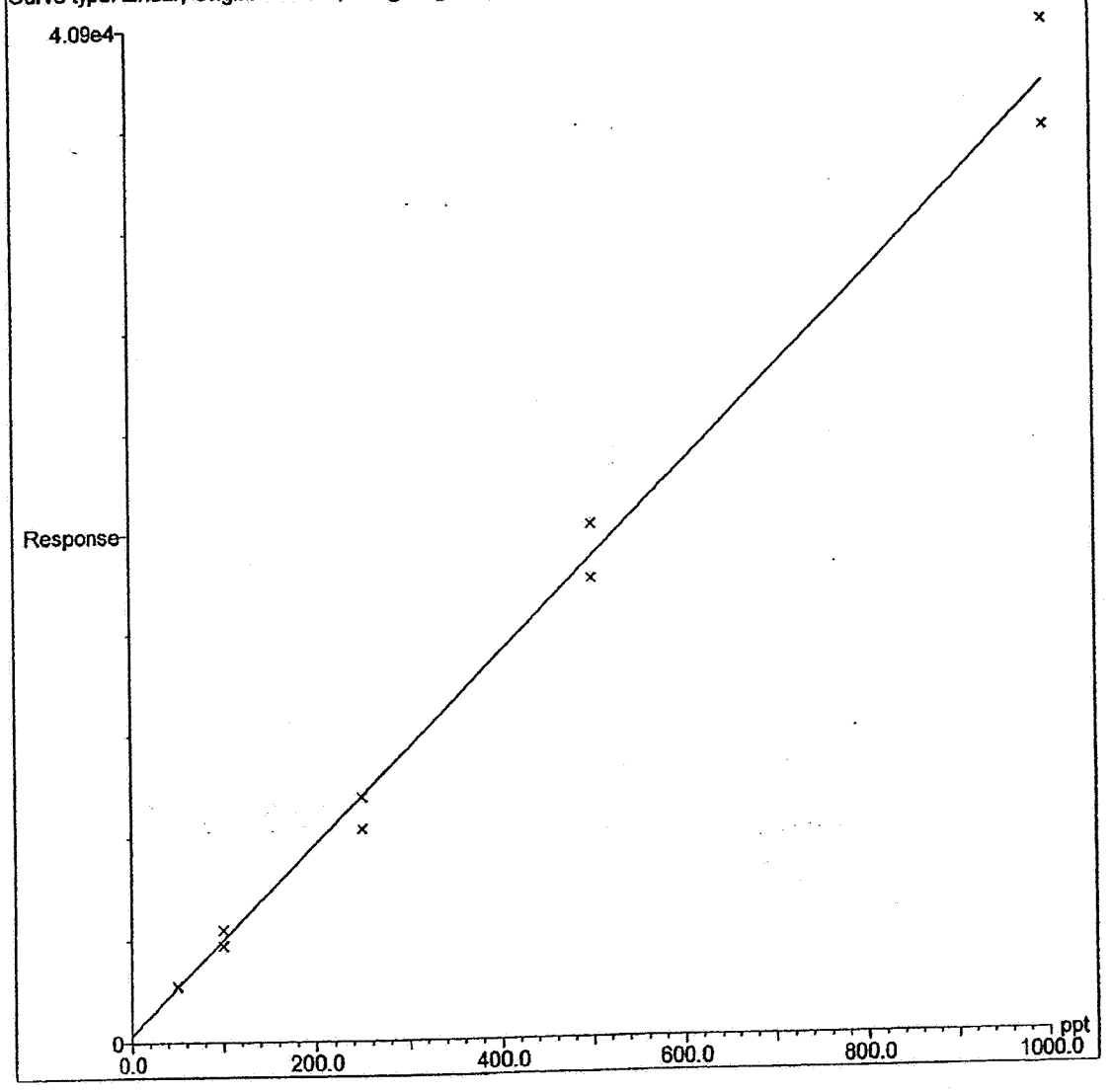
RJZ032630

EID185104

Quantify Calibration Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Calibration: P:\Data\LCMSMS7\Masslynx\008-046.PRO\CurveDB\101701B 10/19/01
Last modified: Fri Oct 19 07:08:10 2001
Printed: Fri Oct 19 07:15:14 2001

Compound 1 name: PFOA
Coefficient of Determination: 0.992303
Calibration curve: 38.1239 * x + 276.530
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

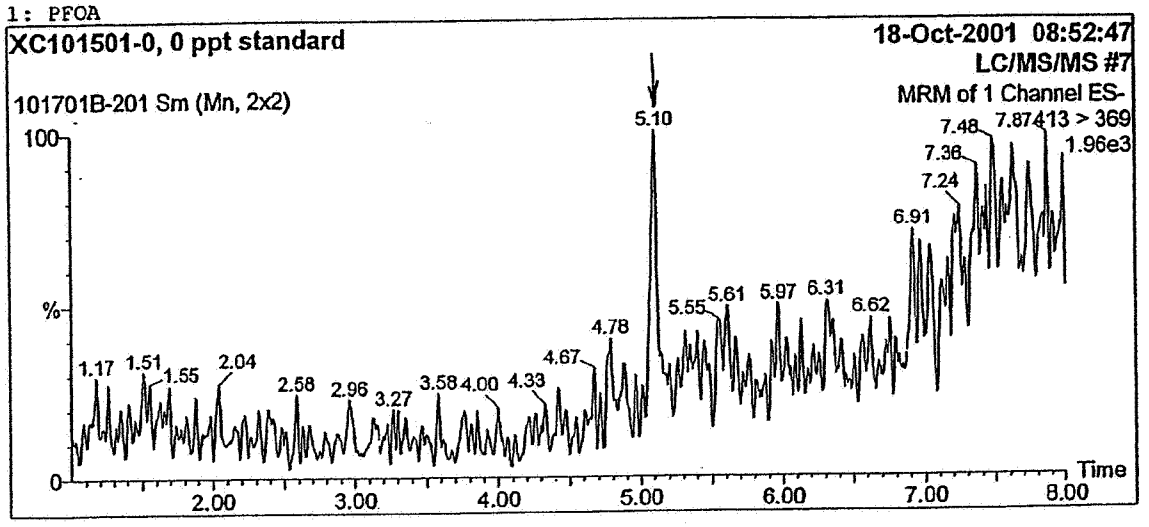
Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Initials KR
Date 10/19/01
Run# 101701B-201 To 101701B-236

Name: 101701B-201
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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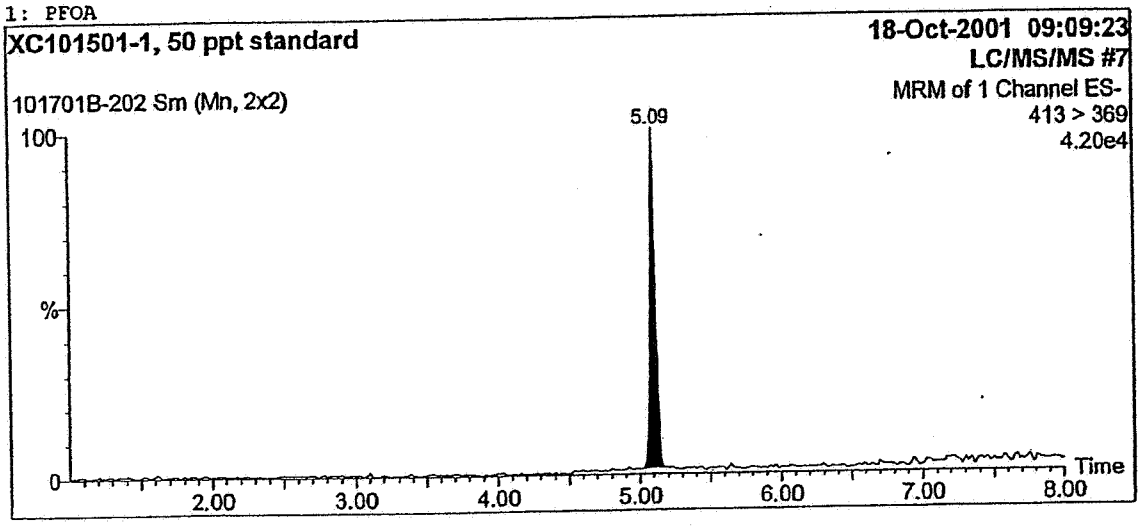
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-202
Text:

1: PFOA



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Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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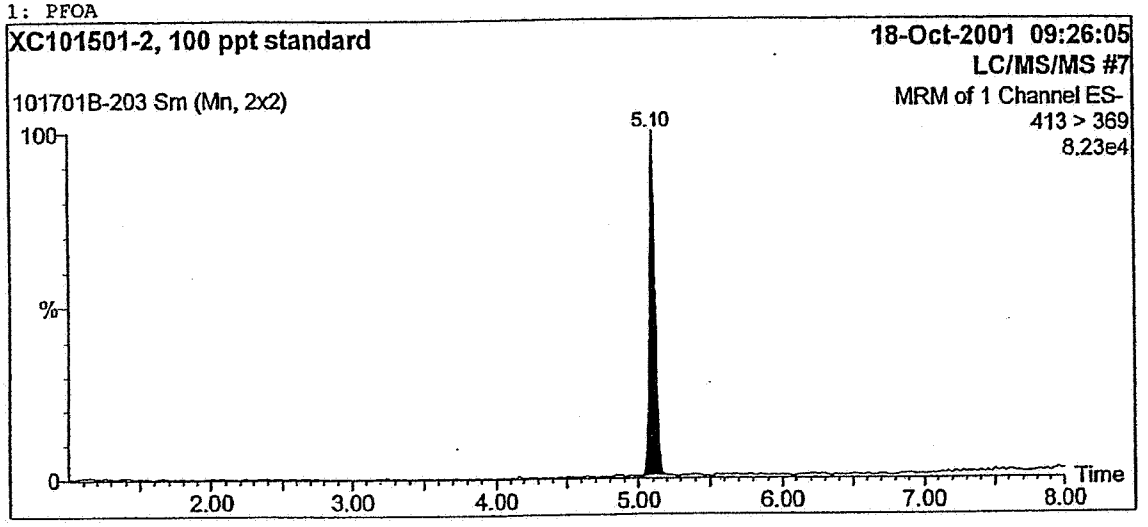
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-203
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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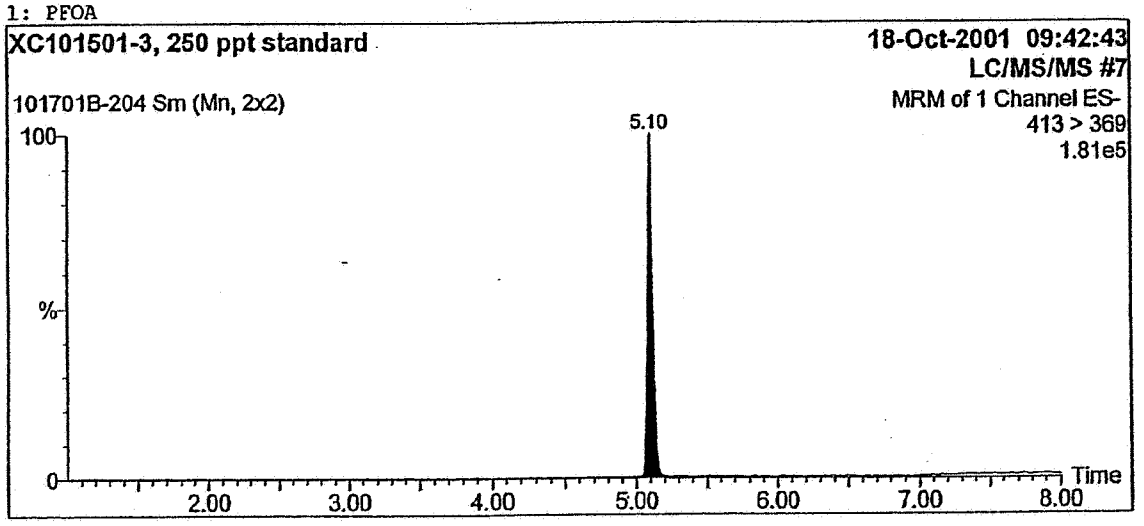
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-204
Text:

1: PFOA



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Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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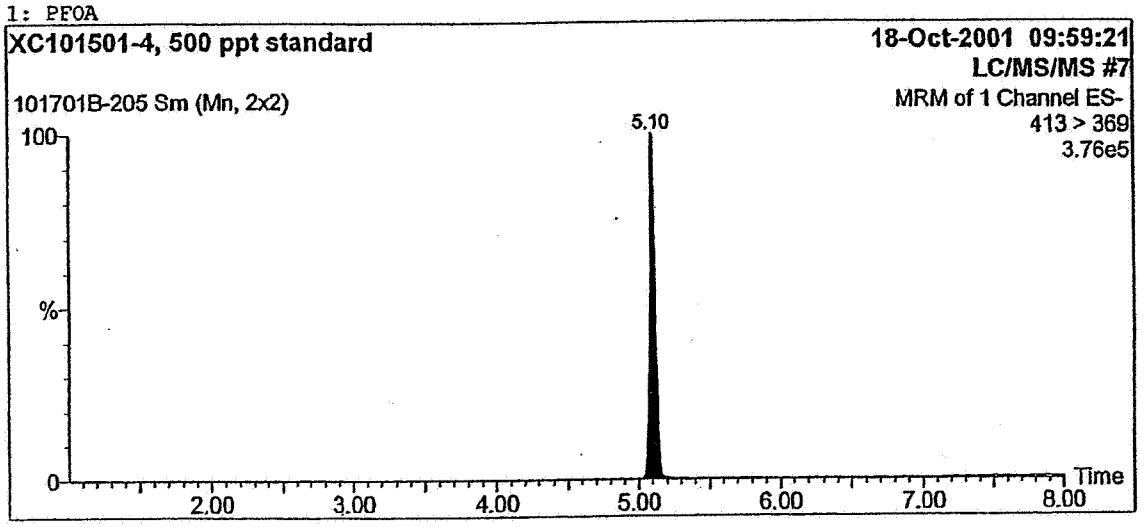
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-205
Text:

1: PFOA



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Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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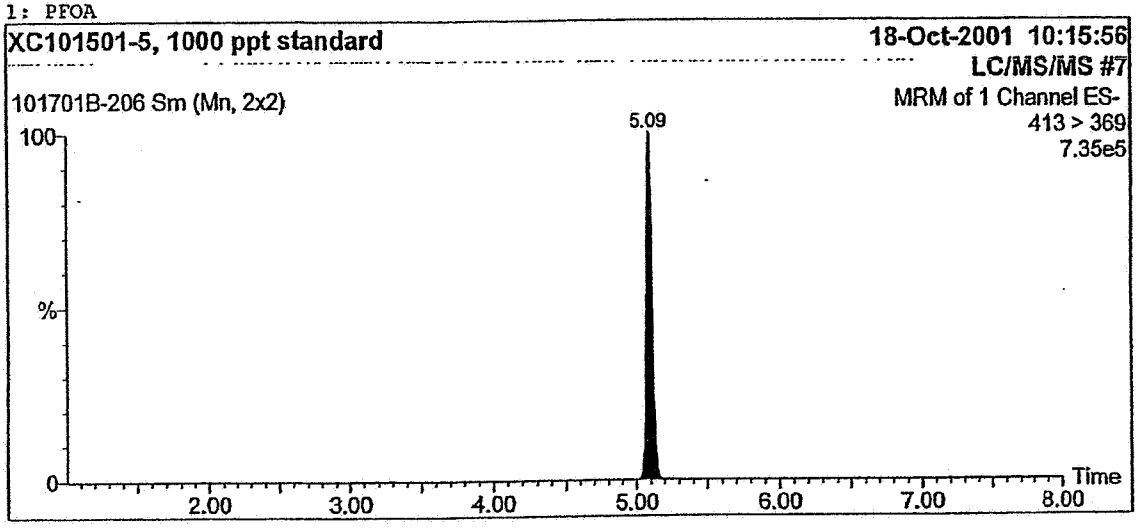
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-206
Text:

1: PFOA



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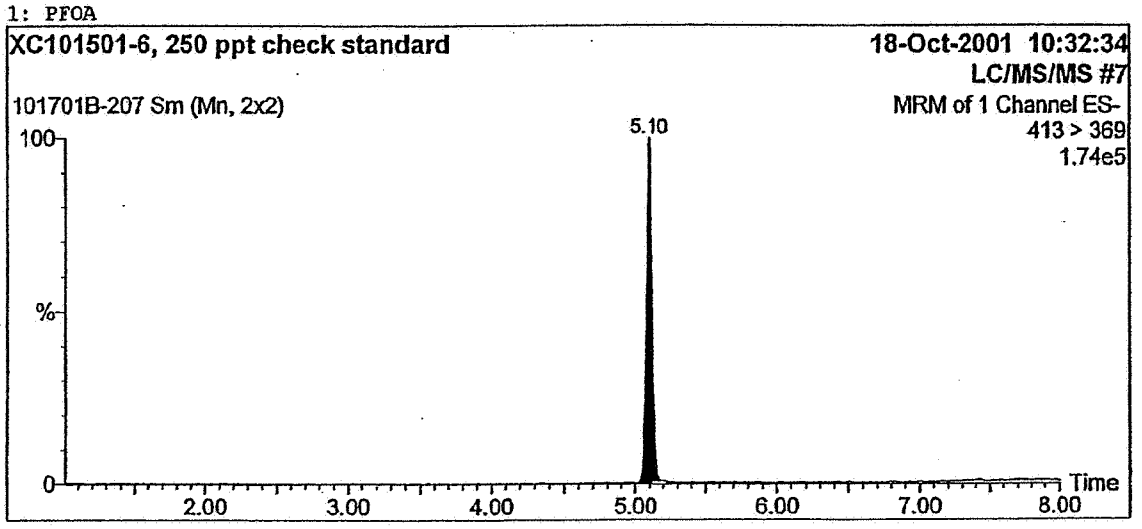
Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-207
Text:

1: PFOA



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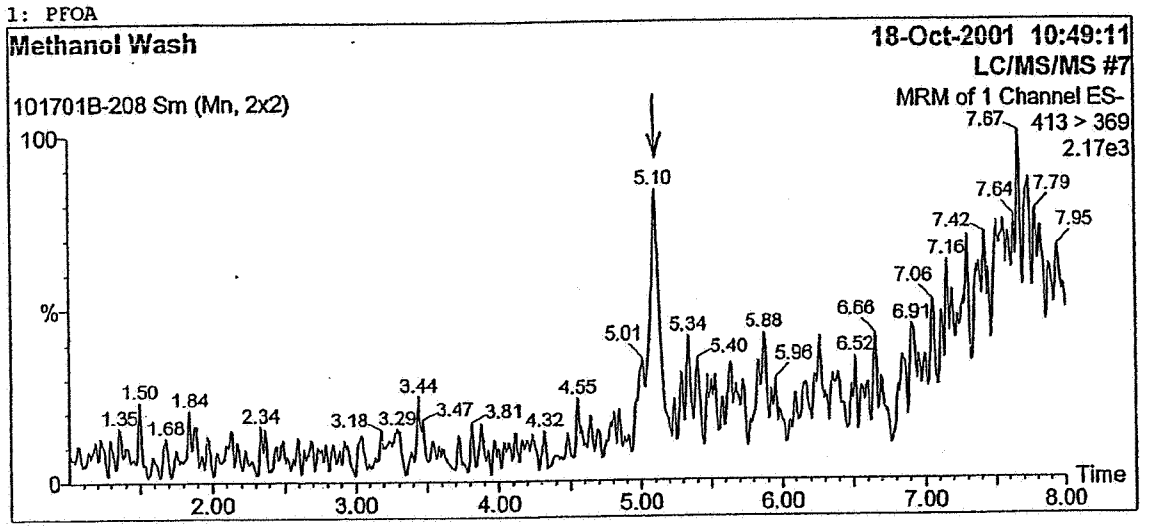
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-208
Text:

1: PFOA



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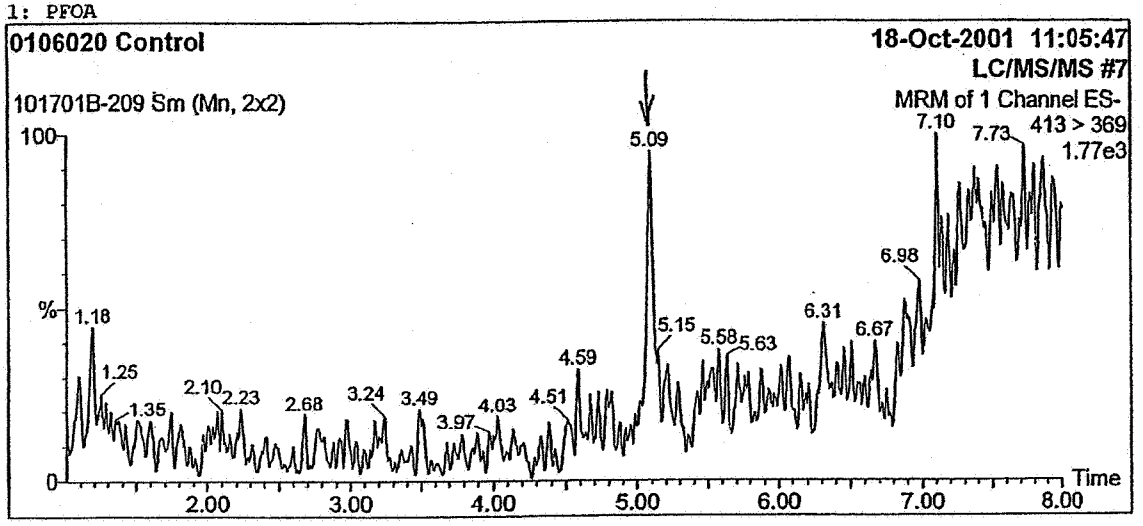
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-209
Text:

