

Study Title

Characterization Study of PFOS, Primary Standard - Test Control Reference
#TCR-00017-046
PHASE: SOLUBILITY OF PFOS IN NATURAL SEAWATER AND AN AQUEOUS SOLUTION OF 3.5%
SODIUM CHLORIDE

Data Requirement

40 CFR 160.105(b)

Author

Mark E. Ellefson

Phase Completion Date

Date of signing

Performing Laboratory

3M Environmental Laboratory
Building 2-3E-09
935 Bush Avenue
St. Paul, MN 55106

Project Identification

3M Environmental Laboratory Study # FACT-TCR002 (LIMS #E00-1716)
Centre Analytical Laboratories Study # 023-021

Total Number of Pages

138

GLP COMPLIANCE STATEMENT

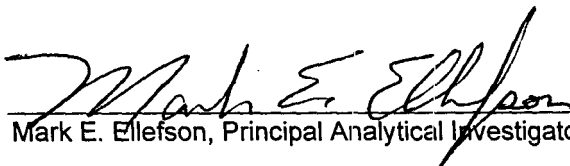
Study Title: Characterization Study of PFOS, Primary Standard – Test Control Reference # TCR-00017-046, PHASE: Solubility of PFOS in Natural Seawater and an Aqueous Solution of 3.5% Sodium Chloride.

Study Identification Number: FACT-TCR002, Centre Analytical Laboratories Study # 023-021

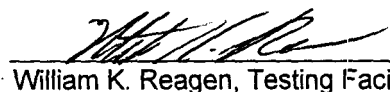
This phase of the study was conducted in compliance with Environmental Protection Agency (EPA) Good Laboratory Practice (GLP) Standards 40 CFR 160 with the exceptions in the bulleted list below.

Exceptions to GLP compliance:

- The electronic data systems in use have not been validated and there is not an electronic audit trail of corrections currently available (40 CFR 160.130 (e)). Authenticated hardcopies of chromatograms and associated documents will be considered as the original raw data.


Mark E. Ellefson, Principal Analytical Investigator

05/21/01
Date


William K. Reagen, Testing Facility Management

05/21/01
Date

QUALITY ASSURANCE STATEMENT

Study Title: Characterization Study of PFOS, Primary Standard – Test Control Reference # TCR-00017-046, PHASE: Solubility of PFOS in Natural Seawater and an Aqueous Solution of 3.5% Sodium Chloride.

Study Identification Number: FACT-TCR002, Centre Analytical Laboratories Study # 023-021

This phase of the study has been inspected by the 3M Environmental Laboratory Quality Assurance Unit (QAU) as indicated in the following table. The findings were reported to the study director and laboratory management.

Inspection Dates	Phase	Date Reported to Management	Study Director
4/23/01 – 4/26/01	Data	4/27/01	4/27/01
4/26/01 – 4/27/01	Draft Phase Report	4/27/01	4/27/01

QA Representative

Cosy Howell

May 21, 2001
Date

TABLE OF CONTENTS

GLP Compliance Statement.....	2
Quality Assurance Statement.....	3
Study Information	6
Summary	7
Purpose	7
Test Substance	8
Test System	8
Method Summaries	9
Preparatory Methods.....	9
Sample Collection and Analysis	10
Analytical Method	11
Analytical Results	12
Data Summary	13
Statistical Methods	16
Statement of Conclusion	16
References	17
List of Attachments.....	17
Signature Page.....	18
Attachment A: Extraction and Analytical Methods	19
Attachment B: Data Summary Tables	40
Attachment C: Sample Chromatograms	63
Attachment D: Deviations from the Protocol	133
Attachment E: Sample Calculations	138

LIST OF TABLES

Table 1a. Summary Table of Solubility of PFOS TCR-00017-046 in Natural Seawater.	7
Table 1b. Summary Table of Solubility of PFOS TCR-00017-046 in an Aqueous solution of 3.5% Sodium Chloride.	7
Table 2. Characterization of the Test Substance	8
Table 3. Characterization of the Analytical Reference Substances	8
Table 4. Test System	8
Table 5a. Shake Flask Method Preparation for Natural Seawater.	9
Table 5b. Shake Flask Method Preparation for an Aqueous Solution of 3.5% Sodium Chloride.	9
Table 6a. Sample Collection and Preparation for Study FACT-TCR002 in Natural Seawater.*	10
Table 6b. Sample Collection and Preparation for Study FACT-TCR002 in Aqueous 3.5% Sodium Chloride.*	10
Table 7a. Calibration Standard Reprocessing Summary for PFOS in Natural Seawater.	12
Table 7b. Calibration Standard Reprocessing Summary for PFOS in an Aqueous Solution of 3.5% Sodium Chloride.	13
Table 8a. PFOS TCR-00017-046 Solubility in Natural Seawater Data Summary	14
Table 8b. PFOS TCR-00017-046 Solubility in an Aqueous Solution of 3.5% Sodium Chloride Data Summary	15
Table 9a. Summary Table of Solubility of PFOS TCR-00017-046 in Natural Seawater.	16
Table 9b. Summary Table of Solubility of PFOS TCR-00017-046 in an Aqueous solution of 3.5% Sodium Chloride.	16

STUDY INFORMATION

Study Director

John Flaherty
Centre Analytical Laboratory
3048 Research Drive
State College, PA 16801

Analytical Chemistry Laboratories

Solubility Testing

3M Environmental Technology
and Safety Services (ET&SS)
3M Environmental Laboratory
Bldg. 2-3E-09
935 Bush Avenue
St. Paul, MN 55106

Certificate of Analysis

Centre Analytical Laboratory
3048 Research Drive
State College, PA 16801

John Flaherty, *Study Director*

Dr. William K. Reagen, *Laboratory Manager*
Mark E. Ellefson, *Principal Analytical Investigator*
Kristin L. Terrell, *Analytical Chemist*
Mark L. Anderson, *Analytical Chemist*
Cindy M. Carlson, *Analytical Chemist*

Sponsor

Dr. William K. Reagen (Sponsor Representative)
3M Environmental Technology and Safety Services (ET&SS)
3M Environmental Laboratory
2-3E-09
935 Bush Avenue
St. Paul, MN 55106

Experimental Dates

Experimental Start Date: 22 January 2001
Experimental Termination Date: 07 February 2001

Archives

Reserve samples of the reference standards will be maintained by the 3M Environmental Laboratory. All study data for this phase and a copy of the phase report are archived by the 3M Environmental Laboratory.

SUMMARY

The solubility of PFOS in natural seawater and an aqueous solution of 3.5% sodium chloride was determined quantitatively via the Shake Flask Method.

Results are presented in Tables 1a and 1b:

Table 1a. Summary Table of Solubility of PFOS TCR-00017-046 in Natural Seawater.

SAMPLING EVENT	PFOS SOLUBILITY IN NATURAL SEAWATER (CORRECTED FOR PURITY)	Standard Deviation	% CV
Day 1	12.2 µg/mL	0.47	3.9%
Day 2	11.9 µg/mL	0.07	0.6%
Day 3	13.2 µg/mL	1.30	9.8%
Average Value	12.4 µg/mL	0.71	5.7%

The solubility of PFOS TCR-00017-046 in natural seawater was determined to be 12.4 µg/mL at 22-23 °C.

Table 1b. Summary Table of Solubility of PFOS TCR-00017-046 in an Aqueous solution of 3.5% Sodium Chloride.

SAMPLING EVENT	PFOS SOLUBILITY IN AN AQUEOUS SOLUTION OF 3.5% SODIUM CHLORIDE (CORRECTED FOR PURITY)	Standard Deviation	% CV
Day 1	Excluded	Excluded	Excluded
Day 2	20.0 µg/mL	2.20	11.0%
Day 3	19.9 µg/mL	1.31	6.6%
Average Value	20.0 µg/mL	0.07	0.4%

The solubility of PFOS TCR-00017-046 in an aqueous solution of 3.5% sodium chloride was determined to be 20.0 µg/mL at 22-24 °C. Day 1 data was excluded because the %CV was greater than 15% as per the quality controls defined in ETS-8-172.0.

PURPOSE

The purpose of this phase of the study was to determine the solubility of PFOS in natural seawater and an aqueous solution of 3.5% sodium chloride as part of the characterization of this test, control, and reference substance.

TEST SUBSTANCE

PFOS, Test Control Reference #TCR-00017-046

Table 2. Characterization of the Test Substance

TEST SUBSTANCE	PFOS
Source	Re-crystallized by George Moore, 3M Specialty Materials, Bldg. 236-1B-10
Expiration Date	8/31/01
Storage Conditions	Frozen
3M Laboratory Identification Number	TCR-00017-046
Physical Description	White Powder
Purity	97.9%

REFERENCE SUBSTANCE

Table 3. Characterization of the Analytical Reference Substances

REFERENCE SUBSTANCE	PFOS	THPFOS	PFBS
Source	Re-crystallized by George Moore, 3M Specialty Materials, Bldg. 236-1B-10	SynQuest Labs	3M, Cottage Grove
Expiration Date	8/31/01	Not Available	12/04/01
Storage Conditions	Frozen	Frozen	Frozen
3M Laboratory Identification Number	TCR-00017-046	TCR-00017-047 Lot #Q75-30	TCR-99030-023 Lot #101
Physical Description	White Powder	White Powder	White Powder
Purity	97.9%	NA	96.7

TEST SYSTEM

Table 4. Test System

TEST SYSTEM	NATURAL SEAWATER	SODIUM CHLORIDE
Source	Ocean Scientific International	EM Science
Expiration Date	2005	2010
Storage Conditions	Ambient	Ambient
3M Laboratory Identification Number	TN-A: 4193	TN-A: 2458
Purity/Grade	Salinity = 3.5%	99%

METHOD SUMMARIES

The solubility determination of PFOS TCR-00017-046 in natural seawater and aqueous 3.5% NaCl was performed according to 3M Environmental Laboratory method ETS-8-172.0 "Shake Flask Method; Solubility Determination of a Test Substance in Various Solvents" as adapted from United States Environmental Protection Agency OPPTS 830.7840, "Water Solubility: Column Elution Method; Shake Flask Method" and OECD 105, "Water Solubility" Guidelines. ETS-8-172.0 was subsequently revised to include additional information/clarification. The new revision, ETS-8-172.1, is procedurally the same as its predecessor and is included in Attachment A of this report. All analyses were performed according to the 3M Environmental Laboratory method ETS-8-155.0 "Analysis of Perfluorooctanesulfonate or Other Fluorochemicals in Waste Stream or Water Extracts Using HPLC-Electrospray/Mass Spectrometry".

The approximate solubility of PFOS in natural seawater and in an aqueous solution of 3.5% NaCl was estimated using the previously determined solubility of PFOS in water. The Shake Flask Method was used for the quantitative determination of PFOS solubility. A detailed description of the Shake Flask method used in this study is located in Attachment A.

PREPARATORY METHODS

- **ETS-8-172.1 "Shake Flask Method: Solubility Determination of a Test Substance in Various Solvents."** Greater than 6.5 mg PFOS was weighed into a tared 15 mL plastic centrifuge tube. Approximately 5 g of natural seawater or 3 g of 3.5% sodium chloride solution was added gravimetrically to each 15 mL centrifuge tube. Nine centrifuge tubes were prepared, along with three method blanks (tubes with natural seawater or 3.5% sodium chloride solution only) for each test system. The centrifuge tubes were placed in a temperature-controlled orbital shaker set at approximately 30°C. See the following tables documenting the shake flask method preparation:

Table 5a. Shake Flask Method Preparation for Natural Seawater.

STEP	CENTRIFUGE TUBE PREPARATION	REPLICATES	DATE(S) PERFORMED
1	>6.5 mg PFOS weighed into tared 15 mL centrifuge tube	9	01-22-01
2	Natural seawater added to centrifuge tube such that total weight of PFOS and solvent was approximately between 5.0 – 5.2 g.	9	01-22-01
3	Method Blank Preparation- Approximately 5 g natural seawater added to a tared centrifuge tube gravimetrically.	3	01-22-01
4	Samples sealed with tape, vortex-mixed, and placed in a temperature-controlled orbital shaker set at approximately 30.0°C, rotation speed set at approximately 150 rpm.	12	01-22-01

Table 5b. Shake Flask Method Preparation for an Aqueous Solution of 3.5% Sodium Chloride.

STEP	CENTRIFUGE TUBE PREPARATION	REPLICATES	DATE(S) PERFORMED
1	>9.5 g PFOS weighed into tared 15 mL centrifuge tube	9	01-29-01
2	3.5% Sodium Chloride solution added to centrifuge tube such that total weight of PFOS and solvent was approximately between 3.0-3.1 g.	9	01-29-01
3	Method Blank Preparation- Approximately 3 g of aqueous solution 3.5% sodium chloride added to a tared centrifuge tube gravimetrically.	3	01-29-01
4	Samples sealed with tape, vortex-mixed, and placed in a temperature-controlled orbital shaker set at approximately 30.0°C, rotation speed set at approximately 150 rpm.	12	01-29-01

SAMPLE COLLECTION AND ANALYSIS

On each of Days 1, 2, and 3 (approximately 24, 48 and 72 hours after initial sample preparation) three centrifuge tubes (containing PFOS) and a method blank were removed from the incubator/shaker and allowed to equilibrate at 22-24 °C for approximately 24 hours. After this, they were centrifuged and then had aliquots of supernatant collected. The aliquots were diluted using 100X dilutions (990 µL methanol:10 µL sample). The diluted samples were spiked with internal standard (17µL of 15 µg THPFOS/mL and 15 µg PFBS/mL) and analyzed via HPLC/ES/MS according to analytical method ETS-8-155.0. Four injections of each final diluted sample were performed.

The following tables describe the sample collection and preparation regimen:

Table 6a. Sample Collection and Preparation for Study FACT-TCR002 in Natural Seawater.*

STEP	PROCEDURE	REPLICATES	DATE(S) PERFORMED
1	At 24, 48, and 72 hours of shaking, 3 PFOS+ solvent, and 1 solvent (method blank) centrifuge tubes are removed from the incubator and equilibrated at 24-25 °C for approximately 24 hours.	4 centrifuge tubes per day	Day1 01-23-01
			Day2 01-24-01
			Day3 01-25-01
2	Aliquots of the supernatant (solvent containing dissolved PFOS or solvent only from the method blanks) were collected and dispensed into autovials.	4 per centrifuge tube	Day1 01-24-01
			Day2 01-25-01
			Day3 01-26-01
3	The aliquots were diluted 100X with methanol (using a dual syringe diluter).	Day 1	01-24-01
		Day 2	01-25-01
		Day 3	01-26-01
4	Internal Standard was added to the 100X sample dilutions (17µL of 15 µg THPFOS/mL and 15 µg PFBS/mL in methanol).	Day 1	01-24-01
		Day 2	01-26-01
		Day 3	01-26-01
5	Samples were analyzed via HPLC/ES/MS on HP 1100 LC/MSD	Day 1	01-25-01
		Day 2	01-27-01 – 01-28-01
		Day 3	01-30-01

Table 6b. Sample Collection and Preparation for Study FACT-TCR002 in Aqueous 3.5% Sodium Chloride.*

STEP	PROCEDURE	REPLICATES		DATE(S) PERFORMED
1	At 24, 48, and 72 hours of shaking, 3 PFOS+ solvent, and 1 solvent (method blank) centrifuge tubes are removed from the incubator and equilibrated at 24-25 °C for approximately 24 hours.	4 centri-fuge tubes per day	Day1	01-30-01
			Day2	01-31-01
			Day3	02-01-01
2	Aliquots of the supernatant (solvent containing dissolved PFOS or solvent only from the method blanks) were collected and dispensed into autovials.	4 per centrifuge tube	Day1	01-31-01
			Day2	02-01-01
			Day3	02-02-01
3	The aliquots were diluted 100X with methanol (using a dual syringe diluter).	Day 1		01-31-01
		Day 2		02-01-01
		Day 3		02-02-01
4	Internal Standard was added to the 100X sample dilutions (17µL of 15 µg THPFOS/mL and 15 µg PFBS/mL in methanol).	Day 1		01-31-01
		Day 2		02-02-01
		Day 3		02-02-01
5	Samples were analyzed via HPLC/ES/MS on HP 1100 LC/MSD	Day 1		01-31-01 – 02-01-01
		Day 2		02-05-01 – 02-06-01
		Day 3		02-06-01 – 02-07-01

*Information pertains to sample preparation and dates of final reported data only.

ANALYTICAL METHOD

- ETS-8-155.0 "Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Waste Stream or Water Extracts Using HPLC-Electrospray/Mass Spectrometry". Diluted samples (i.e. diluted supernatants) were analyzed using HPLC/ES/MS in the negative ion mode. PFOS levels were evaluated versus calibration standards ranging in concentration from 2.5-1002 ng PFOS/mL. Internal Standard quantification was used to normalize the data. Target ions were 499 m/z (PFOS anion), 427 m/z (deprotonated THPFOS), and 299 m/z (PFBS anion).

Analytical Equipment

Liquid Chromatograph: Hewlett-Packard® Series 1100 Liquid Chromatograph system

Analytical column: Keystone® Betasil™ C₁₈ 2x50mm, 5µm particle size

Column temperature: Ambient

Cycle Time: 9.75 minutes

Flow rate: 300µL/min

Injection volume: 10µL

Mobile phase components:

Solvent A: 2.0 mM ammonium acetate in water

Solvent C: Methanol

Solvent Gradient:	<u>Time</u>	<u>% C</u>
	0.00	15 %
	0.50	15 %
	0.80	60 %
	3.50	100 %
	6.50	100 %
	7.00	15 %
	9.75	15 %

Mass Spectrometer: Hewlett-Packard® Series 1100 API/Mass Spectrometer Detector

Software: HP ChemStation™ 6.0

Fragmentor Voltage: m/z 427=100 V; m/z 499= 140 V; m/z 299 = 100 V.

Capillary Voltage: 3500 V

Gain = 2.0 EMV

Mode: Electrospray Negative

Gas Temperature: 350 °C.

Drying Gas: 10.0 L /min.

Nebulizer Pressure: 25 psig

Analysis Type: Single Ion Monitoring (SIM)

ANALYTICAL RESULTS

Data quality objectives outlined in the 3M Environmental Laboratory method were met (see Appendix A).

- **Regressions.** Quadratic curve fit was applied to calibration standards and sample data to improve quantitation over the concentration range appropriate to the data. All calibration curves had least-square fits of 0.990 or greater (R^2 values ranged from 0.9992 - 0.9999).
- **Calibration Standards.** Eleven standards ranging in concentration from approximately 2.5 to 1000 ng PFOS/mL methanol were used for the calibration curves. Calibration curves were run before and after every analytical sequence.
- **Sample Replicates.** Samples 012201-swPFOS-2.3.1, 012201-swPFOS-2.3.2, 012201-swPFOS-2.3.3, 012201-swPFOS-2.3.4 and naclPFOS.2.2.1 were excluded from the reported data using Dixon's Q-Test.
- **Continuing Calibration Verification.** For quantitative determinations, a mid-level matrix calibration check was analyzed every eight samples to monitor instrumental drift, with a limit of $\pm 15\%$ deviation of the target concentrations.
- **Limit of Quantitation (LOQ).** The LOQ is equal to the lowest standard in the calibration curve (either 2.5, 5.0 or 10.0 ng/mL).

Table 7a. Calibration Standard Reprocessing Summary for PFOS in Natural Seawater.

DATA SUB BATCH, ANALYSIS DATE	CALIBRATION STANDARDS	STANDARDS NOT USED, REASON FOR EXCLUSION
Day 1 data: 01-25-01	2.5 — 1002 ng/mL	All curve points were used.*
Day 2 data: 01-27-01 — 01-28-01	2.5 — 1002 ng/mL	All curve points were used.*
Day 3 data: 01-30-01	2.5 — 1002 ng/mL	All curve points were used.*

Table 7b. Calibration Standard Reprocessing Summary for PFOS in an Aqueous Solution of 3.5% Sodium Chloride.

DATA SUB BATCH, ANALYSIS DATE	CALIBRATION STANDARDS	STANDARDS NOT USED, REASON FOR EXCLUSION
Day 1 data: 01-31-01 – 02-01-01	5 – 501 ng/mL	Stnd 2 (2.5 ng/mL), and Stnd 13 (1002 ng/mL) were excluded to better fit the data at the low end of the curve.*
Day 2 data: 02-05-01 – 02-06-01	10 – 1002 ng/mL	Stnd 2 (2.5 ng/mL) and Stnd 3 (5.0 ng/mL) was excluded to better fit the data at the low end of the curve.*
Day 3 data: 02-06-01 – 02-07-01	5 – 501 ng/mL	Stnd 2 (2.5 ng/mL), and Stnd 13 (1002 ng/mL) were excluded to better fit the data at the low end of the curve.*

*The first standard, Stnd 1 (0 ng/mL), is not used since there is no response measurable.

- **Blanks.**

- Method blanks (natural seawater or the aqueous solution of 3.5% sodium chloride taken through the entire sample preparation, dilution, and analysis process) provided a measure of laboratory contamination. Acceptable values for the blanks were less than 50% of the limit of quantitation (LOQ).
- Solvent blanks (methanol injections) provided a measure of instrument contamination. Acceptable solvent blanks must contain levels of target analyte less than 50% of the limit of quantitation (LOQ). Acceptable solvent blanks were analyzed before each calibration curve.

- **Specificity (according to OPPTS 830.7840):** The solubility determination as stated in the EPA Guidelines follows. This method should only be applied to:
 - Pure substance. This study shows a purity of 97.9%.
 - Substances that are stable in water. Hydrolytic studies have been conducted at the 3M Environmental Laboratory showing stability (Report # W1878).

DATA SUMMARY

Tables 8a and 8b summarize individual sample data. Representative chromatograms are presented in Attachment C. The tables display PFOS concentrations for individual injections. Also included for each centrifuge tube replicate are the averages, standard deviation, and % coefficient of variation.

Table 8a. PFOS TCR-00017-046 Solubility in Natural Seawater Data Summary

SAMPLE ID		TCR-00017-046 PFOS UG/mL CORRECTED FOR PURITY	AVERAGE UG/mL STD DEV % COEFFICIENT OF VARIATION
Day 1 Centrifuge Tube #1	Rep 1: 012201-swPFOS-1.1.1	12.3	12.6
	Rep 2: 012201-swPFOS-1.1.2	12.7	0.25
	Rep 3: 012201-swPFOS-1.1.3	12.6	2.0%
	Rep 4: 012201-swPFOS-1.1.4	12.9	
Day 1 Centrifuge Tube #2	Rep 1: 012201-swPFOS -1.2.1	11.6	11.7
	Rep 2: 012201-swPFOS -1.2.2	12.0	0.19
	Rep 3: 012201-swPFOS -1.2.3	11.7	1.6%
	Rep 4: 012201-swPFOS -1.2.4	11.6	
Day 1 Centrifuge Tube #3	Rep 1: 012201-swPFOS -1.3.1	12.2	12.4
	Rep 2: 012201-swPFOS -1.3.2	12.5	0.13
	Rep 3: 012201-swPFOS -1.3.3	12.4	1.0%
	Rep 4: 012201-swPFOS -1.3.4	12.3	
Day 2 Centrifuge Tube #1	Rep 1: 012201-swPFOS -2.1.1	11.9	11.8
	Rep 2: 012201-swPFOS -2.1.2	11.7	0.10
	Rep 3: 012201-swPFOS -2.1.3	11.7	0.8%
	Rep 4: 012201-swPFOS -2.1.4	11.8	
Day 2 Centrifuge Tube #2	Rep 1: 012201-swPFOS -2.2.1	12.0	11.9
	Rep 2: 012201-swPFOS -2.2.2	12.1	0.18
	Rep 3: 012201-swPFOS -2.2.3	11.8	1.5%
	Rep 4: 012201-swPFOS -2.2.4	11.7	
Day 2 Centrifuge Tube #3	Rep 1: 012201-swPFOS -2.3.1	15.2	15.6 *
	Rep 2: 012201-swPFOS -2.3.2	15.3	0.39
	Rep 3: 012201-swPFOS -2.3.3	15.8	2.5%
	Rep 4: 012201-swPFOS -2.3.4	16.0	*Excluded via Dixon's Q-Test
Day 3 Centrifuge Tube #1	Rep 1: 012201-swPFOS -3.1.1	14.5	14.5
	Rep 2: 012201-swPFOS -3.1.2	14.3	0.10
	Rep 3: 012201-swPFOS -3.1.3	14.5	0.7%
	Rep 4: 012201-swPFOS -3.1.4	14.5	
Day 3 Centrifuge Tube #2	Rep 1: 012201-swPFOS -3.2.1	13.4	13.3
	Rep 2: 012201-swPFOS -3.2.2	13.1	0.17
	Rep 3: 012201-swPFOS -3.2.3	13.3	1.3%
	Rep 4: 012201-swPFOS -3.2.4	13.5	
Day 3 Centrifuge Tube #3	Rep 1: 012201-swPFOS -3.3.1	11.7	11.9
	Rep 2: 012201-swPFOS -3.3.2	12.1	0.23
	Rep 3: 012201-swPFOS -3.3.3	11.7	1.9%
	Rep 4: 012201-swPFOS -3.3.4	12.1	

Table 8b. PFOS TCR-00017-046 Solubility in an Aqueous Solution of 3.5% Sodium Chloride Data Summary

SAMPLE ID		TCR-00017-046 PFOS UG/ML CORRECTED FOR PURITY	AVERAGE UG/ML STD DEV %COEFFICIENT OF VARIATION
Day 1 Centrifuge Tube #1	Rep 1: 012901-nacIPFOS -1.1.1	22.2	22.3**
	Rep 2: 012901-nacIPFOS -1.1.2	22.3	0.45
	Rep 3: 012901-nacIPFOS -1.1.3	21.7	2.0%
	Rep 4: 012901-nacIPFOS -1.1.4	22.8	
Day 1 Centrifuge Tube #2	Rep 1: 012901-nacIPFOS -1.2.1	19.8	19.9**
	Rep 2: 012901-nacIPFOS -1.2.2	19.9	0.17
	Rep 3: 012901-nacIPFOS -1.2.3	19.7	0.9%
	Rep 4: 012901-nacIPFOS -1.2.4	20.1	
Day 1 Centrifuge Tube #3	Rep 1: 012901-nacIPFOS -1.3.1	15.5	15.8**
	Rep 2: 012901-nacIPFOS -1.3.2	16.1	0.26
	Rep 3: 012901-nacIPFOS -1.3.3	15.7	1.6%
	Rep 4: 012901-nacIPFOS -1.3.4	15.9	
Day 2 Centrifuge Tube #1	Rep 1: 012901-nacIPFOS -2.1.1	21.0	22.2
	Rep 2: 012901-nacIPFOS -2.1.2	21.4	1.52
	Rep 3: 012901-nacIPFOS -2.1.3	22.1	6.8%
	Rep 4: 012901-nacIPFOS -2.1.4	24.4	
Day 2 Centrifuge Tube #2	Rep 1: 012901-nacIPFOS -2.2.1	28.1*	20.0
	Rep 2: 012901-nacIPFOS -2.2.2	20.3	0.23
	Rep 3: 012901-nacIPFOS -2.2.3	19.9	1.2%
	Rep 4: 012901-nacIPFOS -2.2.4	19.9	*Rep 1 excluded via Dixon's Q-Test
Day 2 Centrifuge Tube #3	Rep 1: 012901-nacIPFOS -2.3.1	18.1	17.8
	Rep 2: 012901-nacIPFOS -2.3.2	17.9	0.25
	Rep 3: 012901-nacIPFOS -2.3.3	17.8	1.4%
	Rep 4: 012901-nacIPFOS -2.3.4	17.5	
Day 3 Centrifuge Tube #1	Rep 1: 012901-nacIPFOS -3.1.1	21.2	21.1
	Rep 2: 012901-nacIPFOS -3.1.2	20.9	0.13
	Rep 3: 012901-nacIPFOS -3.1.3	21.1	0.6%
	Rep 4: 012901-nacIPFOS -3.1.4	21.1	
Day 3 Centrifuge Tube #2	Rep 1: 012901-nacIPFOS -3.2.1	20.4	20.1
	Rep 2: 012901-nacIPFOS -3.2.2	20.3	0.45
	Rep 3: 012901-nacIPFOS -3.2.3	20.1	2.2%
	Rep 4: 012901-nacIPFOS -3.2.4	19.4	
Day 3 Centrifuge Tube #3	Rep 1: 012901-nacIPFOS -3.3.1	18.6	18.5
	Rep 2: 012901-nacIPFOS -3.3.2	18.5	0.24
	Rep 3: 012901-nacIPFOS -3.3.3	18.1	1.3%
	Rep 4: 012901-nacIPFOS -3.3.4	18.6	

** Triplicates not excluded via the Q-Test. However, when the triplicates for Day 1 were averaged, the %CV was outside of the acceptable range. The Day 1 solubility was therefore excluded.

The average PFOS concentration and standard deviation for each day, as well as the final overall average for all non-excluded replicates and time points were as follows:

Table 9a. Summary Table of Solubility of PFOS TCR-00017-046 in Natural Seawater.

SAMPLING EVENT	PFOS SOLUBILITY IN NATURAL SEAWATER (CORRECTED FOR PURITY)	Standard Deviation	% CV
Day 1	12.2 µg/mL	0.47	3.9%
Day 2	11.9 µg/mL	0.07	0.6%
Day 3	13.2 µg/mL	1.30	9.8%
Average Value	12.4 µg/mL	0.71	5.7%

The solubility of PFOS TCR-00017-046 in natural seawater was determined to be 12.4 µg/mL at 22-23 °C.

Table 9b. Summary Table of Solubility of PFOS TCR-00017-046 in an Aqueous solution of 3.5% Sodium Chloride.

SAMPLING EVENT	PFOS SOLUBILITY IN AN AQUEOUS SOLUTION OF 3.5% SODIUM CHLORIDE (CORRECTED FOR PURITY)	Standard Deviation	% CV
Day 1	Excluded	Excluded	Excluded
Day 2	20.0 µg/mL	2.20	11.0%
Day 3	19.9 µg/mL	1.31	6.6%
Average Value	20.0 µg/mL	0.07	0.4%

The solubility of PFOS TCR-00017-046 in an aqueous solution of 3.5% sodium chloride was determined to be 20.0 µg/mL at 22-24 °C. Day 1 data was excluded because the %CV was greater than 15%, as per the quality controls defined in ETS-8-172.0.

Attachment B contains data summary tables.

STATISTICAL METHODS

Statistical methods were limited to calculating means, standard deviations, and %CVs using Dixon's Q-Test to exclude data points. Refer to Attachment E for formulas and example calculations.

STATEMENT OF CONCLUSION

Under the conditions of the present study, the solubility of PFOS TCR-00017-046 in natural seawater and an aqueous solution of 3.5% sodium chloride is 12.4 µg/mL at 22-23 °C and 20.0 µg/mL at 22-24 °C respectively.

REFERENCES

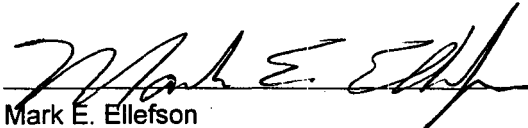
1. Fate, Transport and Transformation Test Guidelines Office of Prevention, Pesticides and Toxic Substances (OPPTS) 830.7840 Water Solubility: Column Elution Method; Shake Flask Method. EPA 712-C-96-041, August 1996.
2. OECD 105: Water Solubility. Adopted 27 July, 1995.

LIST OF ATTACHMENTS

- Attachment A: Extraction and Analytical Methods
- Attachment B: Data Summary Tables
- Attachment C: Sample Chromatograms
- Attachment D: Deviations from the Protocol
- Attachment E: Sample Calculations

SIGNATURE PAGE

We certify that this report is a true and complete representation of the data for this phase of the study:



Mark E. Ellefson

Principal Analytical Investigator

05/21/01
Date



William K. Reagan

Laboratory Manager

05/21/01
Date

ATTACHMENT A: EXTRACTION AND ANALYTICAL METHODS

ETS-8-172.0, the method approved during the study, has been replaced with a new version ETS-8-172.1. The new version is procedurally the same as the original version, but since it gives more details on the actual procedures, this new version is presented instead.