1: PFOA



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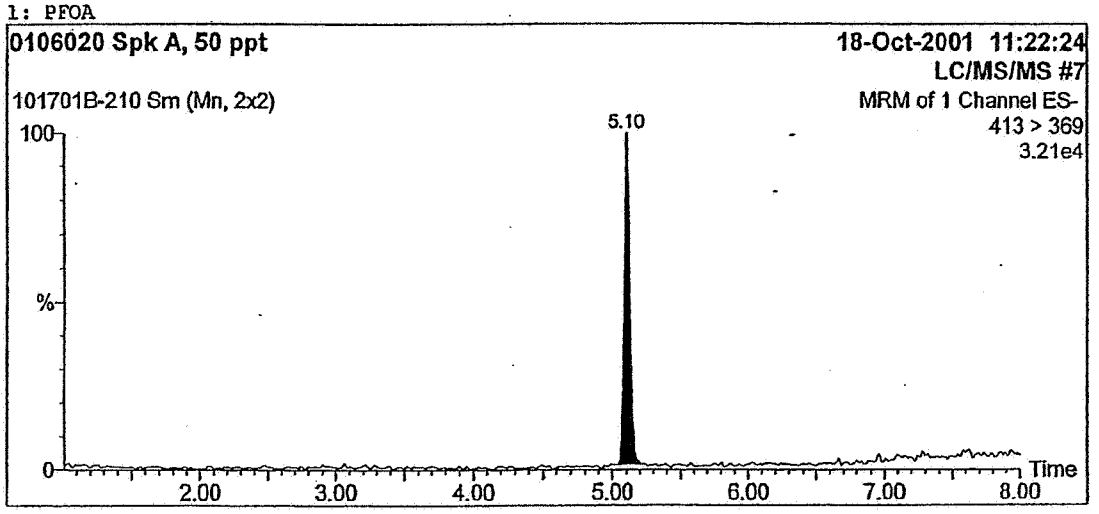
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-210
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
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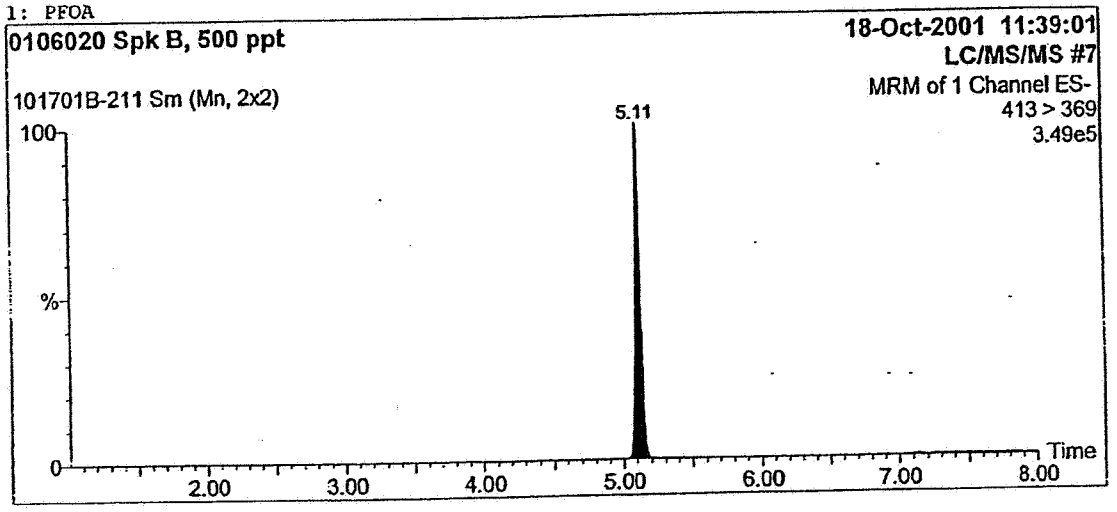
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-211
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-212
Text:

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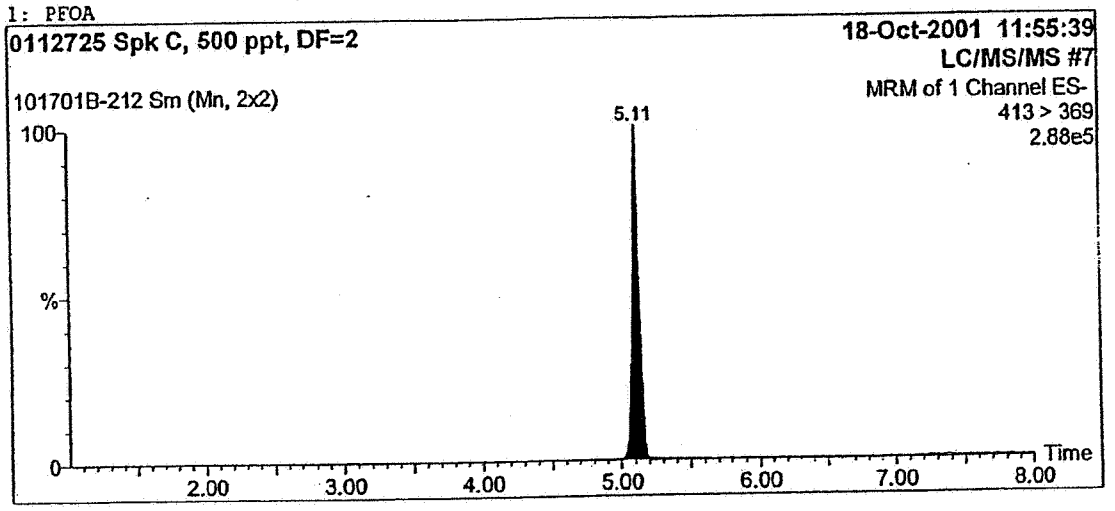

Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-213
Text:

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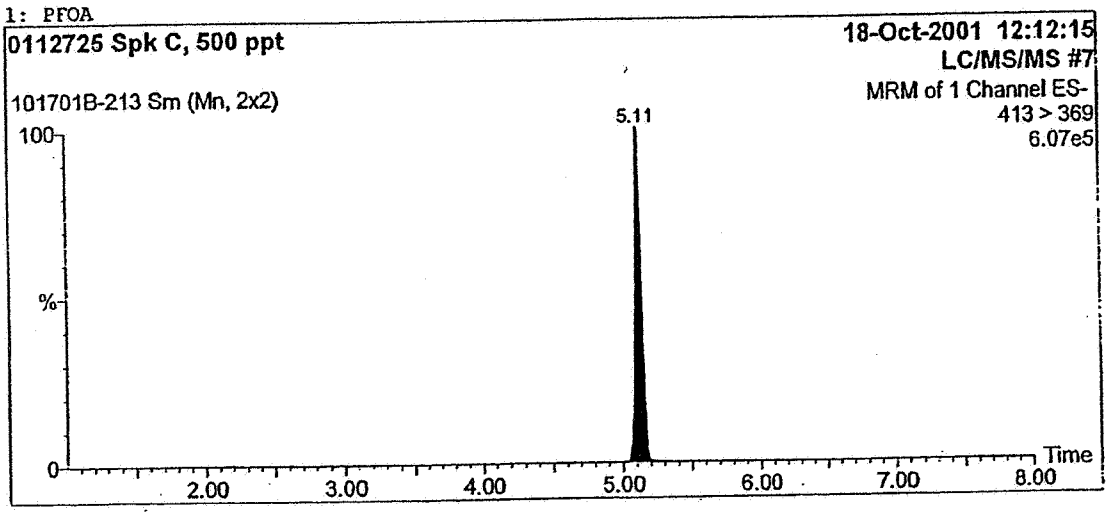

Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-214
Text:

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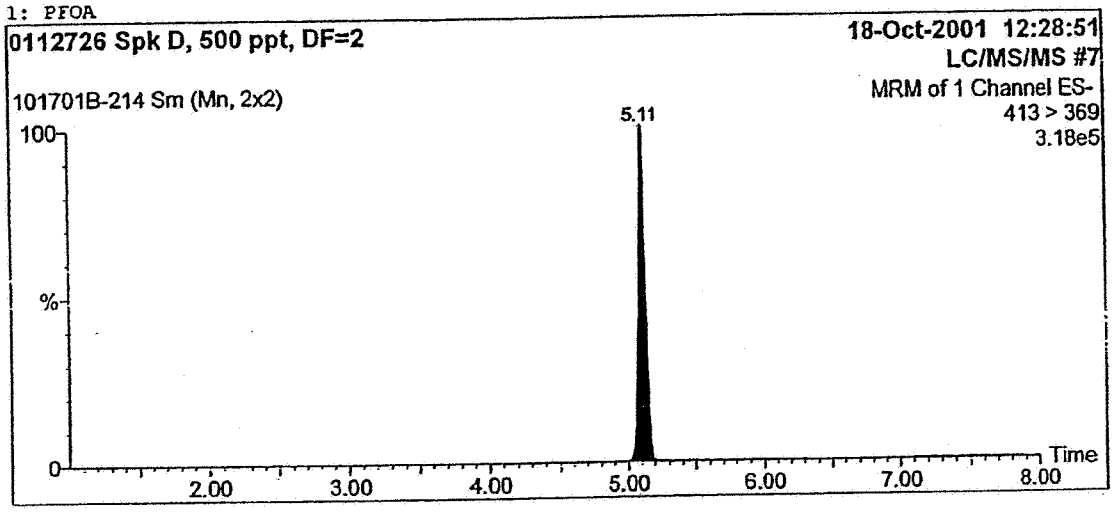