3M ENVIRONMENTAL LABORATORY

SHAKE FLASK METHOD: SOLUBILITY DETERMINATION OF A TEST SUBSTANCE IN VARIOUS SOLVENTS

Method Number: ETS-8-172.1

Adoption Date: 09/08/00

Effective Date: 03/27/01

Authors: Kristin L. Terrell, Mark L. Anderson, and Mark E. Ellefson

Approved By:


William K. Reagen, Laboratory Manager

03/27/01
Date


Mark E. Ellefson, Project Lead

03/27/01
Date

1.0 Scope and Application

- 1.1 **Purpose.** According to the United States Environmental Protection Agency (U.S. EPA) and Organization for the Economic Cooperation and Development (OECD) guidelines, solubility determination of substances that have solubilities in a given test matrix (e.g. water/acetone/methanol) of greater than 100 µg/mL must be analyzed via the shake flask method (OECD Guideline 105, and OPPTS Guideline 830.7840). The prerequisite to this method is a preliminary screen of the test compound for its approximate solubility level. Refer to ETS-8-170.0 for the preliminary solubility screen test procedures.

Exact Copy of Original

MIA 03 Apr 01
Initial Date

ETS-8-172.1

Solubility Test: *Shake Flask Method*
FACT-TCR002 (LIMS #E00-1716), Page 20 of 138

Page 1 of 11

- 1.2 **Compatible Analytes.** Test substance and degradation products for solubility testing.
- 1.3 **Acceptable Matrices.** Water, Acetone, and Methanol, or other solvents of interest.

2.0 SUMMARY OF METHOD

- 2.1 Using the qualitative/semi-quantitative data obtained from the preliminary solubility screen test, weigh out more than five times the estimated soluble concentration of test substance into each of twelve screw-capped vessels (three time points with four test vessels each-sample, duplicate, triplicate, and matrix blank). Add the appropriate amount of test solvent gravimetrically. Cap the tubes, seal the caps with tape, and place horizontally on an incubator/shaker set to $30\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, shaking for approximately 24 hours. At approximately 24 hours, the first time point will be pulled and equilibrated at room temperature (about $20\text{ }^{\circ}\text{C}$ - $26\text{ }^{\circ}\text{C}$) for approximately 24 hours. The equilibrated samples are centrifuged and aliquoted into autovials. Samples are then diluted 1:10 or higher with a suitable solvent for analysis via LC/MS. At approximately 48 hours and 72 hours, the second and third sets, respectively, will be pulled, equilibrated, centrifuged, aliquoted and diluted with a suitable analytical solvent for LC/MS analysis. Depending on the analytical range, dilutions may be made using a diluter or syringe to make successive serial dilutions using a suitable analytical solvent to reach the desired concentration (e.g. 1:10 or higher sample:methanol/acetone serial dilutions), and an appropriate internal standard (e.g. 1H,1H,2H,2H tetrahydroperfluorooctane sulphonic acid (THPFOS)) will be added to the final dilution prior to analysis. Samples are to be analyzed via LC/MS against a standard curve containing the test substance and an appropriate internal standard.

3.0 DEFINITIONS

- 3.1 **Method blank:** An analyte-free matrix (e.g. methanol, water, or acetone) to which all reagents agree are added in the same volumes or proportions as used in the sample processing. The method blank is used to document contamination resulting from the entire sample treatment and analytical process. The method blank is carried through the complete sample preparation, treatment, and analytical procedure.
- 3.2 **Solvent blank:** A sample of analyte-free medium (e.g. methanol/water/acetone solution) that is not taken through the sample treatment process. This blank is used to evaluate instrument and reagent contamination.
- 3.3 **Shake Flask Sample Triplicates:** Three test vessels taken from and representative of the same sample source carried through all steps of the treatment, extraction, and analytical procedures in an identical manner.
- 3.4 **Sample replicates:** Replicate samples (two or more) taken from and representative of the same sample source (e.g. test vessel containing test analyte and solvent) and separately carried through analytical procedures in an identical manner.
- 3.5 **Internal Standard (IS):** A known amount of a compound similar in analytical behavior to the compound(s) of interest, added to all samples and standards, and carried through the entire measurement process.

- 3.6 **Calibration Standard:** A dilution of various amounts of a stock, intermediate or purchased standard to achieve standard solutions in a concentration range of interest.

4.0 WARNINGS AND CAUTIONS

4.1 Health and Safety Warnings:

- 4.1.1 Wear the proper lab attire for all parts of these procedures. Wear gloves and eye protection at all times.
- 4.1.2 Handle all solvents in a hood for all parts of the described sample preparation procedure.
- 4.1.3 For potential hazards of each chemical used, refer to material safety data sheets, packing materials, and 3M Environmental Laboratory's Chemical Hazard Review.
- 4.1.4 No mouth pipetting is allowed.

4.2 Cautions:

- 4.2.1 Glassware in which standards are prepared are to be triple rinsed with acetone and methanol to reduce the possibility of accidental contamination.
- 4.2.2 All test vessels (e.g. centrifuge tubes) are to be sufficiently rinsed with solvent prior to their usage.

5.0 INTERFERENCES

- 5.1 Impurities may significantly affect the solubility of the test substance. The purity of the test substance should be known and documented prior to starting the preliminary solubility screening procedure.
- 5.2 Contaminants in solvents, reagents, glassware and other sample processing or analysis hardware may cause interference. The routine analysis of laboratory method blanks must be used to demonstrate that there is no interference under the conditions of the analysis.

6.0 EQUIPMENT

- 6.1 Analytical balance sensitive to 0.1 mg.
- 6.2 Centrifuge capable of holding 15 mL centrifuge tubes or equivalent.
- 6.3 Incubator with heating/cooling capabilities.
- 6.4 Diluter, Hamilton Microlab® 500 Series, or equivalent.
- 6.5 Vortex-mixer.

7.0 SUPPLIES AND MATERIALS

- 7.1 Thermometer capable of reading at least 15 °C-40 °C.
- 7.2 5-250 mL polypropylene centrifuge tubes, or equivalent.
- 7.3 Disposable glass graduated pipettes, 1 mL to 10 mL.
- 7.4 Disposable glass pasteur pipettes and rubber bulbs.
- 7.5 Glass beakers, various sizes.
- 7.6 Crimp cap autovials-1.5 mL, caps, crimper, and decapper.
- 7.7 Hamilton Gastight® syringes (precision $\pm 1\%$ of the total volume), 5 μL to 1000 μL .
- 7.8 Pipette-man manual pipettor.

8.0 SUPPLIES AND MATERIALS

- 8.1 **Methanol (MeOH)**, HPLC/SPEC/GC grade from EM Science, or equivalent.
- 8.2 **Acetone**, HPLC/SPEC/GC grade from EM Science or equivalent.
- 8.3 **Water**, ASTM Type 1.
- 8.4 **Test substance of known purity**.
- 8.5 **Calcium Chloride Dihydrate**, Approximately 99% or better, from Sigma.
- 8.6 **0.01 M CaCl₂ solution**, Example: A 0.01 M CaCl₂ stock solution is prepared by weighing 1.4 g CaCl₂ in a weigh boat and transferring to a 1 L volumetric flask and diluting to the mark with Milli-QTM water.

9.0 SAMPLE HANDLING

- 9.1 Record times of initial preparation, set-up, and sample aliquoting/analysis on a sample preparation sheet or logbook.
- 9.2 Once the samples have been diluted, they may be analyzed via LC/MS. Alternatively, the diluted samples may be kept in cold storage (e.g. approximately 1-5 °C) in crimp-capped autovials until the time of analysis.

10.0 QUALITY CONTROL

- 10.1 **Shake Flask Sample Triplicates**. Set up each test vessel in triplicate to provide a measure of the precision on sample preparation.
- 10.2 **Sample Replicates**. Prepare and analyze all samples (from each test vessel for each time point) in multiple replicates (2 or more) to provide a measure of the precision on analysis.
- 10.3 **Quality Control Blank Samples**.
 - 10.3.1 **Method blank**. Set up a fourth test vessel without test substance to measure any contamination accrued throughout the sample preparation process. The method blank is carried through the same sample preparation procedures as the three test vessels with test substance, only no test substance is added throughout the entire procedure.
 - 10.3.2 **Solvent Blank**. An aliquot of the dilution solvent (i.e. methanol) directly analyzed for possible contaminants. The solvent blank is used to document any possible contamination of the solvent(s) used during the sample preparation process, and to detect any instrumental contamination or background interferences.

11.0 CALIBRATION AND STANDARIZATION

- 11.1 The compounds of interest must be standardized according to laboratory specifications.
- 11.2 All equipment used, such as the analytical balance and automated diluter, should be calibrated prior to use (daily, weekly, etc.) as specified in its standard operating procedure.

- 11.3 All samples analyzed will be run against a standard curve containing varying amounts of test substance, and a fixed amount of internal standard.

12.0 CALIBRATION AND STANDARIZATION

12.1 Preparation of Test Vessel. Record all data and observations on the example sample preparation worksheet (Attachment A) or equivalent.

12.1.1 Prepare a solution at least five times more concentrated than the concentration at which the substance was known to be dissolved at in the preliminary solubility screen test (ETS-8-170.0).

12.1.2 Weigh out test substance into a tared test vessel (e.g. 15 mL polypropylene centrifuge tube).

12.1.3 Add the test solvent to the test vessel gravimetrically until the desired volume has been obtained (*note: solvents have differing densities, therefore the weight needed to achieve a specific volume will be dependent on the density.*). Record all weights on a standardized preparation sheet or logbook.

12.1.4 Visually confirm that there are undissolved particles. If no particles are present, repeat 12.1.2 with more test substance or add more test substance and record the additional weight.

12.1.5 Prepare the solution described in 12.1.1-12.1.4 nine times and label (at minimum) the test vessels as Day 1, -Rep 1, -Rep 2, and -Rep 3; Day 2, -Rep 1, -Rep 2, and -Rep 3; and Day 3, -Rep 1, -Rep 2, and -Rep 3. Also include the day of initial sample prep, the person(s) responsible for the sample, the test compound, nature of the study (e.g. water solubility), the solvent utilized, and the study number.

12.1.6 Prepare three test vessels as described in 12.1.3 without the test substance and label (at minimum) as Day1,Rep4; Day2,Rep4; and Day3,Rep4 along with the information described under 12.1.5. These three test vessels are the "method blank" samples.

12.1.7 Seal the cap to the test vessel. Mix/shake test vessel to ensure contact between the test substance and the solvent. And wrap the cap with tape.

12.2 Equilibration of samples.

12.2.1 Place all of the test vessels on their side (horizontal) in an orbital incubator set to approximately $30\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ and rotating at a considerable speed to ensure sufficient contact/mixing between the test substance and solvent.

12.2.2 At approximately 24 hours, four test vessels are removed: three containing test substance labeled "Day 1,Rep1", "Day1,Rep2", "Day1,Rep3", and "Day1,Rep4".

12.2.3 The samples are shaken to ensure no test substance is stuck to the side of the tube.

12.2.4 The tubes are then placed in a stable temperature environment of approximately $20\text{--}26\text{ }^{\circ}\text{C}$ (record the actual temperature) for about a 24 hour period.

12.2.5 After about 24 hours of equilibration at $20\text{ }^{\circ}\text{C}\text{--}26\text{ }^{\circ}\text{C}$, centrifuge the samples until the solution is visibly clear. If micelle formation is suspected, an additional high-speed centrifugation step may be added (20,000 RCF for approximately 1 hour). The test vessels are now ready for dilution. See section 12.3 for further sample preparation.

12.2.6 At approximately 48 hours of incubation time, the second set of test vessels may be pulled (labeled "Day2,Rep1", "Day2,Rep2", "Day2,Rep3", and "Day2,Rep4") and equilibrated as described in 12.2.3-12.2.5.

12.2.7 At approximately 72 hours, the final set of samples is pulled (labeled "Day3,Rep1", "Day3,Rep2", "Day3,Rep3", and "Day3,Rep4") and equilibrated as described in 12.2.3-12.2.5.

12.3 Sample preparation for analysis.

12.3.1 Observe the test solution. Due to the possibility of emulsions/or a concentrated layer of solute at the surface of the solution, it may be necessary to pipette off the top layer of solution (or an aliquot of sample may be centrifuged as stated in 12.2.5). The use of a pasteur pipette or equivalent is recommended.

12.3.2 Aliquot the test solution into each of four autovials (e.g. 1.5 mL polypropylene or glass crimp-cap vials).

12.3.2.1 Sample aliquots may be taken by submerging the pipette or syringe tip below the surface of the test solution. *Note: when selecting the type of pipette to be used, take into consideration the test substance's tendency to adsorb to certain materials (i.e. adsorption of test substance to glass or plastic).*

12.3.2.2 Prior to aliquoting sample to the autovial, the pipette tip must be equilibrated/saturated with the test solution by drawing and gently expelling the test solution in and out of the pipette tip, taking care not to disturb the solid particulate.

12.3.3 Label the four autovials and record the sample id.

12.3.4 Using a diluter, make a 1:10 or higher dilution of sample:extraction solvent (i.e. methanol, acetone) into a labeled autovial.

12.3.5 Using the solubility information obtained in the preliminary screen test, estimate the appropriate dilutions, if any, required to bring the concentration of sample into the appropriate analytical range (e.g. approximately 3 ng/mL to 3000 ng/mL test substance). Utilize the diluter to make serial dilutions of the sample using a suitable analytical solvent.

12.3.6 To the final analytical sample, add internal standard at a concentration suitable to the analytical method.

12.3.7 Samples will be analyzed via LC/MS against a standard curve of varied, known concentrations of test substance with a constant concentration of internal standard equal to the concentration in the samples.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Samples will be analyzed via a calibration curve. The amount of test substance in the sample will be quantified against a standard curve.

13.2 Means will be calculated by adding the individual entities and dividing the resultant sum by the number of individual entities.

13.3 Standard deviations will be calculated using either Microsoft Excel® or Microsoft Access® to calculate standard deviation. The built in function contains the following equation which is based on the individual entities (n) being less than 30:

$$\sqrt{\frac{n\sum x^2 - (\sum x)^2}{n(n-1)}}$$

- 13.4 Sample precision will be reported as %RSD (or %CV). Sample precision will be calculated using the following equation:

$$A/B \times 100 = \text{Sample \%RSD}$$

where: A= standard deviation of averaged samples

B= average of samples

Sample RPD will be calculated using the following equation:

$$|A-B| / ((A+B)/2) = \text{Sample RPD}$$

where: A= concentration of first replicate

B= concentration of second replicate

- 13.5 Exclusion of an outlier data point may be performed by utilizing Dixon's Q-Test. The questionable data point may err to the high or low end of the data set. Calculate the variable "Qobserved." If Qobserved > Qtabulated, then the data point may be rejected with 90% confidence (see table 1 below for Qtabulated values).

$$Q_{\text{observed}} = \text{gap}/\text{range}$$

where: Gap= the difference between the questionable point and the nearest value.

Range= total spread of the data.

Table 1 Criteria for Rejection of Outlier Values:

Number of observations	Q _{tabulated} , 90% Confidence Range
3	0.886
4	0.679
5	0.557

14.0 DATA ANALYSIS AND CALCULATIONS

- 14.1 **Precision of data.** Sample data must have a percent relative standard deviation (%RSD) (or relative percent difference) of $\leq 15\%$. Non-compliant data must be evaluated for obvious outliers. The Q-Test may be applied to exclude questionable data points. If an outlier value exists, sample average and precision is re-calculated and reported without the questionable data point. Document the non-compliant data on data summary sheets, and include results of the statistical analysis with the final results.

14.1.1 Sample replicate %RSD's should be $\leq 15\%$. If the average of the sample triplicate data is $> 15\%$ RSD, evaluate the three values for an outlier value. To questionable data points, apply the Dixon's Q-Test. If the outlier value is rejected with 90% confidence, exclude the sample from further data calculations and calculate the average and RPD for the remaining two values. If no data points can be excluded and the data does not meet the criteria, then the data set may not be used in the final reported data. The remaining shake flasks for the timepoint shall be used to report the data.

14.1.2 The %RSD between shake flasks should agree within 15%. If this criteria is not met, apply the Q-test to outlier datum. If the questionable data point is discarded

via the Q-Test, then the non-compliant shake flask data is not to be used for the timepoint. Include justification for dropping the shake flask (e.g. q-test results).

14.1.3 And finally, the %RSD between the three days should be $\leq 15\%$ for the shake flask method. If this criteria is not met, it may be necessary to re-evaluate the data for possible outliers. Three agreeable time intervals are required to report the final solubility. If three consecutive timepoints do not agree within 15%, it may be necessary to repeat the entire test, or re-dilute/re-shoot samples to check for error.

14.2 **Limit of Quantification.** For this study, the LOQ will be equal to the lowest calibration standard used in the calibration curve containing more than twice the area counts of the highest Quality Control Blank.

14.3 **Quality Control Blanks: Method and Solvent Blank samples.** The level of analyte (test analyte OR internal standard analyte) shall be less than 50% of the area counts of the LOQ. If background levels of analyte exist in the initial method blanks, but subsequent blanks prior to the calibration standard curve are clean, the data may be accepted (provided that there are no other indicators that either the samples or the instrument contain significant background levels of analyte).

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

15.1 Dispose of sample waste by placing in high or low BTU containers as appropriate. Use broken glass containers to dispose of glass pipettes.

16.0 RECORDS

16.1 Sign and date all observations and calculations.

16.2 Fill out all appropriate sample preparation worksheets.

17.0 ATTACHMENTS

17.1 Attachment A-Example Sample Preparation Worksheet.

18.0 REFERENCES

18.1 Organization for Economic Cooperation and Development. OECD Guideline for Testing of Chemicals. Water Solubility -OECD Guideline 105: pp. 1-7, Adopted 1995

18.2 United States Environmental Protection Agency. OPPTS 830.7860 Water Solubility (Generator Column Method). Prevention, Pesticides and Toxic Substances: Fate, Transport and Transformation Test Guidelines. EPA 712-C-96-042: pp. 1-17, 1996

18.3 United States Environmental Protection Agency. OPPTS 830.7840 Water Solubility: Column Elution Method; Shake Flask Method. Prevention, Pesticides and Toxic

Substances: Fate, Transport and Transformation Test Guidelines. EPA 712-C-96-041: pp. 1-12, 1996

- 18.4 ETS-8-170.0, Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents.
- 18.5 Harris, Daniel C. Statistics. *Quantitative Chemical Analysis*. 4th ed.; W.H. Freeman and Company: New York, 1982; p 70-71.
- 18.6 Natrella, Mary Gibbons. The Treatment of Outliers. *Experimental Statistics*, National Bureau of Standards Handbook 91; U.S. Government Printing Office: Washington, D.C., 1963; Pages 17-3, and T-27.

19.0 AFFECTED DOCUMENTS

- 19.1 ETS-8-170.0, "Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents."

20.0 REVISIONS

<u>Revision Number</u>	<u>Reason For Revision</u>	<u>Revision Date</u>
1	Section 1.2, references to specific test substances were removed. Section 3.6, removed "internal standard blank" from definition section. Section 12.2.5, an optional high-speed centrifugation step was added. Fixed the incorrect numbering of sections 13.5 through 14.3. Section 14.3, more clearly defined the acceptance criterion for method and solvent blanks.	03-27-01

ATTACHMENT A: Example Sample Preparation Sheet (page 1 of 2).

3M Environmental Laboratory Solubility Test: Shake Flask Method				Study # _____
Sample preparation worksheet				Analyst(s): _____
Temperature of Study: _____ °C		ID#: _____	Date: _____	
Test Substance: _____		Source: _____		
Test Solvent: _____		ID#: _____	Test Vessel: _____ 15ml centrifuge tube/Other(specify): _____	
solvent density: _____ g/ml		Source: _____		
Was the solubility screen test performed prior to start? Y/N If so, what is the approximate concentration? _____ ug/ml				
Balance ID: _____		Thermometer ID: _____	Room Temp: _____ °C	Date/Time/Initials: _____
SFM vials ID#: _____	weight test substance added:	Total mass (solute + solvent) added:	¹ Internal Standard addition ul / ml soln	IS added to dilution factor:
"sampleID" = _____	Sample Description			Observations/Comments:
sampleID-1.1.1 thru -1.1.4	Day1, Smp1		1:	
sampleID-1.2.1 thru -1.2.4	Day1, Smp2		1:	
sampleID-1.3.1 thru -1.3.4	Day1, Smp3		1:	
sampleID-1.4.1 thru -1.4.4	Day1, Smp4 blank	none added	1:	
sampleID-2.1.1 thru -2.1.4	Day2, Smp1		1:	
sampleID-2.2.1 thru -2.2.4	Day2, Smp2		1:	
sampleID-2.3.1 thru -2.3.4	Day2, Smp3		1:	
sampleID-2.4.1 thru -2.4.4	Day2, Smp4 blank	none added	1:	
sampleID-3.1.1 thru -3.1.4	Day3, Smp1		1:	
sampleID-3.2.1 thru -3.2.4	Day3, Smp2		1:	
sampleID-3.3.1 thru -3.3.4	Day3, Smp3		1:	
sampleID-3.4.1 thru -3.4.4	Day3, Smp4 blank	none added	1:	
Date/Time: _____				
Initials: _____				
Test Vessels sealed w/ tape? Y/N		Test Vessels shaken/vortex-mixed? Y/N		¹ Internal Standard: _____ ID#: _____
Test vessels placed on orbital incubator? Y/N		Incubator ID: _____ Speed: _____ rpm		Int. Std. Conc.: _____ ug/ml
		Initial Temperature: _____ °C		
Day 1:				
Four "Day 1" samples are pulled and allowed to be placed in a temperature controlled environment for approx. 24 hours with no agitation.				Date/Time/Initials: _____
Location of samples: _____		Temperature of environment: _____ °C		Thermometer ID: _____
Visual observations: _____				

ATTACHMENT A CONTINUED (page 2 of 2).

Day 2:

Four "Day 2" samples are pulled and allowed to be placed in a temperature controlled environment for approx. 24 hours with no agitation.

Date/Time/Initials: _____

Location of samples: _____ Temperature of environment: _____ °C Thermometer ID: _____

Visual observations: _____

"Day 1" Test Vessels Centrifuged: Cent. ID: _____ RPM or RCF readout: _____ duration: _____ min.

Date/Time/Initials: _____

Visual observations: _____

Four aliquots from each of the four samples are to be made into glass/plastic autovials (also, check autovials for correct labeling). Yes/No

Immediately dilute the solution 1: _____ with methanol. Methanol TN-A: _____ Date/Time/Initials: _____

Additional dilutions: Dilution factor (test solution:methanol): 1: _____ X's how many dilutions: _____ =final dilution factor: 1: _____

Diluter ID used: _____ Amounts used: _____ uL methanol, _____ uL samples

Store samples and extracts in a cooler at until time of analysis. Cooler ID: _____ Cooler Temp: _____ °C Date/Time/Initials: _____

Day 3:

Four "Day 3" samples are pulled and allowed to be placed in a temperature controlled environment for approx. 24 hours with no agitation.

Date/Time/Initials: _____

Location of samples: _____ Temperature of environment: _____ °C Thermometer ID: _____

Visual observations: _____

"Day 2" Test Vessels Centrifuged: Cent. ID: _____ RPM or RCF readout: _____ duration: _____ min.

Date/Time/Initials: _____

Visual observations: _____

Four aliquots from each of the four replicates are to be made into glass/plastic autovials (also, check autovials for correct labeling). Yes/No

Immediately dilute the solution 1: _____ with methanol. Methanol TN-A: _____ Date/Time/Initials: _____

Additional dilutions: Dilution factor (test solution:methanol): 1: _____ X's how many dilutions: _____ =final dilution factor: 1: _____

Diluter ID used: _____ Amounts used: _____ uL methanol, _____ uL samples

Store samples and extracts in a cooler at until time of analysis. Cooler ID: _____ Cooler Temp: _____ °C Date/Time/Initials: _____

Day 4:

At approx. 24 hours of equilibration, the "Day 3" samples are to be aliquoted/extracted. Environment Temp.: _____ °C Therm. ID: _____

Test Vessels Centrifuged: Cent. ID: _____ RPM or RCF readout: _____ duration: _____ min. Date/Time/Initials: _____

Visual observations: _____

Four aliquots from each of the four samples are to be made into glass/plastic autovials (also, check autovials for correct labeling). Yes/No

Immediately dilute the solution 1: _____ with methanol. Methanol TN-A: _____ Date/Time/Initials: _____

Additional dilutions: Dilution factor (test solution:methanol): 1: _____ X's how many dilutions: _____ =final dilution factor: 1: _____

Diluter ID used: _____ Amounts used: _____ uL methanol, _____ uL samples

Store samples and extracts in a cooler at until time of analysis. Cooler ID: _____ Cooler Temp: _____ °C Date/Time/Initials: _____

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROchemicals IN WASTE STREAM OR WATER EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-155.0

Adoption Date: 11/10/00

Revision Date:

Author: Mark L. Anderson, Mark E. Ellefson

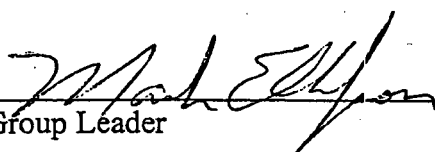
Approved By:



Laboratory Manager

11/10/00

Date



Group Leader

11/10/00

Date

Exact Copy of Original

MA
Initial

03 Apr 01
Date

1.0 SCOPE AND APPLICATION

- 1.1 Scope:** This method describes the analysis of waste stream or water extracts using HPLC-electrospray/mass spectrometry.
- 1.2 Applicable Compounds:** Fluorochemicals or other electrospray ionizable compounds.
- 1.3 Matrices:** Tap water, ground water, wastewater and other aqueous solutions.

2.0 SUMMARY OF METHOD

- 2.1** This method describes the analysis of fluorochemicals or other electrospray ionizable compounds extracted from water, using HPLC-electrospray mass spectrometry (HPLC-ES/MS). The analysis is performed by the mass selection of a single ion characteristic of a particular compound, such as the perfluorooctanesulfonate (PFOS) anion, $m/z = 499$ or perfluorooctanoate (PFOA), $m/z = 413$.

3.0 DEFINITIONS

- 3.1 Atmospheric Pressure Ionization (API):** The Micromass Platform LCZ single quadrupole system and other commercially available LC/MS systems allow for various methods of ionization by utilizing a variety of sources, probes, and interfaces. These include but are not limited to: Electrospray Ionization (ESI), Atmospheric Pressure chemical Ionization (APCI), Thermospray, etc. The ionization in these processes occurs at atmospheric pressure (i.e., not under a vacuum).
- 3.2 Electrospray Ionization (ES, ESI):** A method of ionization performed at atmospheric pressure, whereby ions in solution are transferred to the gas phase via tiny charged droplets. These droplets are produced by the application of a strong electrical field.
- 3.3 Mass Spectrometer (MS):** The Platform LCZ and other commercially manufactured ES/MS systems are equipped with a single quadrupole mass selective detector. Ions are selectively discriminated by mass to charge ratio (m/z) and subsequently detected.
- 3.4 Conventional vs. Z-spray probe interface:** The Micromass Platform LCZ system utilizes a "Z-spray" conformation. The spray emitted from the probe is orthogonal to the cone aperture. In the conventional conformation it is aimed directly at the cone aperture, after passing through a tortuous pathway in the counter electrode. Though the configuration is different, the methods of operation, cleaning, and maintenance are the same. However, Z-spray components and conventional components are not compatible with one another, but only with similar systems (i.e., Z-spray components are compatible with some other Z-spray systems, etc.). Other commercially manufactured ES/MS systems may have similar features.
- 3.5 Mass Lynx Software:** System software designed for the specific operation of Micromass LCZ Mass spectrometer. Currently MassLynx has Windows 95 and WindowsNT 4.0 versions. All versions are similar. For more details see the manual specific to the instrument (MassLynx NT User's Guide or Micromass Platform LCZ User's Guide).

4.0 WARNINGS AND CAUTIONS

4.1 Health and Safety Warnings:

- 4.1.1 Use caution with the voltage cables for the probe. When engaged, the probe employs a voltage of approximately 5000 Volts.
- 4.1.2 When handling samples or solvents wear appropriate protective clothing, gloves, and eyewear.

4.2 Cautions:

- 4.2.1 Operate solvent pumps below a backpressure of 400 bar (5800 psi). If the backpressure exceeds 400 bar, the HP1100 will initiate automatic shutdown.
- 4.2.2 Do not run solvent pumps to dryness.

5.0 Interferences

- 5.1 To minimize interferences when analyzing samples, Teflon should not be used for sample storage or any part of instrumentation that comes in contact with the sample or extract.

6.0 EQUIPMENT

- 6.1 Equipment listed below may be modified in order to optimize the system. Document any modifications in the raw data as method deviations.
 - 6.1.1 Micromass Platform LCZ Mass Spectrometer equipped with an electrospray ionization source.
 - 6.1.2 HP1100 low pulse solvent pumping system, solvent degasser, column compartment, and autosampler.

7.0 SUPPLIES AND MATERIALS

7.1 Supplies

- 7.1.1 High purity grade nitrogen gas regulated to approximately 100 psi (or house air system.).
- 7.1.2 HPLC analytical column, such as a Betasil C18 column (50x2mm, 5 μ m particle size) or equivalent.
- 7.1.3 Capped autovials or capped 15 mL centrifuge tubes.

8.0 REAGENTS AND STANDARDS

8.1 Reagents

- 8.1.1 Methanol, HPLC grade or equivalent.
- 8.1.2 Milli-Q™ water (ASTM type I), all water used in this method should be Milli-Q™ water or equivalent, and may be provided by a Milli-Q TOC Plus system or other vendor.
- 8.1.3 Ammonium acetate, reagent grade or equivalent.
 - 8.1.3.1 When preparing different amounts than those listed, adjust accordingly.
 - 8.1.3.2 2.0 mM ammonium acetate solution: Weigh approximately 0.300 g ammonium acetate. Pour into a 2000 L volumetric flask, add the appropriate volume of Milli-Q water, mix until all solids are dissolved. Store at room temperature.

8.2 Calibration Standards

- 8.2.1 Typically two method blanks (Milli-Q water), two matrix blanks, and solvent standards are prepared during the sample extraction procedure.

9.0 SAMPLE HANDLING

- 9.1 Standards and sample extracts are stored in capped autovials or capped 15 mL centrifuge tubes until analysis.
- 9.2 If analysis will be delayed, standards and sample extracts may be refrigerated at approximately 4° C until analyses can be performed.

10.0 QUALITY CONTROL

10.1 Solvent Blanks, Method Blanks and Matrix Blanks

- 10.1.1 Solvent blanks, method blanks, and matrix blanks are prepared and analyzed with each sample set to determine contamination or carryover.
- 10.1.2 Analyze a method blank and a matrix blank prior to each calibration curve.

10.2 Matrix Spikes

- 10.2.1 Matrix spikes are prepared for each sample set and analyzed to determine the matrix effect on the recovery efficiency.
- 10.2.2 Matrix spike duplicates are prepared periodically to measure the precision associated with the analysis.
- 10.2.3 Analyze the matrix spike and matrix spike duplicate (if prepared) in the same run as the original sample.
- 10.2.4 Matrix spike and matrix spike duplicate concentrations should fall in the mid-range of the initial calibration curve or should be prepared at 1.5-5 times the endogenous

concentration of the analyte. Spike concentrations should fall in the low-range of the initial calibration curve if extremely low levels are expected.

10.3 Continuing Calibration Verifications

10.3.1 Continuing calibration verifications (CCV) are analyzed to verify the continued accuracy of the calibration curve.

10.3.2 Analyze a mid-range calibration standard after every tenth sample, with a minimum of one per sample set.

10.4 Internal Standard/Surrogate Standard

10.4.1 An internal standard (IS) may be used to quantify the target analytes by establishing a relationship between the ratio of analyte response to IS response and a known concentration of the analyte of interest. The IS should be spiked at an amount that will fall within the mid-range of the calibration curve. The IS should be added after the extraction process and before analysis.