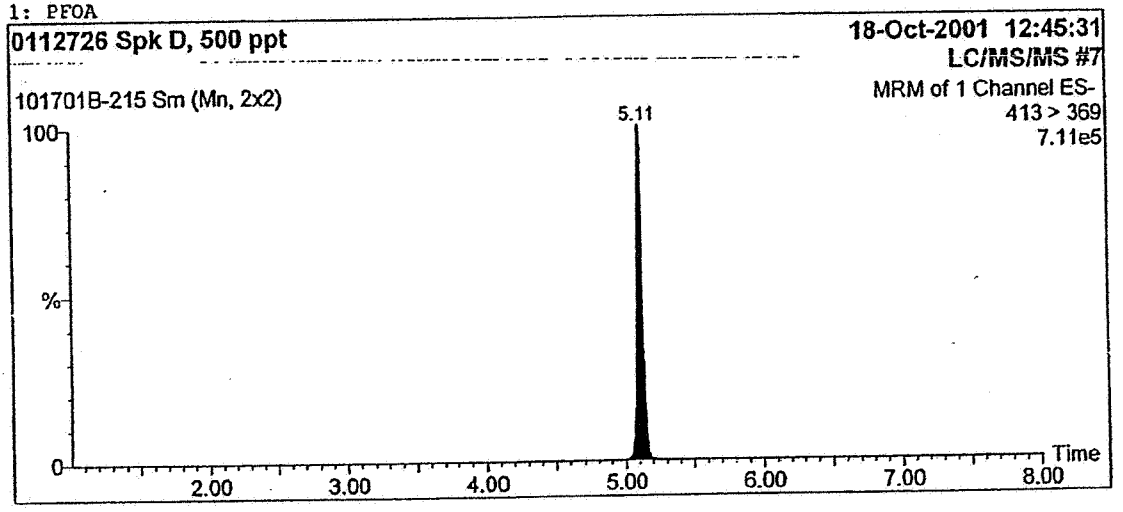
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-215
Text:

1: PFOA



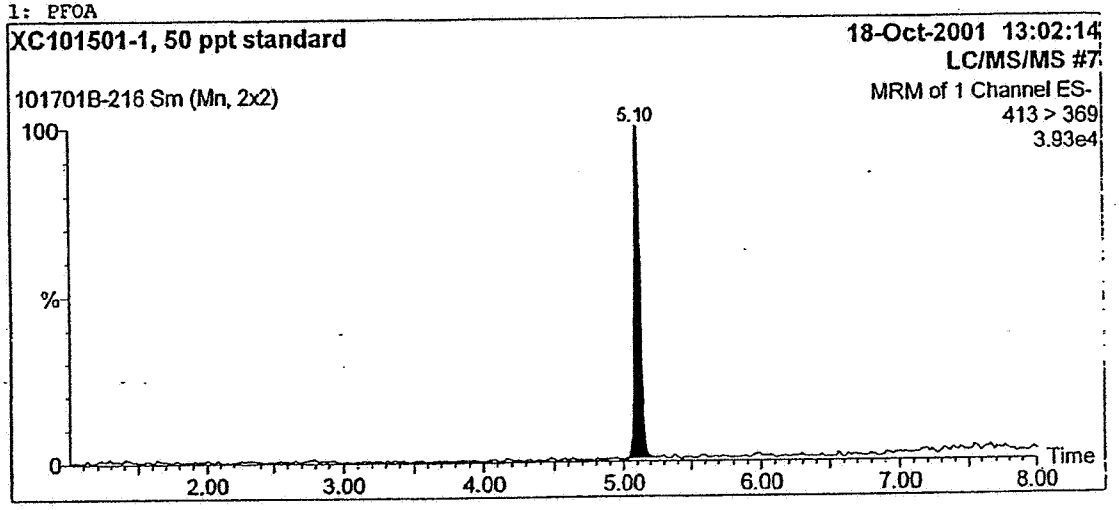
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-216
Text:

1: PFOA



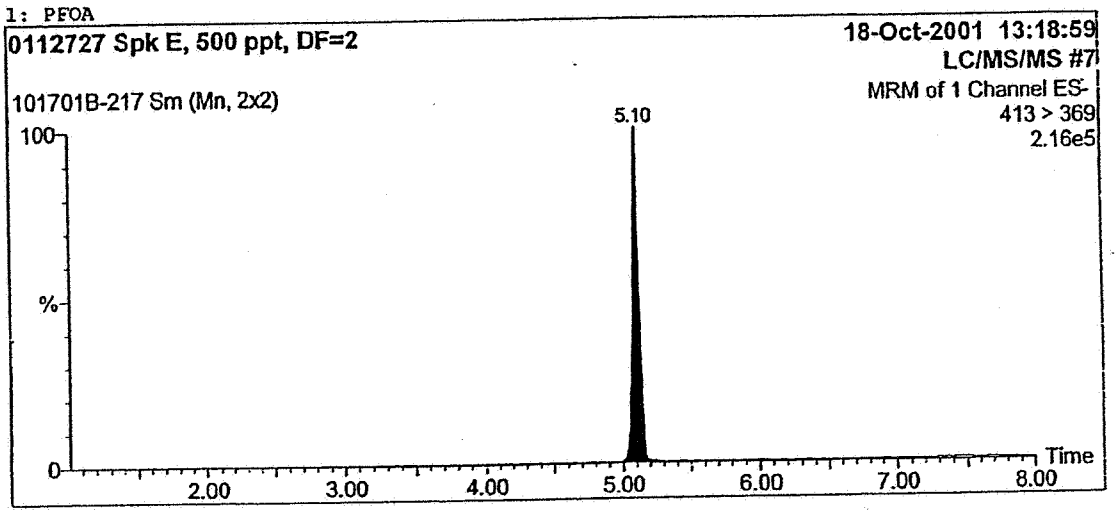
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-217
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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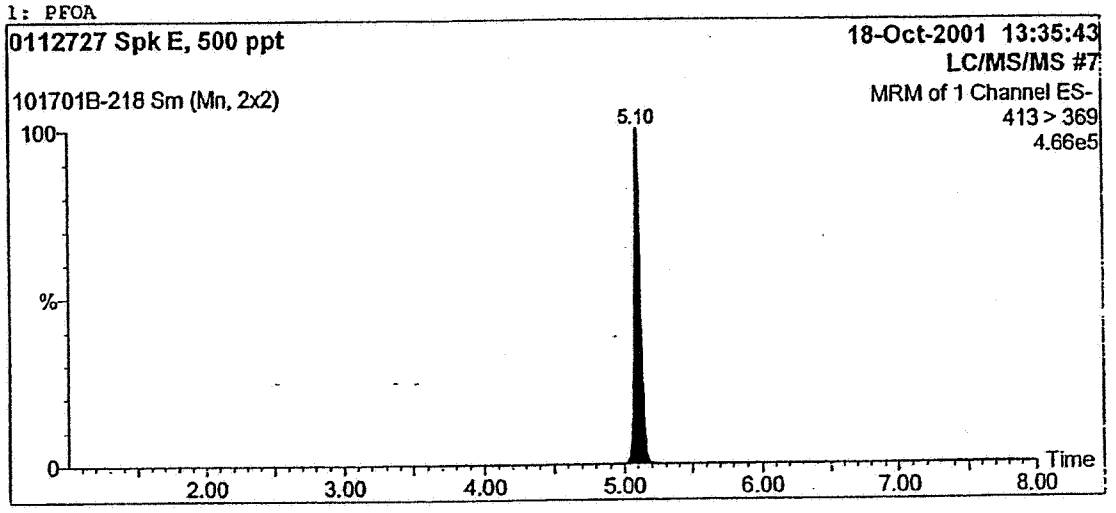
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-218
Text:

1: PFOA



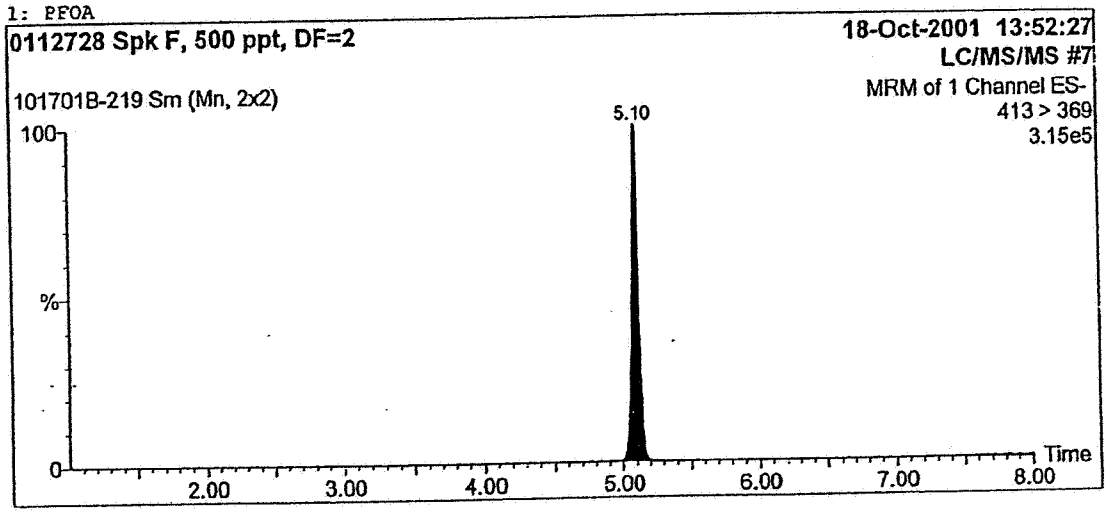
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-219
Text:

1: PFOA



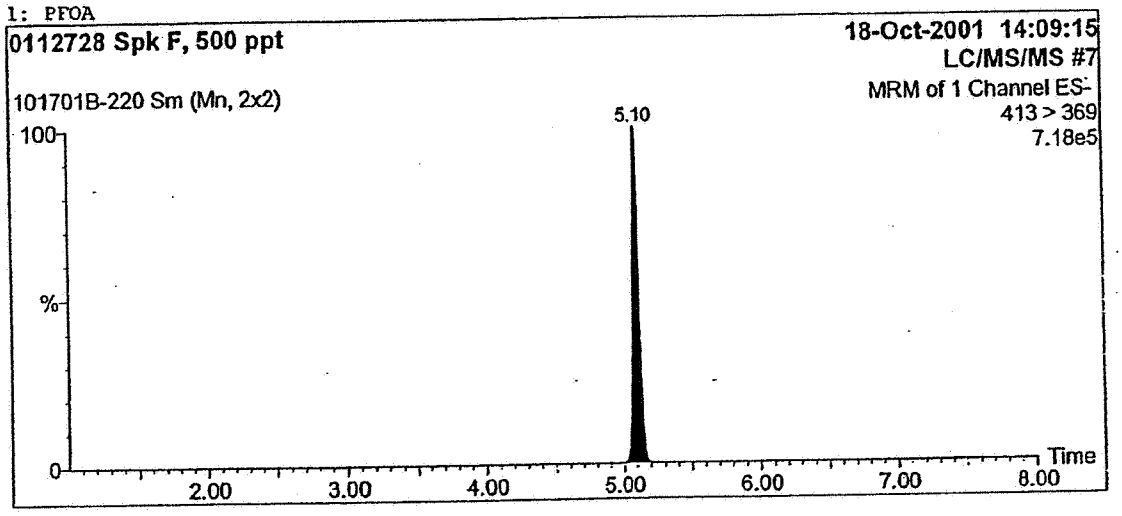
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-220
Text:

1: PFOA



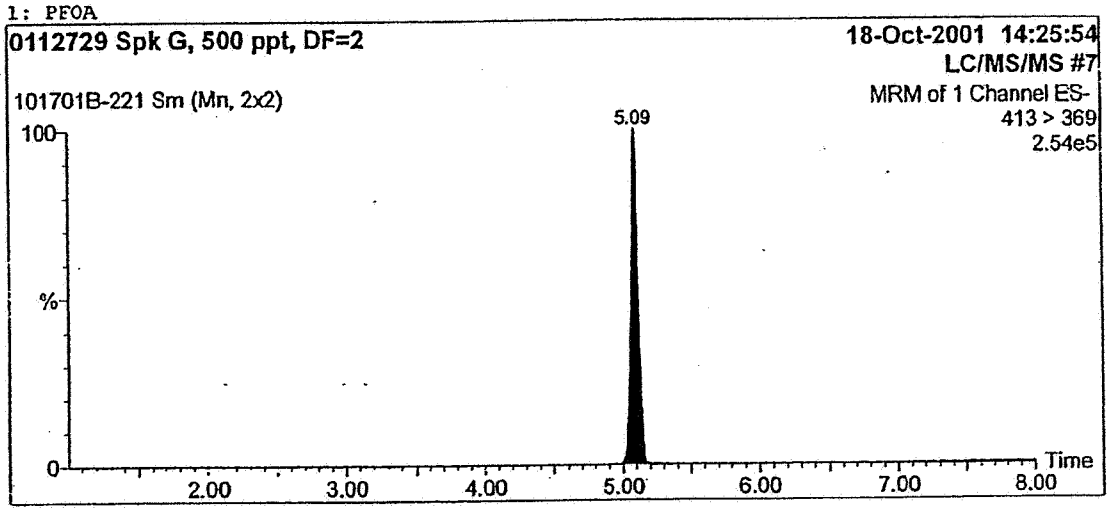
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-221
Text:

1: PFOA



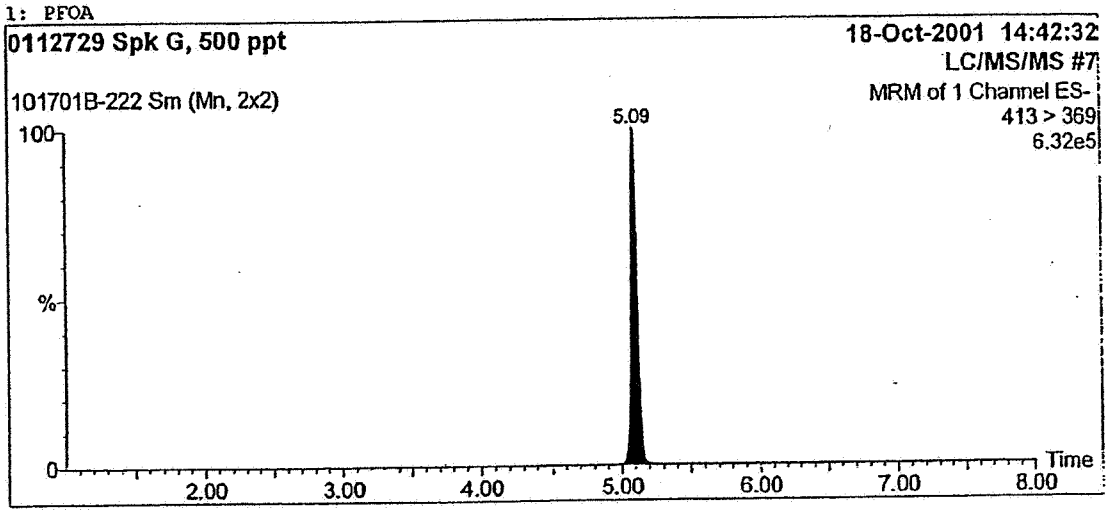
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-222
Text:

1: PFOA



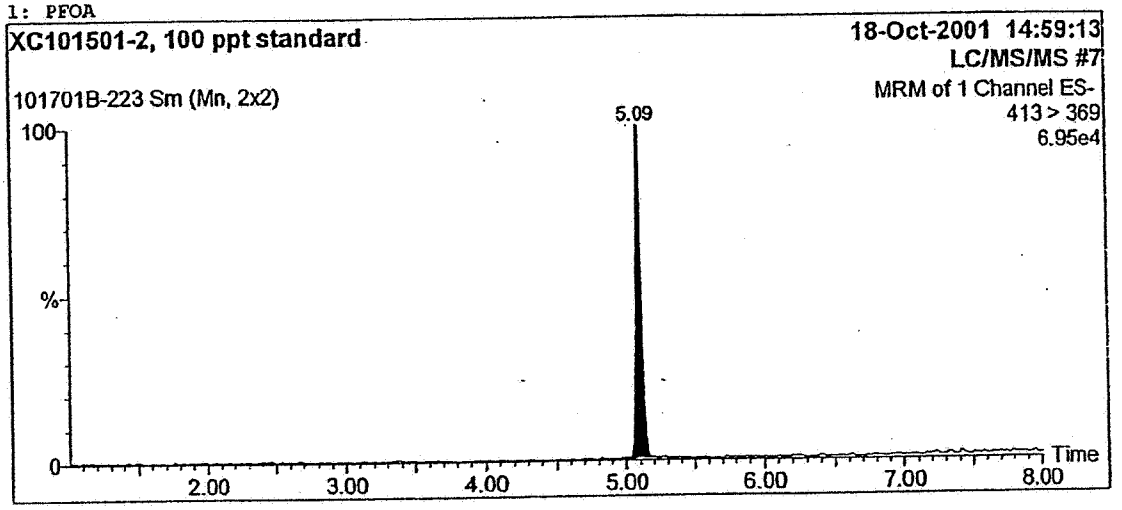
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-223
Text:

1: PFOA



Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
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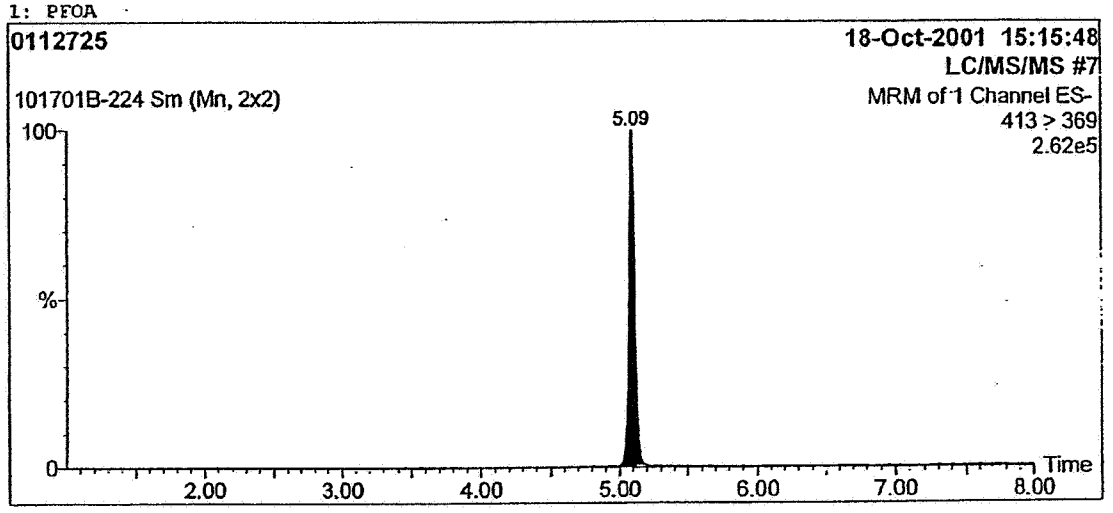
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-224
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
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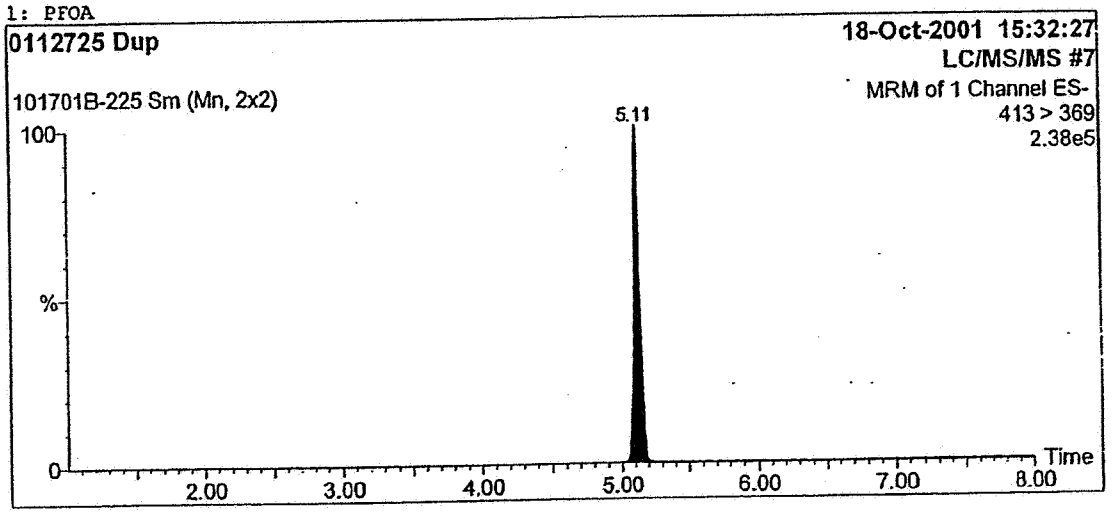
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-225
Text:

1: PFOA



Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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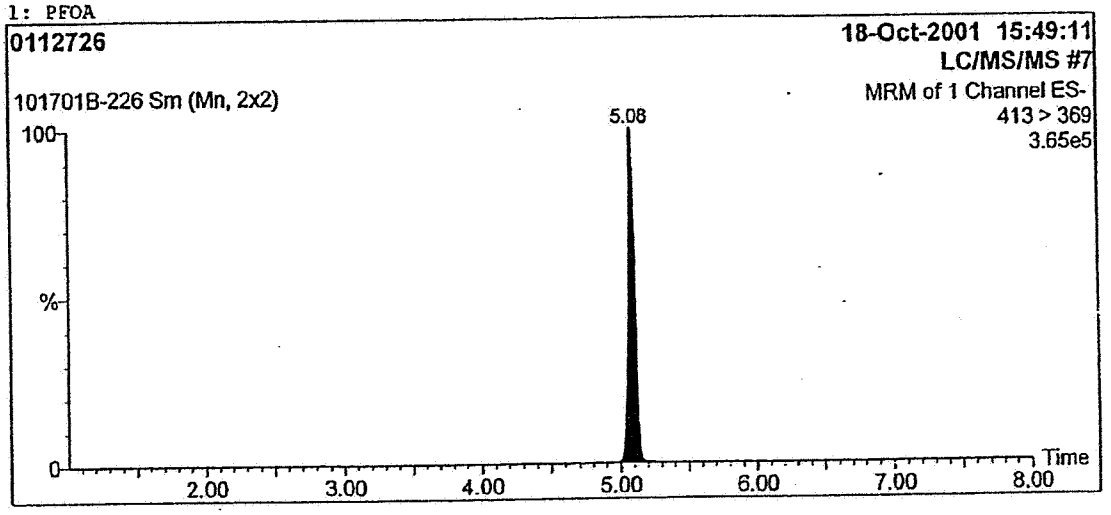
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-226
Text:

1: PFOA



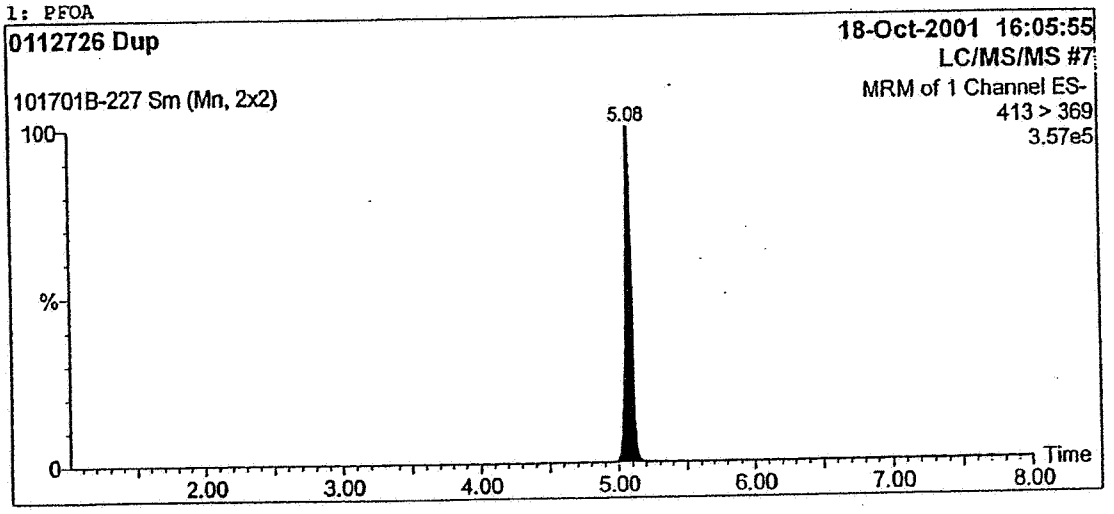
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-227
Text:

1: PFOA



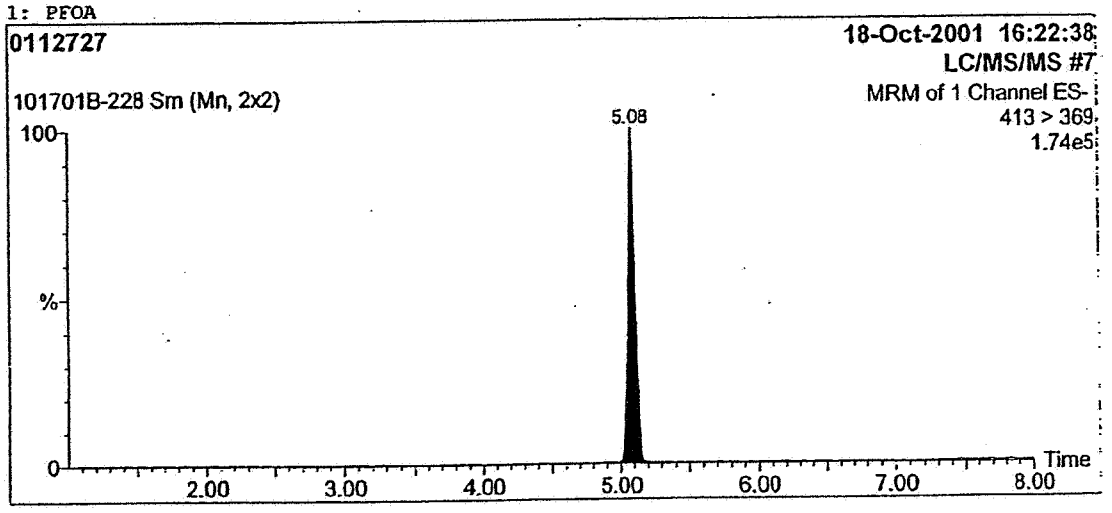
Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-228
Text:

1: PFOA



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Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
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Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

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Text:

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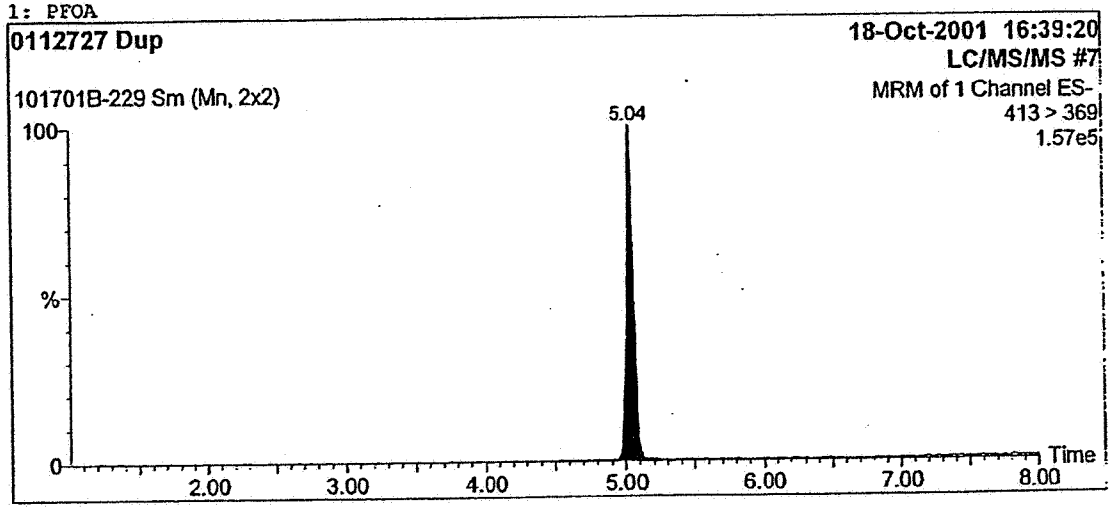

Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-230
Text:

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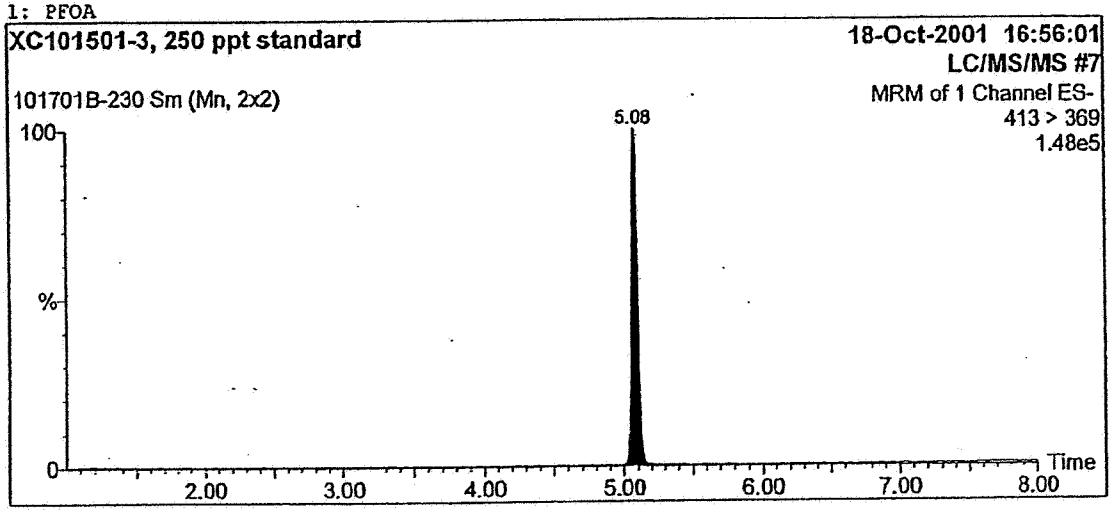

Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

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Last modified: Fri Oct 19 07:07:33 2001
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Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-231
Text:

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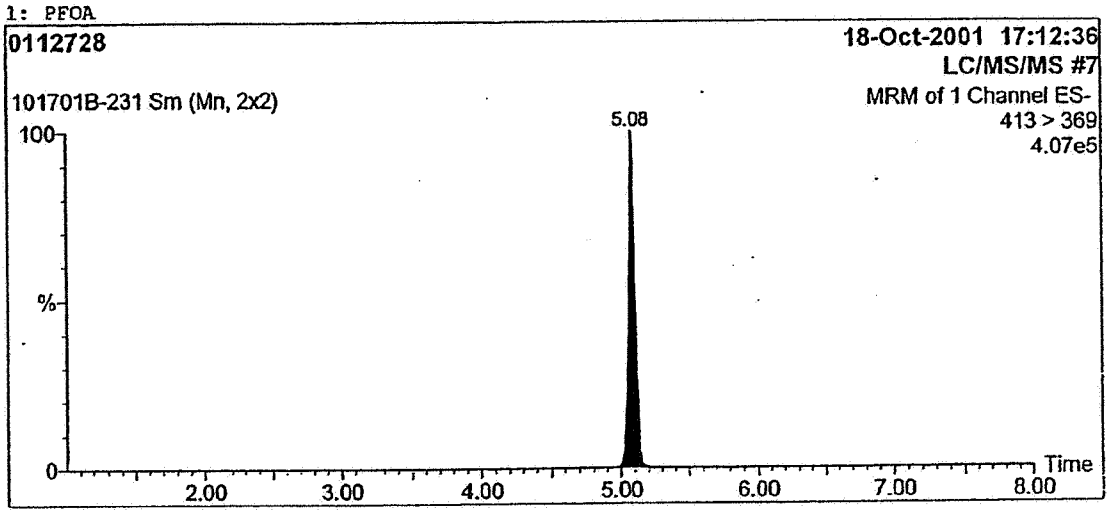

Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-232
Text:

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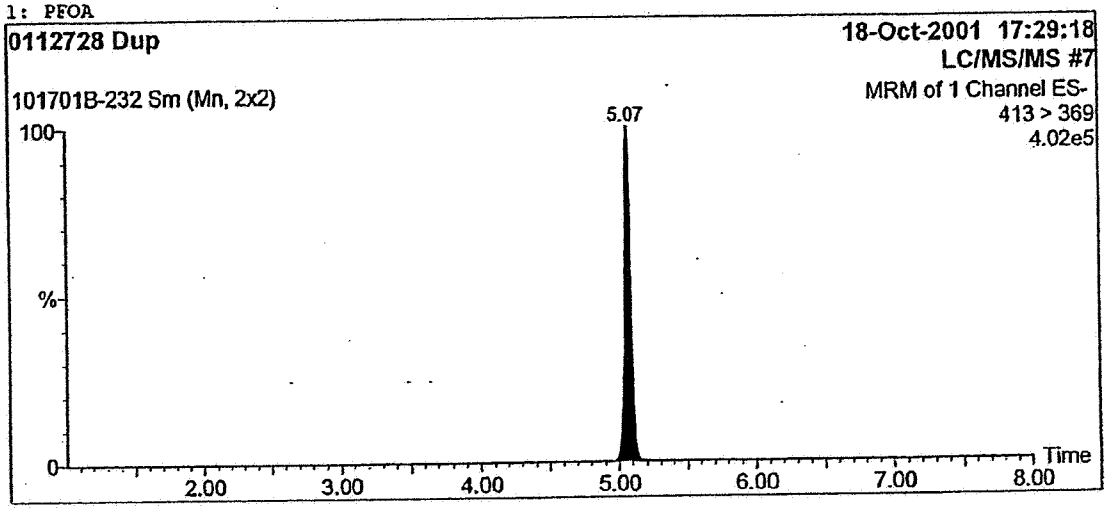

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Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-233
Text:

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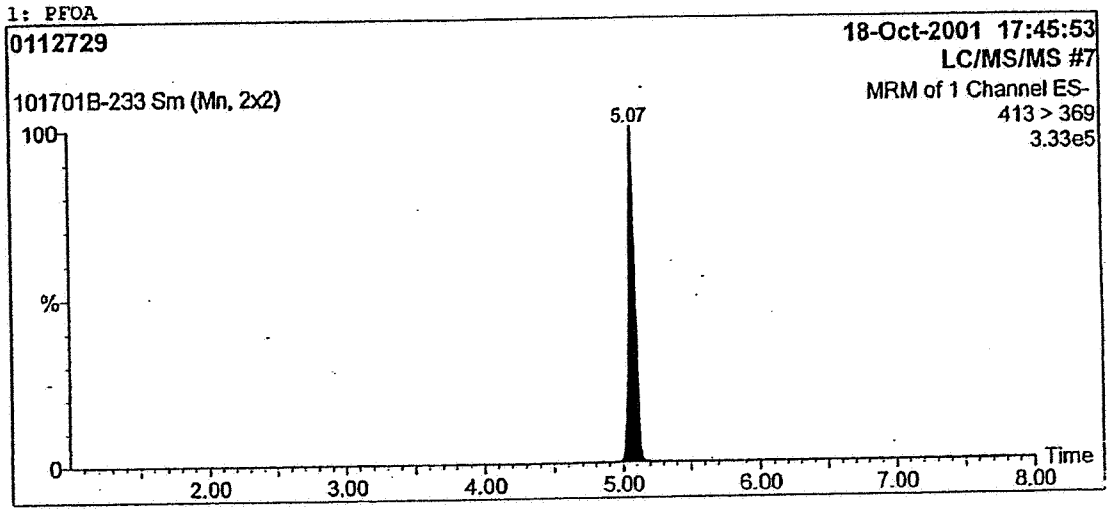

Quantify Sample Report
Study No.: 008-046, Set No: 101701B, Ext. Date: 10/17/01, Analyst: K Risha

Sample List: P:\Data\LCMSMS7\Masslynx\008-046.PRO\SampleDB\101701b
Last modified: Fri Oct 19 07:07:33 2001
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-234
Text:

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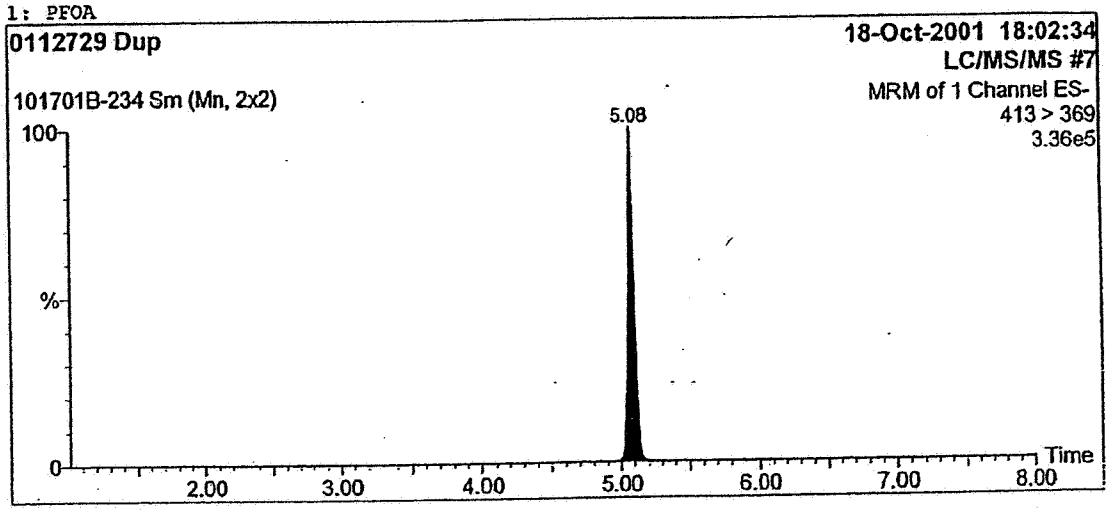

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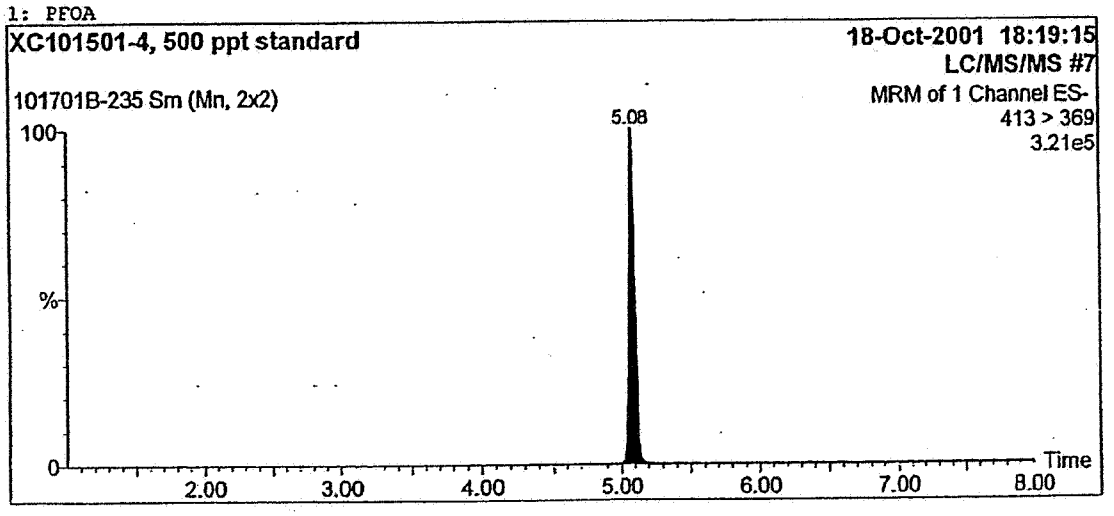
Method: P:\Data\LCMSMS7\Masslynx\008-046.PRO\MethDB\101601 PFOA
Last modified: Wed Oct 17 07:42:45 2001
Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-235
Text:

1: PFOA



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Last modified: Fri Oct 19 07:07:33 2001
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Job Code:

Printed: Fri Oct 19 07:15:16 2001

Name: 101701B-236
Text:

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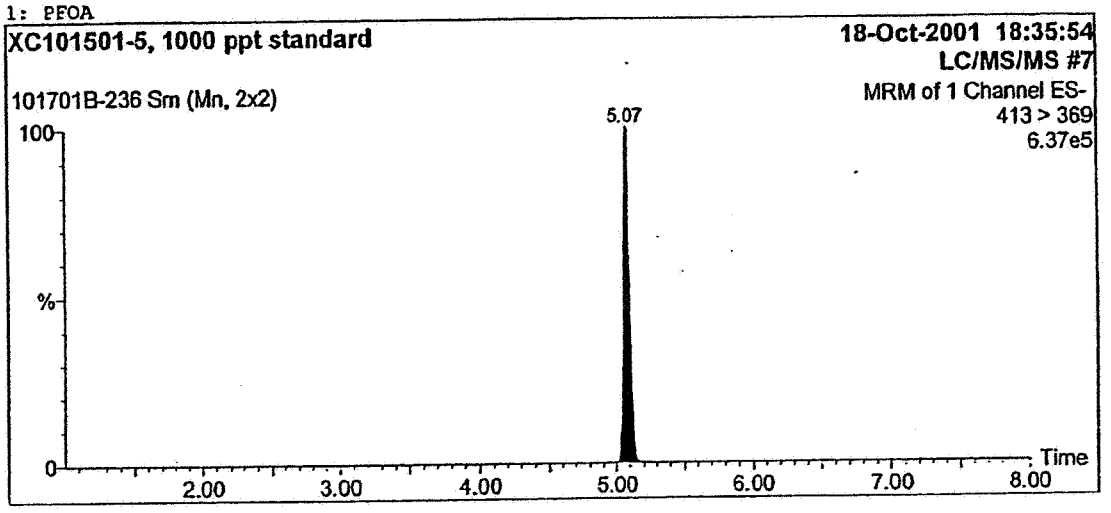

E

RJZ032668

EID185142

Exygen™ RESEARCH
Precise Research. Proven Results.