10.4.2 A surrogate standard may be used for quality control. The surrogate is used to quantitatively evaluate the entire analytical procedure including sample preparation and analysis. The surrogate should be spiked to fall within the low to mid-range of the calibration curve.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Analyze the standard curves prior to and following each set of extracts. The average of two standard curves may be plotted by linear regression ($y = mx + b$) weighted $1/x$, or quadratic fit ($y = ax^2 + bx + c$) using MassLynx or other suitable software. The calibration curves should not be forced through zero.

11.2 If the calibration curve does not meet acceptance criteria perform routine maintenance or prepare a new standard curve (if necessary) and reanalyze.

11.3 For purposes of accuracy when quantitating low levels of analyte, it may be necessary to use the low end of the calibration curve rather than the full range. Example: when attempting to quantitate approximately 10 ppb of analyte, generate a calibration curve consisting of the standards from 5 ppb to 100 ppb rather than the full range of the curve (5 ppb to 1000 ppb). This will reduce inaccuracy attributed to linear regression weighting of high concentration standards.

12.0 PROCEDURES

12.1 Acquisition Set up

12.1.1 Set up the sample list.

12.1.1.1 Assign a sample list filename using the first letter of the name of the instrument (T for Tucker), the year (00 for 2000), the month (04 for April), and the day (T001012 for October 12, 2000). If more than one list is made on the same day, use increasing letters of the alphabet starting with A at the end of the list.

- 12.1.1.2 Assign a method (MS) file.
- 12.1.1.3 Assign an HPLC program (Inlet file).
- 12.1.1.4 Type in sample descriptions and vial position numbers.

12.1.2 To create a method, click on method in the Acquisition control panel then mass spectrometer headings and select SIR. Set ionization mode as appropriate and mass to 499 or other appropriate masses. A full scan is usually collected in addition to the SIRs. Save acquisition method. See the Micromass MassLynx GUIDE TO DATA ACQUISITION for additional information.

12.1.3 Typically the analytical batch run sequence begins and ends with a set of solvent standards.

12.1.4 Samples are analyzed with a continuing calibration verification (CCV) injected after every tenth sample. Solvent blanks should be analyzed periodically to monitor for possible analyte carryover.

12.2 Using the Autosampler/Column Heater

12.2.1 Place sample vials into the sample tray according to the sample list prepared in Section 12.1.1.

12.2.2 Attach the proper analytical column in the column heater compartment. If using the switching valve, make sure that the tubing is run to the appropriate ports.

12.3 Using the Inlet Editor

12.1.1 Set-up the HP1100 using the following conditions or at conditions the analyst considers appropriate for optimal response. Record actual conditions in the instrument logbook:

12.1.1.1 Sample size = 10 μ L injection

12.1.1.2 Flow rate = 300 μ L/min.

12.1.1.3 Cycle time = 10.0 minutes

12.1.1.4 Mobile phase components:

Solvent A: 2.0 mM Ammonium Acetate

Solvent B: Methanol (MeOH)

Solvent Gradient:	<u>Time (min.)</u>	<u>% B</u>
	0.00	5.00 %
	1.00	5.00 %
	4.50	95.0 %
	8.00	95.0 %
	8.50	5.00 %
	10.0	Stop

12.4 Instrument Set-up

- 12.4.1 Refer to the Platform LCZ User's Guide, the MassLynx NT User's Guide or ETS-9-36, "Operation and Maintenance of the Micromass Platform LCZ Electrospray/Mass Spectrometer".
- 12.4.2 Check the solvent level in reservoirs and refill if necessary.
- 12.4.3 Check the tip of the stainless steel capillary at the end of the probe with an eyepiece. The tip should be flat with no jagged edges. If the tip is found to be unsatisfactory, disassemble the probe and replace the stainless steel capillary.
- 12.4.4 Turn on the nebulizing gas.
- 12.4.5 Open the tune page. Click on 'Operate' to initiate the desolvation heaters.
- 12.4.6 Open the Inlet Editor.
 - 12.4.5.1 Set HPLC pump to "On".
 - 12.4.5.2 Set the solvent flow to the desired flow rate.
 - 12.4.5.3 Observe droplets coming out of the tip of the probe. A fine mist should be expelled with no nebulizing gas leaking around the tip of the probe. Readjust the tip of the probe if no mist is observed.
 - 12.4.5.4 Allow to equilibrate for at least 10 minutes.
- 12.4.6 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:
 - 12.4.6.1 Drying gas 250-425 liters/hour
 - 12.4.6.2 ESI nebulizing gas 10-15 liters/hour
 - 12.4.6.3 HPLC constant flow mode, flow rate 10 – 500 $\mu\text{L}/\text{min}$
 - 12.4.6.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the HPLC is operating correctly.)
 - 12.4.6.5 Source Block temperature 150°.
 - 12.4.6.6 Desolvation temperature 250°.
- 12.4.7 Print the tune page with its parameters, the Inlet page, sample list, mass spec information, and all other applicable information and store it in the study binder with copies taped into the instrument run logbook.
 - 12.4.7.1 All copies must be initialed and dated.
- 12.4.8 Click on start button on the MassLynx toolbar. Ensure start and end sample numbers include all samples to be analyzed.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

- 13.1.1 Calculate matrix spike percent recoveries using the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed Result} - \text{Background Result}}{\text{Expected Result}} \times 100$$

13.1.2 Calculate percent difference using the following equation:

$$\% \text{ Difference} = \frac{\text{Expected Conc.} - \text{Calculated Conc.}}{\text{Expected Conc.}} \times 100$$

13.1.3 Calculate actual concentration of analyte in matrix (µg/mL):

$$\text{On-Column Concentration (µg/mL)} \times \text{Dilution Factors} = \text{Calculated Concentration}$$

14.0 METHOD PERFORMANCE

- 14.1 The Limit of Quantitation (LOQ) is method, analyte, and matrix specific. For many analytes, the LOQ concentration is selected as the lowest acceptable non-zero standard in the calibration curve.
- 14.2 Solvent and method blank values must be < ½ that of the lowest standard used in the calibration curve.
- 14.3 The coefficient of determination (r^2) value for the calibration curve must be greater than or equal to 0.980.
- 14.4 Continuing Calibration Verification (CCV) percent recoveries must be ± 30% of the standard concentration.
- 14.5 Internal Standard recoveries should be within ± 50% of the spiked concentration.
- 14.6 If criteria listed in this method performance section are not met, maintenance may be performed on the system and samples reanalyzed or other actions as determined by the analyst. Document all actions in the raw data.
- 14.7 If data is to be reported when performance criteria have not been met, the data must be footnoted on tables and discussed in the text of the report.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 15.1 Sample extract waste and flammable solvent is disposed in high BTU containers, and glass pipette waste is disposed in broken glass containers located in the laboratory.

16.0 RECORDS

- 16.1 Each page generated for a study must have the following information included either in the header or hand written on the page: study or project number, acquisition method, integration method, sample name, extraction date, dilution factor (if applicable), and analyst.
- 16.2 Print the tune page, sample list, and acquisition method from MassLynx and other applicable information to include in the appropriate study folder. Copy these pages and tape into the instrument runlog.
- 16.3 Plot the calibration curve then print these graphs and store in the study folder.

- 16.4 Print data integration summary, integration method, and chromatograms, from MassLynx, and store in the study folder.
- 16.5 Summarize data using suitable software and store in the study folder.
- 16.6 Back up electronic data to appropriate medium. Record in study notebook the file name and location of backup electronic data.

17.0 ATTACHMENTS

17.1 None

18.0 REFERENCES

- 18.1 Platform LCZ User's Guide, Micromass UK Limited, Tudor Road, Altrincham, WA14 5RZ; or Floats Road, Wythenshawe M23 9LZ; United Kingdom.
- 18.2 MassLynx NT User's Guide, Micromass UK Limited, Tudor Road, Altrincham, WA14 5RZ; or Floats Road, Wythenshawe M23 9LZ; United Kingdom.
- 18.3 MassLynx NT Guide To Data Acquisition, Micromass UK Limited, Tudor Road, Altrincham, WA14 5RZ; or Floats Road, Wythenshawe M23 9LZ; United Kingdom.
- 18.4 ETS-9-36.0, "Operation and Maintenance of the Micromass Platform LCZ Electrospray/Mass Spectrometer".

19.0 AFFECTED DOCUMENTS

19.1 None

20.0 REVISIONS

<u>Revision Number.</u>	<u>Reason For Revision</u>	<u>Revision Date</u>
-----------------------------	----------------------------	--------------------------

ATTACHMENT B: DATA SUMMARY TABLES

TCR-002, 012201-swPFOS-1.4.4, 100X Dil.													
swPFOS-1.4.4 Method Blank, SFM 01-24-01 MEE(MLA)	43	H0125A1.M	HILL0043.D	24	4.11	2,194,236.0	254.0	4.94	1,141,147.0	254.4	0.00	0.0	0.0
MeOH Blank TN-A-4740, 04 Dec 00	44	H0125A1.M	HILL0044.D	93	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
TCR-002, 012201-swPFOS-1.1.1, 100X Dil.													
swPFOS-1.1.1 SFM 01-24-01 MEE(MLA)	45	H0125A1.M	HILL0045.D	31	4.11	2,102,494.3	254.0	4.92	1,112,681.4	254.4	5.14	3,780,994.0	125.7
TCR-002, 012201-swPFOS-1.1.2, 100X Dil.													
swPFOS-1.1.2 SFM 01-24-01 MEE(MLA)	46	H0125A1.M	HILL0046.D	32	4.11	2,158,897.3	254.0	4.93	1,116,756.6	254.4	5.14	3,919,050.5	129.9
TCR-002, 012201-swPFOS-1.1.3, 100X Dil.													
swPFOS-1.1.3 SFM 01-24-01 MEE(MLA)	47	H0125A1.M	HILL0047.D	33	4.11	2,209,667.8	254.0	4.94	1,119,839.4	254.4	5.14	3,895,319.3	128.8
TCR-002, 012201-swPFOS-1.1.4, 100X Dil.													
swPFOS-1.1.4 SFM 01-24-01 MEE(MLA)	48	H0125A1.M	HILL0048.D	34	4.11	2,201,234.8	254.0	4.94	1,126,401.0	254.4	5.14	4,009,264.5	131.9
01003-07-09 100.2ppb PFOS, CCV	49	H0125A1.M	HILL0049.D	9	4.11	2,265,761.0	254.0	4.86	1,118,630.0	254.4	5.14	3,109,402.5	102.2
MeOH Blank TN-A-4740, 04 Dec 00	50	H0125A1.M	HILL0050.D	93	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank TN-A-4740, 04 Dec 00	51	H0125A1.M	HILL0051.D	93	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
TCR-002, 012201-swPFOS-1.2.1, 100X Dil.													
swPFOS-1.2.1 SFM 01-24-01 MEE(MLA)	52	H0125A1.M	HILL0052.D	35	4.11	2,127,831.0	254.0	4.92	1,118,494.1	254.4	5.14	3,576,569.5	118.0
TCR-002, 012201-swPFOS-1.2.2, 100X Dil.													
swPFOS-1.2.2 SFM 01-24-01 MEE(MLA)	53	H0125A1.M	HILL0053.D	36	4.11	2,140,157.8	254.0	4.94	1,113,286.9	254.4	5.14	3,702,940.0	122.9
TCR-002, 012201-swPFOS-1.2.3, 100X Dil.													
swPFOS-1.2.3 SFM 01-24-01 MEE(MLA)	54	H0125A1.M	HILL0054.D	37	4.11	2,234,175.8	254.0	4.94	1,141,315.6	254.4	5.14	3,689,192.8	119.4
TCR-002, 012201-swPFOS-1.2.4, 100X Dil.													
swPFOS-1.2.4 SFM 01-24-01 MEE(MLA)	55	H0125A1.M	HILL0055.D	38	4.11	2,133,177.8	254.0	4.94	1,121,531.1	254.4	5.14	3,597,279.5	118.4
MeOH Blank TN-A-4740, 04 Dec 00	56	H0125A1.M	HILL0056.D	94	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
TCR-002, 012201-swPFOS-1.3.1, 100X Dil.													
swPFOS-1.3.1 SFM 01-24-01 MEE(MLA)	57	H0125A1.M	HILL0057.D	39	4.11	2,171,835.0	254.0	4.94	1,127,920.3	254.4	5.14	3,797,896.8	124.5
TCR-002, 012201-swPFOS-1.3.2, 100X Dil.													
swPFOS-1.3.2 SFM 01-24-01 MEE(MLA)	58	H0125A1.M	HILL0058.D	40	4.11	2,169,104.3	254.0	4.94	1,115,169.9	254.4	5.14	3,862,721.8	128.2
TCR-002, 012201-swPFOS-1.3.3, 100X Dil.													
swPFOS-1.3.3 SFM 01-24-01 MEE(MLA)	59	H0125A1.M	HILL0059.D	41	4.11	2,169,278.8	254.0	4.94	1,100,957.0	254.4	5.14	3,785,649.3	127.2
TCR-002, 012201-swPFOS-1.3.4, 100X Dil.													
swPFOS-1.3.4 SFM 01-24-01 MEE(MLA)	60	H0125A1.M	HILL0060.D	42	4.11	2,124,585.3	254.0	4.92	1,098,603.0	254.4	5.14	3,743,934.3	126.1
01003-07-09 100.2ppb PFOS, CCV	61	H0125A1.M	HILL0061.D	9	4.11	2,256,450.0	254.0	4.86	1,132,609.1	254.4	5.14	3,095,466.3	100.4
MeOH Blank TN-A-4740, 04 Dec 00	62	H0125A1.M	HILL0062.D	94	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank TN-A-4740, 04 Dec 00	63	H0125A1.M	HILL0063.D	94	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank TN-A-4740, 04 Dec 00	64	H0125A1.M	HILL0064.D	94	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
01003-07-01 0ppb PFOS	65	H0125A1.M	HILL0065.D	1	4.11	2,223,529.8	254.0	4.86	1,103,581.8	254.4	0.00	0.0	0.0
01003-07-02 2.5ppb PFOS	66	H0125A1.M	HILL0066.D	2	4.11	2,218,475.3	254.0	4.86	1,145,372.3	254.4	5.14	89,005.9	2.0
01003-07-03 5ppb PFOS	67	H0125A1.M	HILL0067.D	3	4.11	2,246,805.0	254.0	4.86	1,132,338.8	254.4	5.14	157,410.9	4.2
01003-07-04 10ppb PFOS	68	H0125A1.M	HILL0068.D	4	4.11	2,228,379.8	254.0	4.86	1,155,401.0	254.4	5.14	294,185.4	8.5
01003-07-05 25ppb PFOS	69	H0125A1.M	HILL0069.D	5	4.11	2,213,923.0	254.0	4.86	1,134,933.8	254.4	5.14	762,781.4	23.7
01003-07-06 40ppb PFOS	70	H0125A1.M	HILL0070.D	6	4.11	2,240,633.3	254.0	4.86	1,146,786.6	254.4	5.14	1,228,505.9	38.4
01003-07-07 50.1ppb PFOS	71	H0125A1.M	HILL0071.D	7	4.11	2,212,715.5	254.0	4.86	1,137,843.9	254.4	5.14	1,540,320.4	48.8
01003-07-08 75.1ppb PFOS	72	H0125A1.M	HILL0072.D	8	4.11	2,188,168.8	254.0	4.86	1,132,896.5	254.4	5.14	2,309,656.3	74.3
01003-07-09 100.2ppb PFOS	73	H0125A1.M	HILL0073.D	9	4.11	2,235,874.0	254.0	4.86	1,130,818.8	254.4	5.14	3,059,424.8	99.4
01003-07-10 250.5ppb PFOS	74	H0125A1.M	HILL0074.D	10	4.11	2,186,509.0	254.0	4.86	1,132,445.8	254.4	5.14	7,190,109.5	241.2
01003-07-11 400.8ppb PFOS	75	H0125A1.M	HILL0075.D	11	4.11	2,224,174.0	254.0	4.86	1,115,520.0	254.4	5.14	11,059,693.0	389.1
01003-07-12 501.0ppb PFOS	76	H0125A1.M	HILL0076.D	12	4.11	2,213,476.0	254.0	4.86	1,120,492.8	254.4	5.15	13,505,453.0	482.9
01003-07-13 1002.0ppb PFOS	77	H0125A1.M	HILL0077.D	13	4.11	2,203,661.3	254.0	4.86	1,072,897.5	254.4	5.14	23,746,392.0	1,000.9
MeOH Blank TN-A-4740, 04 Dec 00	78	H0125A1.M	HILL0078.D	94	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank TN-A-4740, 04 Dec 00	79	H0125A1.M	HILL0079.D	95	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0

2.0%

0.2%

19.6%

15.2%

15.6%

5.4%

4.2%

2.6%

1.1%

0.8%

3.7%

2.9%

3.6%

0.1%

PFOS in Natural Seawater
Shake Flask Method
Day 2

RAW DATA

Agilent 1100 HPLC/MSD "Hilary"

Acquisition Run: H010126

Raw Data

Sample Description	Run #	Sample Name	Peak #	Area	Height	Retention Time (min)	Response	Concentration (ppb)	Concentration (ppb)	Concentration (ppb)	Concentration (ppb)	Concentration (ppb)	Concentration (ppb)	Concentration (ppb)
MeOH Blank	TN-A-4740, 04 Dec 00	1	H010126C.M	HILL0137.D	98	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	2	H010126C.M	HILL0138.D	98	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
01003-07-01	0ppb PFOS	3	H010126C.M	HILL0139.D	46	4.11	2096939.6	254.0	4.92	1174597.3	254.4	0.00	0.0	0.0
01003-07-02	2.5ppb PFOS	4	H010126C.M	HILL0140.D	47	4.11	2130418.5	254.0	4.92	1173146.4	254.4	5.14	81704.0	2.2
01003-07-03	5ppb PFOS	5	H010126C.M	HILL0141.D	48	4.11	2141519.8	254.0	4.93	1194294.9	254.4	5.14	159329.1	4.7
01003-07-04	10ppb PFOS	6	H010126C.M	HILL0142.D	49	4.11	2104440.5	254.0	4.92	1183290.1	254.4	5.14	284697.0	8.9
01003-07-05	25ppb PFOS	7	H010126C.M	HILL0143.D	50	4.11	2090966.6	254.0	4.92	1189051.0	254.4	5.14	760289.3	24.8
01003-07-06	40ppb PFOS	8	H010126C.M	HILL0144.D	51	4.11	2091922.9	254.0	4.93	1187906.5	254.4	5.14	1195676.5	40.2
01003-07-07	50.1ppb PFOS	9	H010126C.M	HILL0145.D	52	4.11	2075323.6	254.0	4.93	1188761.4	254.4	5.14	1513715.1	50.2
01003-07-08	75.1ppb PFOS	10	H010126C.M	HILL0146.D	53	4.11	2114582.0	254.0	4.93	1169896.0	254.4	5.14	2240501.9	76.6
01003-07-09	100.2ppb PFOS	11	H010126C.M	HILL0147.D	54	4.11	2099818.0	254.0	4.93	1166198.0	254.4	5.14	2934306.3	100.9
01003-07-10	250.5ppb PFOS	12	H010126C.M	HILL0148.D	55	4.11	2100878.0	254.0	4.93	1160032.3	254.4	5.14	7028210.5	251.9
01003-07-11	400.8ppb PFOS	13	H010126C.M	HILL0149.D	56	4.11	2115191.3	254.0	4.93	1144943.4	254.4	5.14	10446112.0	392.0
01003-07-12	501.0ppb PFOS	14	H010126C.M	HILL0150.D	57	4.11	2092776.1	254.0	4.92	1149592.4	254.4	5.14	12728628.0	466.4
01003-07-13	1002.0ppb PFOS	15	H010126C.M	HILL0151.D	58	4.11	2112700.5	254.0	4.93	1110896.4	254.4	5.14	22114552.0	993.7
MeOH Blank	TN-A-4740, 04 Dec 00	16	H010126C.M	HILL0152.D	98	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	17	H010126C.M	HILL0153.D	98	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	18	H010126C.M	HILL0154.D	99	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	19	H010126C.M	HILL0155.D	99	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
swPFOS-2.4.1	TCR-002, 012201-swPFOS-2.4.1, 100X Dil. Method Blank, SFM 01-25-01 MEE(MLA)	20	H010126C.M	HILL0156.D	59	4.11	2043336.4	254.0	4.93	1126808.1	254.4	0.00	0.0	0.0
swPFOS-2.4.2	TCR-002, 012201-swPFOS-2.4.2, 100X Dil. Method Blank, SFM 01-25-01 MEE(MLA)	21	H010126C.M	HILL0157.D	60	4.11	2117351.0	254.0	4.92	1132002.3	254.4	0.00	0.0	0.0
swPFOS-2.4.3	TCR-002, 012201-swPFOS-1.4.3, 100X Dil. Method Blank, SFM 01-25-01 MEE(MLA)	22	H010126C.M	HILL0158.D	61	4.11	2050763.3	254.0	4.92	1126241.6	254.4	0.00	0.0	0.0
swPFOS-2.4.4	TCR-002, 012201-swPFOS-2.4.4, 100X Dil. Method Blank, SFM 01-25-01 MEE(MLA)	23	H010126C.M	HILL0159.D	62	4.11	2056015.4	254.0	4.93	1129527.3	254.4	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	24	H010126C.M	HILL0160.D	99	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
swPFOS-2.1.1	TCR-002, 012201-swPFOS-2.1.1, 100X Dil. SFM 01-25-01 MEE(MLA)	25	H010126C.M	HILL0161.D	63	4.11	2073482.0	254.0	4.92	1116196.9	254.4	5.14	3379581.8	122.1
swPFOS-2.1.2	TCR-002, 012201-swPFOS-2.1.2, 100X Dil. SFM 01-25-01 MEE(MLA)	26	H010126C.M	HILL0162.D	64	4.11	2018667.1	254.0	4.92	1111696.8	254.4	5.14	3307267.8	119.9
swPFOS-2.1.3	TCR-002, 012201-swPFOS-2.1.3, 100X Dil. SFM 01-25-01 MEE(MLA)	27	H010126C.M	HILL0163.D	65	4.11	2037984.0	254.0	4.92	1107790.4	254.4	5.14	3271451.3	119.0
swPFOS-2.1.4	TCR-002, 012201-swPFOS-2.1.4, 100X Dil. SFM 01-25-01 MEE(MLA)	28	H010126C.M	HILL0164.D	66	4.11	2037142.3	254.0	4.93	1106296.8	254.4	5.15	3334039.3	121.5
01003-07-09	100.2ppb PFOS, CCV	29	H010126C.M	HILL0165.D	54	4.11	2060367.5	254.0	4.92	1155895.6	254.4	5.14	2889710.8	100.2
MeOH Blank	TN-A-4740, 04 Dec 00	30	H010126C.M	HILL0166.D	99	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	31	H010126C.M	HILL0167.D	100	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
swPFOS-2.2.1	TCR-002, 012201-swPFOS-2.2.1, 100X Dil. SFM 01-25-01 MEE(MLA)	32	H010126C.M	HILL0168.D	67	4.11	1998534.4	254.0	4.92	1107285.0	254.4	5.14	3367552.8	122.6
swPFOS-2.2.2	TCR-002, 012201-swPFOS-2.2.2, 100X Dil. SFM 01-25-01 MEE(MLA)	33	H010126C.M	HILL0169.D	68	4.11	2011509.9	254.0	4.92	1102966.4	254.4	5.14	3381544.5	123.7
swPFOS-2.2.3	TCR-002, 012201-swPFOS-2.2.3, 100X Dil. SFM 01-25-01 MEE(MLA)	34	H010126C.M	HILL0170.D	69	4.11	2076255.3	254.0	4.92	1119362.8	254.4	5.14	3372612.8	121.5
swPFOS-2.2.4	TCR-002, 012201-swPFOS-2.2.4, 100X Dil. SFM 01-25-01 MEE(MLA)	35	H010126C.M	HILL0171.D	70	4.11	2085483.0	254.0	4.92	1109671.5	254.4	5.14	3277744.3	119.0
MeOH Blank	TN-A-4740, 04 Dec 00	36	H010126C.M	HILL0172.D	100	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0

FACT-TCR002 (LIMS #E00-1716), Page 43 of 138

swPFOS-2.3.1	TCR-002, 012201-swPFOS-2.3.1, 100X Dil.	37	H010126C.M	HILL0173.D	71	4.11	2060058.4	254.0	4.93	1103978.5	254.4	5.14	4200268.0	154.6	
	SFM 01-25-01 MEE(MLA)														
swPFOS-2.3.2	TCR-002, 012201-swPFOS-2.3.2, 100X Dil.	38	H010126C.M	HILL0174.D	72	4.11	2075784.8	254.0	4.93	1111200.3	254.4	5.14	4268221.0	156.2	
	SFM 01-25-01 MEE(MLA)														
swPFOS-2.3.3	TCR-002, 012201-swPFOS-2.3.3, 100X Dil.	39	H010126C.M	HILL0175.D	73	4.11	1989603.4	254.0	4.93	1098706.6	254.4	5.14	4350863.0	161.2	
	SFM 01-25-01 MEE(MLA)														
swPFOS-2.3.4	TCR-002, 012201-swPFOS-2.3.4, 100X Dil.	40	H010126C.M	HILL0176.D	74	4.11	2027539.0	254.0	4.92	1114992.9	254.4	5.14	4451651.5	162.6	
	SFM 01-25-01 MEE(MLA)														
01003-07-09	100.2ppb PFOS, CCV	41	H010126C.M	HILL0177.D	54	4.11	2098973.5	254.0	4.92	1158588.0	254.4	5.14	2959959.5	102.5	2.3%
MeOH Blank	TN-A-4740, 04 Dec 00	42	H010126C.M	HILL0178.D	100	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
MeOH Blank	TN-A-4740, 04 Dec 00	43	H010126C.M	HILL0179.D	100	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
MeOH Blank	TN-A-4740, 04 Dec 00	44	H010126C.M	HILL0180.D	90	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
01003-07-01	0ppb PFOS	45	H010126C.M	HILL0181.D	46	4.11	2101871.5	254.0	4.92	1160805.3	254.4	0.00	0.0	0.0	
01003-07-02	2.5ppb PFOS	46	H010126C.M	HILL0182.D	47	4.11	2121610.3	254.0	4.93	1176425.8	254.4	5.14	83007.1	2.2	11.6%
01003-07-03	5ppb PFOS	47	H010126C.M	HILL0183.D	48	4.11	2147140.8	254.0	4.93	1180347.3	254.4	5.14	152217.0	4.5	9.7%
01003-07-04	10ppb PFOS	48	H010126C.M	HILL0184.D	49	4.11	2105029.8	254.0	4.92	1157096.3	254.4	5.14	277895.2	8.9	10.9%
01003-07-05	25ppb PFOS	49	H010126C.M	HILL0185.D	50	4.11	2099930.5	254.0	4.93	1170934.4	254.4	5.14	735110.4	24.3	2.9%
01003-07-06	40ppb PFOS	50	H010126C.M	HILL0186.D	51	4.11	2102480.8	254.0	4.92	1157893.0	254.4	5.14	1160993.9	39.3	1.9%
01003-07-07	50.1ppb PFOS	51	H010126C.M	HILL0187.D	52	4.11	2091009.0	254.0	4.92	1159863.8	254.4	5.14	1481490.6	50.4	0.6%
01003-07-08	75.1ppb PFOS	52	H010126C.M	HILL0188.D	53	4.11	2096784.3	254.0	4.93	1153628.9	254.4	5.14	2222884.0	76.7	2.1%
01003-07-09	100.2ppb PFOS	53	H010126C.M	HILL0189.D	54	4.11	2089134.9	254.0	4.93	1148130.8	254.4	5.14	2895124.5	101.3	1.1%
01003-07-10	250.5ppb PFOS	54	H010126C.M	HILL0190.D	55	4.11	2049022.3	254.0	4.93	1134578.9	254.4	5.14	6880929.5	252.2	0.7%
01003-07-11	400.8ppb PFOS	55	H010126C.M	HILL0191.D	56	4.11	2091091.1	254.0	4.92	1112228.0	254.4	5.14	10583437.0	410.8	2.4%
01003-07-12	501.0ppb PFOS	56	H010126C.M	HILL0192.D	57	4.11	2071210.9	254.0	4.93	1109517.9	254.4	5.14	12823226.0	510.6	1.9%
01003-07-13	1002.0ppb PFOS	57	H010126C.M	HILL0193.D	58	4.11	2082979.1	254.0	4.92	1082716.6	254.4	5.14	21835648.0	1011.5	1.0%
MeOH Blank	TN-A-4740, 04 Dec 00	58	H010126C.M	HILL0194.D	90	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
MeOH Blank	TN-A-4740, 04 Dec 00	59	H010126C.M	HILL0195.D	90	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	

PFOS in Natural Seawater
Shake Flask Method
Day 3

RAW DATA

Agilent 1100 HPLC/MSD "Hillary"

Acquisition Run: H010129

Raw Data

Sample	Sample Description	Peak #	Raw Data	Retention Time	PFOS Area	PFOS Amount	PFOS Conc	PFOS Conc	PFOS Conc	PFOS Conc	PFOS Conc	PFOS Conc	PFOS Conc
MeOH Blank	TN-A-4740, 04 Dec 00	1	H010129B.M HILL0080.D	95	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	2	H010129B.M HILL0081.D	95	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
01003-07-01	0ppb PFOS	3	H010129B.M HILL0082.D	1	4.11	1,842,994.6	254.0	4.91	1,031,596.8	254.4	0.00	0.0	0.0
01003-07-02	2.5ppb PFOS	4	H010129B.M HILL0083.D	2	4.11	1,857,425.3	254.0	4.91	1,036,276.1	254.4	5.14	72,946.1	2.1
01003-07-03	5ppb PFOS	5	H010129B.M HILL0084.D	3	4.11	1,866,181.9	254.0	4.91	1,060,254.5	254.4	5.14	135,320.8	4.4
01003-07-04	10ppb PFOS	6	H010129B.M HILL0085.D	4	4.11	1,845,838.8	254.0	4.91	1,039,973.3	254.4	5.14	243,659.0	8.6
01003-07-05	25ppb PFOS	7	H010129B.M HILL0086.D	5	4.11	1,855,556.4	254.0	4.91	1,037,191.3	254.4	5.14	653,528.8	24.5
01003-07-06	40ppb PFOS	8	H010129B.M HILL0087.D	6	4.11	1,837,022.4	254.0	4.91	1,041,723.2	254.4	5.14	1,070,409.0	40.4
01003-07-07	50.1ppb PFOS	9	H010129B.M HILL0088.D	7	4.11	1,867,504.0	254.0	4.91	1,040,650.0	254.4	5.14	1,313,965.6	49.9
01003-07-08	75.1ppb PFOS	10	H010129B.M HILL0089.D	8	4.11	1,849,679.9	254.0	4.91	1,035,925.8	254.4	5.14	1,998,355.9	76.9
01003-07-09	100.2ppb PFOS	11	H010129B.M HILL0090.D	9	4.11	1,842,005.6	254.0	4.91	1,041,005.9	254.4	5.14	2,624,728.3	101.1
01003-07-10	250.5ppb PFOS	12	H010129B.M HILL0091.D	10	4.11	1,844,809.3	254.0	4.91	1,018,040.1	254.4	5.14	6,294,823.5	254.7
01003-07-11	400.8ppb PFOS	13	H010129B.M HILL0092.D	11	4.11	1,856,680.0	254.0	4.91	1,012,298.0	254.4	5.14	9,555,366.0	399.1
01003-07-12	501.0ppb PFOS	14	H010129B.M HILL0093.D	12	4.11	1,868,779.4	254.0	4.91	1,006,363.1	254.4	5.14	11,624,301.0	496.5
01003-07-13	1002.0ppb PFOS	15	H010129B.M HILL0094.D	13	4.11	1,857,213.8	254.0	4.91	963,085.4	254.4	5.14	20,656,982.0	1,009.0
MeOH Blank	TN-A-4740, 04 Dec 00	16	H010129B.M HILL0095.D	96	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	17	H010129B.M HILL0096.D	96	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	18	H010129B.M HILL0097.D	96	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	19	H010129B.M HILL0098.D	96	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
TCR-002, 012201-swPFOS-3.4.1, 100X Dil.													
swPFOS-3.4.1	Method Blank, SFM 01-26-01 MEE(MLA)	20	H010129B.M HILL0099.D	51	4.11	1,755,606.0	254.0	4.92	1,010,853.3	254.4	0.00	0.0	0.0
TCR-002, 012201-swPFOS-3.4.2, 100X Dil.													
swPFOS-3.4.2	Method Blank, SFM 01-26-01 MEE(MLA)	21	H010129B.M HILL0100.D	52	4.11	1,742,145.9	254.0	4.91	1,006,413.9	254.4	0.00	0.0	0.0
TCR-002, 012201-swPFOS-3.4.3, 100X Dil.													
swPFOS-3.4.3	Method Blank, SFM 01-26-01 MEE(MLA)	22	H010129B.M HILL0101.D	53	4.11	1,812,297.9	254.0	4.91	1,004,414.6	254.4	0.00	0.0	0.0
TCR-002, 012201-swPFOS-3.4.4, 100X Dil.													
swPFOS-3.4.4	Method Blank, SFM 01-26-01 MEE(MLA)	23	H010129B.M HILL0102.D	54	4.11	1,745,632.9	254.0	4.91	1,006,855.0	254.4	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	24	H010129B.M HILL0103.D	97	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
TCR-002, 012201-swPFOS-3.1.1, 100X Dil.													
swPFOS-3.1.1	SFM 01-26-01 MEE(MLA)	25	H010129B.M HILL0104.D	61	4.11	1,793,595.8	254.0	4.91	989,057.5	254.4	5.14	3,606,191.8	147.6
TCR-002, 012201-swPFOS-3.1.2, 100X Dil.													
swPFOS-3.1.2	SFM 01-26-01 MEE(MLA)	26	H010129B.M HILL0105.D	62	4.11	1,750,895.1	254.0	4.91	997,469.1	254.4	5.14	3,599,729.5	146.1
TCR-002, 012201-swPFOS-3.1.3, 100X Dil.													
swPFOS-3.1.3	SFM 01-26-01 MEE(MLA)	27	H010129B.M HILL0106.D	63	4.11	1,840,319.5	254.0	4.91	991,852.5	254.4	5.14	3,632,102.3	148.3
TCR-002, 012201-swPFOS-3.1.4, 100X Dil.													
swPFOS-3.1.4	SFM 01-26-01 MEE(MLA)	28	H010129B.M HILL0107.D	64	4.11	1,828,943.3	254.0	4.91	998,891.9	254.4	5.14	3,656,226.8	148.2
01003-07-09	100.2ppb PFOS, CCV	29	H010129B.M HILL0108.D	9	4.11	1,833,049.3	254.0	4.91	1,041,913.1	254.4	5.14	2,644,160.3	101.8
MeOH Blank	TN-A-4740, 04 Dec 00	30	H010129B.M HILL0109.D	97	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	31	H010129B.M HILL0110.D	97	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0
TCR-002, 012201-swPFOS-3.2.1, 100X Dil.													
swPFOS-3.2.1	SFM 01-26-01 MEE(MLA)	32	H010129B.M HILL0111.D	65	4.11	1,827,610.6	254.0	4.91	996,184.2	254.4	5.14	3,371,343.3	136.7
TCR-002, 012201-swPFOS-3.2.2, 100X Dil.													
swPFOS-3.2.2	SFM 01-26-01 MEE(MLA)	33	H010129B.M HILL0112.D	66	4.11	1,813,043.8	254.0	4.91	994,797.9	254.4	5.14	3,298,727.8	133.9