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

Pentadecafluorooctanoic acid (Inventory #: 01-008-75), obtained from Aldrich (lot # 08316DO) was used to make a stock solution (ID # S070901-1) which in turn, was used to make intermediate solutions (ID#'s F070901-1, F070901-2 and F070901-3). These intermediate solutions are used to prepare extracted calibration standards, laboratory control samples, and matrix spike samples.

Pentadecafluorooctanoic acid (Inventory #:01-008-092), obtained from Fluorochem USA (lot # U14D) was used to make a stock solution (ID # S101501-1) which in turn, was used to make intermediate solutions (ID#'s F101501-1 and F101501-2). 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.

KJR 11/09/01

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3048 Research Drive State College, PA 16801
Phone: (814) 231-8032 Fax: (814) 231-1253 or (814) 231-1580

ANALYTICAL TEST, CONTROL AND REFERENCE STANDARDS RECEIPT & INVENTORY LOG

Substance: PENTADECAFLUOROOCTANOIC ACID Page _____

Centre Analytical Inventory Number: 01-008-75

Amount Received (g): 5g Gross Weight Received (g): ① ~~34.3759~~ 34.59642

Date Received: 07/05/01 Condition Upon Receipt: Frozen-Refrigerated-Ambient

Received From: ALDRICH CHEM Lot/Batch No.: 08316DO

	Date Last Characterized	Expiration Date	% Purity	Initials/Date
1.	NA	07/05/02	96%	KR 07/05/01
2.				
3.				

Storage Location: FREEZER 13A STANDARD CABINET #3

Logged In By: [signature] Date: 07/05/01

Date	Initial Gross Wt. (g)	Amount Withdrawn (g)	Gross Wt. After Withdrawal (g)	Balance ID	Purpose/CAL Study Number	Initials
07/09/01	34.59582	0.01042	34.57692	16	008-046	KR

Comments: ① WEIGHT AFTER AFFIXING OXIGEN LABEL. KR 07/05/01

☐ Log continued next page

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT."
BY KR DATE 11/08/01

☐ Removed from Inventory _____ (Initials/Date)

☐ Returned to Sponsor _____ (Initials/Date)

8 February 2001/4

RJZ032670

EID185144

Product Number:171468

Product Name:Pentadecafluorooctanoic acid, 96%

ALDRICH

Certificate of Analysis

TEST	SPECIFICATION	LOT {08316DO} RESULTS
Product Name	Pentadecafluorooctanoic acid, 96%	
Product Number	171468	
CAS Number	335671	
Formula	$CF_3(CF_2)_6CO_2H$	
Formula Weight	414.06	
APPEARANCE	WHITE TO OFF-WHITE POWDER	WHITE POWDER
INFRARED SPECTRUM	CONFORMS TO STRUCTURE AND STANDARD AS ILLUSTRATED	CONFORMS TO STRUCTURE AND STANDARD.
TITRATION	95.5% - 104.5% (WITH NAOH)	100.4% (WITH NAOH)
GAS LIQUID CHROMATOGRAPHY	95.5% (MINIMUM)	99.9%
TITRATION	2 % H2O (MAXIMUM)	1.48% H2O (WITH 'KARL FISCHER' REAGENT)
QUALITY CONTROL ACCEPTANCE DATE		JUNE 2001

David Swessel

David Swessel, Supervisor
Quality Control

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT."
BY [illegible] DATE 11/08/01

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

ANALYTICAL TEST, CONTROL AND REFERENCE STANDARDS RECEIPT & INVENTORY LOG

Substance: PENTADECAFLUOROCTANOIC ACID OR PERFLUOROCTANOIC ACID (PFOA) Page 1

Exygen Inventory Number: 01-008-092

Amount Received (g): ~5 Gross Weight Received (g): 15.81911

Date Received: 10/12/01 Condition Upon Receipt: Frozen-Refrigerated-Ambient

Received From: FLUOROCHEM USA Lot/Batch No.: U14D

	Date Last Characterized	Expiration Date	% Purity	Initials/Date
1.	UNKNOWN	10/12/02	97	KJC 10/12/01
2.				
3.				

Storage Location: STANDARD CABINET #2 (REF. 13)

Logged In By: [illegible] Date: 10/12/01

Standard Custodian Review: [illegible] Date: 10/15/01

Date	Initial Gross Wt. (g)	Amount Withdrawn (g)	Gross Wt. After Withdrawal (g)	Balance ID	Purpose/Exygen Study Number	Initials
10/15/01	15.82014	0.01031	15.80300	16	008-046	KJC
11/01/01	15.79667	—	—	16	Inventory	CEG

Comments:

☐ Log continued next page

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☐ Removed from Inventory _____ (Initials/Date)

☐ Returned to Sponsor _____ (Initials/Date)

16 August 2001/5

RJZ032674

EID185147

Centre Analytical Laboratories, Inc.
3048 Research Drive State College, PA 16801
Phone: (814) 231-8032 Facsimile: (814) 231-1253

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PREPARATION OF STOCK STANDARD SOLUTION

Protocol Number: – CAL Study Number: 008-046

Compound: PENTADECAFLUOROOCTANOIC ACID (APFO) (PFOA) @ KR 11/05/01 Lot Number: 083160

Source: ALDRICH Purity: 96 % Date Last Characterized: NA

Date Standard Received: 07/05/01 Storage Location: STANDARD CABINET #3

Compound Expiration Date: 07/05/02 CAL Inventory #: 01-008-75

A calibration check must have been conducted with the balance on the day of sample weighing.

Balance Calibration	Stock Solution
Balance Number: 16 @ KR 07/09/01	Net Wt. of Substance: 0.01042 g
Logbook page for calibration data: 4	Wt. Adjusted for Purity: 0.01000 g
Solvent: METHANOL	Final Volume: 100 mL
Manufacturer: EMSCIENCE	Concentration: 100 µg/mL
Lot Number: 41025	
Stock Solution Expiration Date*: 02/09/02	Stock Solution ID: S070901-1 Stock Soln ID #: SMMDDYY-1,2,3,etc.

* Assign a one year expiration date for all stock solutions unless otherwise indicated by sponsor.

Storage Conditions for Solution:

Freezer Number: – Refrigerator Number: 13 Room Temperature: –

Indicate which of the above is applicable, and location.

REMARKS:

Signature: [signature] Date: 07/09/01

November 6, 1996/3

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PREPARATION OF FORTIFICATION STANDARD SOLUTIONS

Protocol Number: – CAL Study Number: 008-046

Date Prepared: 07/09/01 Expiration Date*: 02/09/02

*Assign a one year expiration date for all dilutions unless otherwise indicated by sponsor.

Separate sheets should be used for fortification standards and calibration standards.

Dilution of solution ID #	Compound (code no.)	Initial Conc. (µg/mL)	Aliquot volume (mL)	Diluted to volume (mL)	Solvent (or solvent number)	Final conc. (µg/mL)	Final solution ID # **
S070901-1	~~APF~~ PFOA	100	1.0	100	MeOH	1.0	F070901-1
F070901-1	↓	1.0	10.0	100	↓	0.1	F070901-2
F070901-2	↓	0.1	10.0	100	↓	0.01	F070901-3
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –

If 2 or more standard solutions are combined to make a mixed standard solution, indicate with a bracket of the solutions used.

Solvent 1: METHANOL Manufacturer: EMSCIENCE Lot No.: 41025

Solvent 2: – Manufacturer: – Lot No.: –

Solvent 3: – Manufacturer: – Lot No.: –

*** This must be a unique number. Use this system: Fortification Soln ID #: FMMDDYY-1,2,3, etc.*

Storage Conditions for Solution:

Freezer Number: – Refrigerator Number: 13 Room Temperature: –

Indicate which of the above is applicable, and location.

REMARKS: 0@KR 11/05/01

Signature: [signature] Date: 07/09/01

February 1995/2

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PREPARATION OF STOCK STANDARD SOLUTION

Protocol Number: NA Exygen Study Number: 008-046

Compound: PENTADECAFLUOROCTANOIC ACID (PFOA) Lot Number: ~~U15B~~ U14D (KP 10/15/01)

Source: FLUOROCHEM USA Purity: 97 % Date Last Characterized: NA

Date Standard Received: 10/12/01 Storage Location: STAND. CAB. #2

Compound Expiration Date: 10/12/02 Exygen Inventory #: 01-008-09

A calibration check must have been conducted with the balance on the day of sample weighing.

Balance Calibration	Stock Solution
Balance Number: 16	Net Wt. of Material: 0.01031 g
Logbook page for calibration data: 7	Wt. Adjusted for Purity: 0.01000 g
Solvent: METHANOL	Final Volume: 100 mL
Manufacturer: EM SCIENCE	Concentration: 100 µg/mL
Lot Number: 41202	

Stock Solution Expiration Date*: 04/~~12~~ 15/02 (KP 10/15/01) Stock Solution ID: S 101501-1
Stock Soln ID #: SMMDDYY-1,2,3,etc.

* Assign a one year expiration date for all stock solutions unless otherwise indicated by sponsor.

Storage Conditions for Solution:

Freezer Number: — Refrigerator Number: 13 Room Temperature: —
Indicate which of the above is applicable, and location.

REMARKS:

Signature: [signature] Date: 10/15/01

July 18, 2001/4

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PREPARATION OF FORTIFICATION STANDARD SOLUTIONS

Protocol Number: NA Exygen Study Number: 008-046

Date Prepared: 10/15/01 Expiration Date*: 04/15/02

*Assign a one year expiration date for all dilutions unless otherwise indicated by sponsor.

Separate sheets should be used for fortification standards and calibration standards.

Dilution of solution ID #	Compound (code no.)	Initial Conc. (µg/mL)	Aliquot volume (mL)	Diluted to volume (mL)	Solvent (or solvent number)	Final conc. (µg/mL)	Final solution ID # **
S101501-1	PFOA	100	1.0	100	MeOH	1.0	F101501-1
F101501-1	PFOA	1.0	10.0	100	MeOH	0.1	F 101501-2
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –
–	–	–	–	–	–	–	F –

If 2 or more standard solutions are combined to make a mixed standard solution, indicate the solutions used with a bracket.

Solvent 1: METHANOL Manufacturer: EMSCIENCE Lot No.: 41202

Solvent 2: – Manufacturer: – Lot No.: –

Solvent 3: – Manufacturer: – Lot No.: –

*** This must be a unique number. Use this system: Fortification Soln ID #: FMMDDYY-1,2,3, etc.*

Storage Conditions for Solution:

Freezer Number: – Refrigerator Number: 13 Room Temperature: –

Indicate which of the above is applicable, and location.

REMARKS:

Signature: [signature] Date: 10/15/01

July 16, 2001/3

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