FACT-TCR002 (LIMS #E00-1716), Page 45 of 138

TCR-002, 012201-swPFOS-3.2.3, 100X Dil.															
swPFOS-3.2.3	SFM 01-26-01 MEE(MLA)	34	H010129B.M	HILL0113.D	67	4.11	1,821,905.5	254.0	4.91	1,001,176.3	254.4	5.14	3,367,698.0	135.9	
TCR-002, 012201-swPFOS-3.2.4, 100X Dil.															
swPFOS-3.2.4	SFM 01-26-01 MEE(MLA)	35	H010129B.M	HILL0114.D	68	4.11	1,804,143.4	254.0	4.91	983,479.1	254.4	5.14	3,367,747.0	138.4	
MeOH Blank	TN-A-4740, 04 Dec 00	36	H010129B.M	HILL0115.D	97	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
TCR-002, 012201-swPFOS-3.3.1, 100X Dil.															
swPFOS-3.3.1	SFM 01-26-01 MEE(MLA)	37	H010129B.M	HILL0116.D	69	4.11	1,763,726.3	254.0	4.91	987,376.1	254.4	5.14	2,909,405.8	118.6	
TCR-002, 012201-swPFOS-3.3.2, 100X Dil.															
swPFOS-3.3.2	SFM 01-26-01 MEE(MLA)	38	H010129B.M	HILL0117.D	70	4.11	1,824,344.0	254.0	4.91	990,274.6	254.4	5.14	3,057,538.8	124.4	
TCR-002, 012201-swPFOS-3.3.3, 100X Dil.															
swPFOS-3.3.3	SFM 01-26-01 MEE(MLA)	39	H010129B.M	HILL0118.D	71	4.11	1,808,923.3	254.0	4.91	995,121.1	254.4	5.14	2,972,588.3	120.3	
TCR-002, 012201-swPFOS-3.3.4, 100X Dil.															
swPFOS-3.3.4	SFM 01-26-01 MEE(MLA)	40	H010129B.M	HILL0119.D	72	4.11	1,817,528.4	254.0	4.91	992,111.4	254.4	5.14	3,043,574.5	123.6	
01003-07-09	100.2ppb PFOS, CCV	41	H010129B.M	HILL0120.D	9	4.11	1,823,161.8	254.0	4.91	1,039,414.1	254.4	5.14	2,664,370.3	102.8	2.6%
MeOH Blank	TN-A-4740, 04 Dec 00	42	H010129B.M	HILL0121.D	97	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
MeOH Blank	TN-A-4740, 04 Dec 00	43	H010129B.M	HILL0122.D	97	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
MeOH Blank	TN-A-4740, 04 Dec 00	44	H010129B.M	HILL0123.D	97	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
01003-07-01	0ppb PFOS	45	H010129B.M	HILL0124.D	1	4.11	1,797,068.0	254.0	4.91	1,041,622.9	254.4	0.00	0.0	0.0	
01003-07-02	2.5ppb PFOS	46	H010129B.M	HILL0125.D	2	4.11	1,807,103.9	254.0	4.91	1,046,423.3	254.4	5.14	73,997.4	2.1	15.1%
01003-07-03	5ppb PFOS	47	H010129B.M	HILL0126.D	3	4.11	1,806,916.9	254.0	4.91	1,054,742.1	254.4	5.14	134,066.8	4.4	12.7%
01003-07-04	10ppb PFOS	48	H010129B.M	HILL0127.D	4	4.11	1,808,355.9	254.0	4.91	1,046,836.9	254.4	5.14	248,733.9	8.8	12.4%
01003-07-05	25ppb PFOS	49	H010129B.M	HILL0128.D	5	4.11	1,792,502.9	254.0	4.91	1,048,370.6	254.4	5.14	653,704.9	24.2	3.4%
01003-07-06	40ppb PFOS	50	H010129B.M	HILL0129.D	6	4.11	1,790,624.0	254.0	4.91	1,042,150.7	254.4	5.14	1,053,882.6	39.6	0.8%
01003-07-07	50.1ppb PFOS	51	H010129B.M	HILL0130.D	7	4.11	1,809,404.6	254.0	4.91	1,041,860.6	254.4	5.14	1,323,609.8	50.2	0.2%
01003-07-08	75.1ppb PFOS	52	H010129B.M	HILL0131.D	8	4.11	1,801,089.3	254.0	4.91	1,028,674.6	254.4	5.14	1,972,099.5	76.4	1.7%
01003-07-09	100.2ppb PFOS	53	H010129B.M	HILL0132.D	9	4.11	1,773,768.8	254.0	4.91	1,036,261.8	254.4	5.14	2,618,876.0	101.4	1.2%
01003-07-10	250.5ppb PFOS	54	H010129B.M	HILL0133.D	10	4.11	1,809,450.4	254.0	4.91	1,025,689.3	254.4	5.14	6,271,318.5	252.2	0.7%
01003-07-11	400.8ppb PFOS	55	H010129B.M	HILL0134.D	11	4.11	1,756,825.4	254.0	4.91	1,009,207.4	254.4	5.14	9,627,486.0	403.7	0.7%
01003-07-12	501.0ppb PFOS	56	H010129B.M	HILL0135.D	12	4.11	1,816,221.0	254.0	4.91	1,003,265.8	254.4	5.14	11,622,508.0	498.1	0.6%
01003-07-13	1002.0ppb PFOS	57	H010129B.M	HILL0136.D	13	4.11	1,840,075.4	254.0	4.91	977,330.4	254.4	5.14	20,749,418.0	996.5	0.6%
MeOH Blank	TN-A-4740, 04 Dec 00	58	H010129B.M	HILL0137.D	98	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	
MeOH Blank	TN-A-4740, 04 Dec 00	59	H010129B.M	HILL0138.D	98	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	

PFOS in Seawater

Day 1

Hillary, H010125

Sample ID	Sample Description	Lot	Rep (ML)	Date (LC)	Vial	PFOS RT	PFOS Area	ng/mL PFOS (ppb)	dilution factor correction (pp/mL)	purity factor correction (µg/mL)	Average Std Dev %CV
swPFOS-1.1.1	TCR-002, 012201-swPFOS-1.1.1, 100X Dil. SFM 01-24-01 MEE(MLA)	45	H0125A1.M	HILL0045.D	31	5.14	3,780,994	125.7	12.6	12.3	12.6
swPFOS-1.1.2	TCR-002, 012201-swPFOS-1.1.2, 100X Dil. SFM 01-24-01 MEE(MLA)	46	H0125A1.M	HILL0046.D	32	5.14	3,919,051	129.9	13.0	12.7	0.25
swPFOS-1.1.3	TCR-002, 012201-swPFOS-1.1.3, 100X Dil. SFM 01-24-01 MEE(MLA)	47	H0125A1.M	HILL0047.D	33	5.14	3,895,319	128.8	12.9	12.6	2.0%
swPFOS-1.1.4	TCR-002, 012201-swPFOS-1.1.4, 100X Dil. SFM 01-24-01 MEE(MLA)	48	H0125A1.M	HILL0048.D	34	5.14	4,009,265	131.9	13.2	12.9	
swPFOS-1.2.1	TCR-002, 012201-swPFOS-1.2.1, 100X Dil. SFM 01-24-01 MEE(MLA)	52	H0125A1.M	HILL0052.D	35	5.14	3,576,570	118.0	11.8	11.6	11.7
swPFOS-1.2.2	TCR-002, 012201-swPFOS-1.2.2, 100X Dil. SFM 01-24-01 MEE(MLA)	53	H0125A1.M	HILL0053.D	36	5.14	3,702,940	122.9	12.3	12.0	0.19
swPFOS-1.2.3	TCR-002, 012201-swPFOS-1.2.3, 100X Dil. SFM 01-24-01 MEE(MLA)	54	H0125A1.M	HILL0054.D	37	5.14	3,689,193	119.4	11.9	11.7	1.6%
swPFOS-1.2.4	TCR-002, 012201-swPFOS-1.2.4, 100X Dil. SFM 01-24-01 MEE(MLA)	55	H0125A1.M	HILL0055.D	38	5.14	3,597,279	118.4	11.8	11.6	
swPFOS-1.3.1	TCR-002, 012201-swPFOS-1.3.1, 100X Dil. SFM 01-24-01 MEE(MLA)	57	H0125A1.M	HILL0057.D	39	5.14	3,797,897	124.5	12.5	12.2	12.4
swPFOS-1.3.2	TCR-002, 012201-swPFOS-1.3.2, 100X Dil. SFM 01-24-01 MEE(MLA)	58	H0125A1.M	HILL0058.D	40	5.14	3,862,722	128.2	12.8	12.5	0.13
swPFOS-1.3.3	TCR-002, 012201-swPFOS-1.3.3, 100X Dil. SFM 01-24-01 MEE(MLA)	59	H0125A1.M	HILL0059.D	41	5.14	3,785,649	127.2	12.7	12.4	1.0%
swPFOS-1.3.4	TCR-002, 012201-swPFOS-1.3.4, 100X Dil. SFM 01-24-01 MEE(MLA)	60	H0125A1.M	HILL0060.D	42	5.14	3,743,934	126.1	12.6	12.3	

Yellowed/grayed areas indicate points that are not used.

Day 1
 average: 12.2 µg/mL
 std dev: 0.47
 %C.V.: 3.9%

PFOS in Seawater

Day 2

Hillary, H010126

Sample ID	Sample Description	Run#	Rep#	Date	Vol	PFOS (ng)	PFOS (pg)	Normal PFOS (pg)	Dilution factor	Purity factor	Average
swPFOS-2.1.1	TCR-002, 012201-swPFOS-2.1.1, 100X Dil. SFM 01-25-01 MEE(MLA)	25	H010126C.M	HILL0161.D	63	5.14	3379582	122.1	12.2	11.9	11.8
swPFOS-2.1.2	TCR-002, 012201-swPFOS-2.1.2, 100X Dil. SFM 01-25-01 MEE(MLA)	26	H010126C.M	HILL0162.D	64	5.14	3307268	119.9	12.0	11.7	0.10
swPFOS-2.1.3	TCR-002, 012201-swPFOS-2.1.3, 100X Dil. SFM 01-25-01 MEE(MLA)	27	H010126C.M	HILL0163.D	65	5.14	3271451	119.0	11.9	11.7	0.8%
swPFOS-2.1.4	TCR-002, 012201-swPFOS-2.1.4, 100X Dil. SFM 01-25-01 MEE(MLA)	28	H010126C.M	HILL0164.D	66	5.15	3334039	121.5	12.1	11.8	
swPFOS-2.2.1	TCR-002, 012201-swPFOS-2.2.1, 100X Dil. SFM 01-25-01 MEE(MLA)	32	H010126C.M	HILL0168.D	67	5.14	3367553	122.6	12.3	12.0	11.9
swPFOS-2.2.2	TCR-002, 012201-swPFOS-2.2.2, 100X Dil. SFM 01-25-01 MEE(MLA)	33	H010126C.M	HILL0169.D	68	5.14	3381545	123.7	12.4	12.1	0.18
swPFOS-2.2.3	TCR-002, 012201-swPFOS-2.2.3, 100X Dil. SFM 01-25-01 MEE(MLA)	34	H010126C.M	HILL0170.D	69	5.14	3372613	121.5	12.1	11.8	1.5%
swPFOS-2.2.4	TCR-002, 012201-swPFOS-2.2.4, 100X Dil. SFM 01-25-01 MEE(MLA)	35	H010126C.M	HILL0171.D	70	5.14	3277744	119.0	11.9	11.7	
swPFOS-2.3.1	TCR-002, 012201-swPFOS-2.3.1, 100X Dil. SFM 01-25-01 MEE(MLA)	37	H010126C.M	HILL0173.D	71	5.14	4200268	154.6	15.5	15.2	15.6
swPFOS-2.3.2	TCR-002, 012201-swPFOS-2.3.2, 100X Dil. SFM 01-25-01 MEE(MLA)	38	H010126C.M	HILL0174.D	72	5.14	4268221	156.2	15.6	15.3	0.39
swPFOS-2.3.3	TCR-002, 012201-swPFOS-2.3.3, 100X Dil. SFM 01-25-01 MEE(MLA)	39	H010126C.M	HILL0175.D	73	5.14	4350863	161.2	16.1	15.8	2.5%
swPFOS-2.3.4	TCR-002, 012201-swPFOS-2.3.4, 100X Dil. SFM 01-25-01 MEE(MLA)	40	H010126C.M	HILL0176.D	74	5.14	4451652	162.6	16.3	16.0	

* Yellowed/grayed areas indicate points that are not used.

Day 2
average: 11.9 µg/mL
std dev: 0.07
%CV.: 0.6%

PFOS in Seawater

Day 3

Hillary, H010129

Sample ID	Sample Description	Run	Rep. No.	Lab File	Vials	PFOS RT	PFOS Area	ng/mL PFOS (DD)	dilution factor correction	purity factor correction	Average Std Dev %CV
Sample Data, DAY 3	swPFOS-3.1.1 TCR-002, 012201-swPFOS-3.1.1, 100X Dil. SFM 01-26-01 MEE(MLA)	25	H010129B.M	HILL0104.D	61	5.14	3,606,192	147.6	14.8	14.5	14.5
	swPFOS-3.1.2 TCR-002, 012201-swPFOS-3.1.2, 100X Dil. SFM 01-26-01 MEE(MLA)	26	H010129B.M	HILL0105.D	62	5.14	3,599,730	146.1	14.6	14.3	0.10
	swPFOS-3.1.3 TCR-002, 012201-swPFOS-3.1.3, 100X Dil. SFM 01-26-01 MEE(MLA)	27	H010129B.M	HILL0106.D	63	5.14	3,632,102	148.3	14.8	14.5	0.7%
	swPFOS-3.1.4 TCR-002, 012201-swPFOS-3.1.4, 100X Dil. SFM 01-26-01 MEE(MLA)	28	H010129B.M	HILL0107.D	64	5.14	3,656,227	148.2	14.8	14.5	
	swPFOS-3.2.1 TCR-002, 012201-swPFOS-3.2.1, 100X Dil. SFM 01-26-01 MEE(MLA)	32	H010129B.M	HILL0111.D	65	5.14	3,371,343	136.7	13.7	13.4	13.3
	swPFOS-3.2.2 TCR-002, 012201-swPFOS-3.2.2, 100X Dil. SFM 01-26-01 MEE(MLA)	33	H010129B.M	HILL0112.D	66	5.14	3,298,728	133.9	13.4	13.1	0.17
	swPFOS-3.2.3 TCR-002, 012201-swPFOS-3.2.3, 100X Dil. SFM 01-26-01 MEE(MLA)	34	H010129B.M	HILL0113.D	67	5.14	3,367,698	135.9	13.6	13.3	1.3%
	swPFOS-3.2.4 TCR-002, 012201-swPFOS-3.2.4, 100X Dil. SFM 01-26-01 MEE(MLA)	35	H010129B.M	HILL0114.D	68	5.14	3,367,747	138.4	13.8	13.5	
	swPFOS-3.3.1 TCR-002, 012201-swPFOS-3.3.1, 100X Dil. SFM 01-26-01 MEE(MLA)	37	H010129B.M	HILL0116.D	69	5.14	2,909,406	118.6	11.9	11.7	11.9
	swPFOS-3.3.2 TCR-002, 012201-swPFOS-3.3.2, 100X Dil. SFM 01-26-01 MEE(MLA)	38	H010129B.M	HILL0117.D	70	5.14	3,057,539	124.4	12.4	12.1	0.23
	swPFOS-3.3.3 TCR-002, 012201-swPFOS-3.3.3, 100X Dil. SFM 01-26-01 MEE(MLA)	39	H010129B.M	HILL0118.D	71	5.14	2,972,588	120.3	12.0	11.7	1.9%
	swPFOS-3.3.4 TCR-002, 012201-swPFOS-3.3.4, 100X Dil. SFM 01-26-01 MEE(MLA)	40	H010129B.M	HILL0119.D	72	5.14	3,043,575	123.6	12.4	12.1	

* Yellowed/greyed areas indicate points that are not used.

Day 3
 average: 13.2 µg/mL
 std dev: 1.30
 %C.V.: 9.8%

FACT-TCR002 PFOS Solubility of TCR-00017-46 in Seawater

Day 1 data source acquisition instrument/sequence: Agilent 1100 HPLC/MSD "Hillary"/H010125.s

Day 2 data source acquisition instrument/sequence: Agilent 1100 HPLC/MSD "Hillary"/H010126.s

Day 3 data source acquisition instrument/sequence: Agilent 1100 HPLC/MSD "Hillary"/H010129.s

DATA SUMMARY FOR INDIVIDUAL SAMPLES

swPFOS-1.1.1	12.6	12.3	12.6
swPFOS-1.1.2	13.0	12.7	0.25
swPFOS-1.1.3	12.9	12.6	2.0%
swPFOS-1.1.4	13.2	12.9	
swPFOS-1.2.1	11.8	11.6	11.7
swPFOS-1.2.2	12.3	12.0	0.19
swPFOS-1.2.3	11.9	11.7	1.6%
swPFOS-1.2.4	11.8	11.6	
swPFOS-1.3.1	12.5	12.2	12.4
swPFOS-1.3.2	12.8	12.5	0.13
swPFOS-1.3.3	12.7	12.4	1.0%
swPFOS-1.3.4	12.6	12.3	
swPFOS-2.1.1	12.2	11.9	11.8
swPFOS-2.1.2	12.0	11.7	0.10
swPFOS-2.1.3	11.9	11.7	0.8%
swPFOS-2.1.4	12.1	11.8	
swPFOS-2.2.1	12.3	12.0	11.9
swPFOS-2.2.2	12.4	12.1	0.18
swPFOS-2.2.3	12.1	11.8	1.5%
swPFOS-2.2.4	11.9	11.7	
swPFOS-2.3.1	15.5	15.2	15.6
swPFOS-2.3.2	15.6	15.3	0.39
swPFOS-2.3.3	16.1	15.8	2.5%
swPFOS-2.3.4	16.3	16.0	
swPFOS-3.1.1	14.8	14.5	14.5
swPFOS-3.1.2	14.6	14.3	0.10
swPFOS-3.1.3	14.8	14.5	0.7%
swPFOS-3.1.4	14.8	14.5	
swPFOS-3.2.1	13.7	13.4	13.3
swPFOS-3.2.2	13.4	13.1	0.17
swPFOS-3.2.3	13.6	13.3	1.3%
swPFOS-3.2.4	13.8	13.5	
swPFOS-3.3.1	11.9	11.7	11.9
swPFOS-3.3.2	12.4	12.1	0.23
swPFOS-3.3.3	12.0	11.7	1.9%
swPFOS-3.3.4	12.4	12.1	

FACT-TCR002 PFOS Solubility of TCR-00017-46 in Seawater.**DAILY AND TOTAL DATA SUMMARIES****Day 1 data summary**

Average: 12.2 ppm

Std Dev: 0.47

%CV: 3.9%

Day 2 data summary

Average: 11.9 ppm

Std Dev: 0.07

%CV: 0.6%

(Sample 3 excluded via Q-Test)

Day 3 data summary

Average: 13.2 ppm

Std Dev: 1.30

%CV: 9.8%

TOTAL data summary

Average: 12.4 ppm

Std Dev: 0.71

%CV: 5.7%

* Greyed / yellowed areas have been omitted from the calculations.

PFOS in Seawater Shake Flask Method

Q-TEST Data Calculations:

Sample ID:	swPFOS-2.1 swPFOS-2.2 swPFOS-2.3
Sample Conc., Rep1	11.8
Sample Conc., Rep2	11.9
Sample Conc., Rep3	15.5
Range:	3.70
Gap:	3.60
Qobs:	0.97
No. observations:	3
Retain?	no

Exclude sample set swPFOS-2.3

Qobserved = gap/range where "gap" is the difference between the questionable data point and the closest value of the data set, and "range" is the difference between the highest and lowest value of the data set. If Q-observed is > Q-tabulated, then the data point may be excluded with 90% confidence. Q-tabulated for 3 observations (or data points) is 0.88. The samples highlighted in yellow in the above table have Q-observed > 0.88, and were therefore excluded from the reported data.

PFOS in 3.5% NaCl Solution
Shake Flask Method
Day 1

RAW DATA

Agilent 1100 HPLC/MSD "Hillary"

Acquisition Run: H010131

Raw Data

Sample ID	Sample Description	Time	Area	Height	Area%	Height%	Area	Height	Area%	Height%	Area	Height	Area%	Height%
MeOH Blank	TN-A-4740, 04 Dec 00	1	H010131A.M HILL0001.D	91	4.124	21409.6	0.0	0.000	0.0	0.0	5.153	10335.3	0.9	
MeOH Blank	TN-A-4740, 04 Dec 00	2	H010131A.M HILL0002.D	91	0.000	0.0	0.0	0.000	0.0	0.0	5.156	7224.9	0.9	
01003-07-13	1002.0ppb PFOS	3	H010131A.M HILL0003.D	13	4.121	2143601.3	301.1	4.821	993486.1	250.7	5.206	22127404.0	0.0	
01003-07-13	1002.0ppb PFOS	4	H010131A.M HILL0004.D	13	4.121	2124774.3	305.6	4.821	970509.3	250.7	5.206	21844558.0	0.0	
MeOH Blank	TN-A-4740, 04 Dec 00	5	H010131A.M HILL0005.D	91	4.119	11608.7	0.0	0.000	0.0	0.0	5.156	8180.2	0.9	
MeOH Blank	TN-A-4740, 04 Dec 00	6	H010131A.M HILL0006.D	91	4.117	12030.3	0.0	0.000	0.0	0.0	5.160	3006.9	0.9	
MeOH Blank	TN-A-4740, 04 Dec 00	7	H010131A.M HILL0007.D	91	4.118	11730.2	0.0	0.000	0.0	0.0	5.161	7961.4	0.9	
01003-07-01	0ppb PFOS	8	H010131A.M HILL0008.D	1	4.121	2048396.3	269.5	4.821	1060946.9	250.7	5.157	12540.2	1.3	
01003-07-02	2.5ppb PFOS	9	H010131A.M HILL0009.D	2	4.122	2093037.3	270.5	4.823	1080061.5	250.7	5.152	87037.7	4.0	
01003-07-03	5ppb PFOS	10	H010131A.M HILL0010.D	3	4.118	2110525.8	269.8	4.822	1091921.6	250.7	5.162	155338.9	6.4	
01003-07-04	10ppb PFOS	11	H010131A.M HILL0011.D	4	4.119	2097033.9	268.2	4.819	1091357.9	250.7	5.155	283237.4	10.9	
01003-07-05	25ppb PFOS	12	H010131A.M HILL0012.D	5	4.118	2087465.6	266.3	4.822	1094229.1	250.7	5.161	731161.4	26.8	
01003-07-06	40ppb PFOS	13	H010131A.M HILL0013.D	6	4.121	2089456.8	268.0	4.821	1088254.4	250.7	5.157	1153655.3	42.2	
01003-07-07	50.1ppb PFOS	14	H010131A.M HILL0014.D	7	4.118	2089359.4	268.0	4.819	1087986.1	250.7	5.155	1443086.4	52.8	
01003-07-08	75.1ppb PFOS	15	H010131A.M HILL0015.D	8	4.115	2087226.1	264.3	4.823	1102227.4	250.7	5.159	2167099.3	78.4	
01003-07-09	100.2ppb PFOS	16	H010131A.M HILL0016.D	9	4.122	2097466.5	264.9	4.822	1105173.9	250.7	5.158	2856232.3	103.6	
01003-07-10	250.5ppb PFOS	17	H010131A.M HILL0017.D	10	4.119	2091827.3	264.0	4.820	1105860.1	250.7	5.156	6777085.5	256.7	
01003-07-11	400.8ppb PFOS	18	H010131A.M HILL0018.D	11	4.118	2081840.1	262.9	4.819	1105371.5	250.7	5.232	10322257.0	411.4	
01003-07-12	501.0ppb PFOS	19	H010131A.M HILL0019.D	12	4.119	2127462.5	270.6	4.820	1097313.3	250.7	5.240	12413629.0	517.0	
01003-07-13	1002.0ppb PFOS	20	H010131A.M HILL0020.D	13	4.121	2128281.3	277.4	4.821	1070927.4	250.7	5.200	22093346.0	1319.8	
MeOH Blank	TN-A-4740, 04 Dec 00	21	H010131A.M HILL0021.D	92	4.119	11259.9	0.0	0.000	0.0	0.0	5.163	4968.3	0.9	
MeOH Blank	TN-A-4740, 04 Dec 00	22	H010131A.M HILL0022.D	92	4.121	11279.0	0.0	0.000	0.0	0.0	5.165	6091.6	0.9	
MeOH Blank	TN-A-4740, 04 Dec 00	23	H010131A.M HILL0023.D	92	4.122	11475.7	0.0	0.000	0.0	0.0	5.158	7088.3	0.9	
01003-07-01	0ppb PFOS	24	H010131A.M HILL0024.D	1	4.119	2095800.8	254.9	4.819	1147396.9	250.7	5.155	8761.4	1.2	
01003-07-02	2.5ppb PFOS	25	H010131A.M HILL0025.D	2	4.119	2092956.8	253.9	4.819	1150438.9	250.7	5.155	85318.9	3.7	
01003-07-03	5ppb PFOS	26	H010131A.M HILL0026.D	3	4.121	2136092.8	255.4	4.822	1167485.3	250.7	5.158	158275.4	6.1	22.1
01003-07-04	10ppb PFOS	27	H010131A.M HILL0027.D	4	4.119	2134188.0	254.6	4.819	1169935.9	250.7	5.156	294889.3	10.6	6.1
01003-07-05	25ppb PFOS	28	H010131A.M HILL0028.D	5	4.121	2101620.5	251.3	4.821	1167190.9	250.7	5.158	731035.8	25.2	0.3
01003-07-06	40ppb PFOS	29	H010131A.M HILL0029.D	6	4.122	2118756.3	254.2	4.822	1163371.8	250.7	5.158	1194338.3	40.9	2.0
01003-07-07	50.1ppb PFOS	30	H010131A.M HILL0030.D	7	4.117	2094413.3	250.6	4.821	1166273.0	250.7	5.161	1473480.0	50.3	0.4
01003-07-08	75.1ppb PFOS	31	H010131A.M HILL0031.D	8	4.118	2107698.3	253.3	4.819	1161204.9	250.7	5.155	2231440.0	76.6	1.9
01003-07-09	100.2ppb PFOS	32	H010131A.M HILL0032.D	9	4.118	2107941.5	253.2	4.822	1161693.0	250.7	5.162	2926687.3	101.0	0.7
01003-07-10	250.5ppb PFOS	33	H010131A.M HILL0033.D	10	4.118	2085320.1	251.0	4.822	1159443.8	250.7	5.162	6886002.0	248.1	-1.0
01003-07-11	400.8ppb PFOS	34	H010131A.M HILL0034.D	11	4.118	2104751.3	260.1	4.822	1129470.5	250.7	5.218	10541540.0	411.2	2.6
01003-07-12	501.0ppb PFOS	35	H010131A.M HILL0035.D	12	4.121	2089080.4	254.8	4.821	1144288.3	250.7	5.157	12617936.0	501.1	0.0
01003-07-13	1002.0ppb PFOS	36	H010131A.M HILL0036.D	13	4.121	2132100.5	270.5	4.821	1099943.1	250.7	5.164	22841106.0	1346.3	
MeOH Blank	TN-A-4740, 04 Dec 00	37	H010131A.M HILL0037.D	92	4.118	11790.3	0.0	0.000	0.0	0.0	5.161	4108.8	0.9	
MeOH Blank	TN-A-4740, 04 Dec 00	38	H010131A.M HILL0038.D	92	4.119	8487.4	0.0	0.000	0.0	0.0	5.155	7473.4	0.9	
MeOH Blank	TN-A-4740, 04 Dec 00	39	H010131A.M HILL0039.D	93	0.000	0.0	0.0	0.000	0.0	0.0	5.162	6551.3	0.9	
TCR-002, 012901-nadPFOS-1.4.1, 100X Dil. Method Blank, SFM 01-31-														
nadPFOS-1.4.1 01 MEE (MLA)		40	H010131A.M HILL0040.D	21	4.117	2022771.3	250.5	4.821	1126861.6	250.7	5.161	4319.0	1.0	
TCR-002, 012901-nadPFOS-1.4.2, 100X Dil. Method Blank, SFM 01-31-														
nadPFOS-1.4.2 01 MEE (MLA)		41	H010131A.M HILL0041.D	22	4.119	2012977.0	252.9	4.819	1111055.0	250.7	5.156	2599.3	1.0	

Exact Copy of Original

Initial
 Solubility, Shake Flask Method
 5/2/2001

MLA
 05 May 01
 page 1 of 11

TCR-002, 012901-nadPFOS-1.4.3, 100X Dil. Method Blank, SFM 01-31-													
nadPFOS-1.4.3	01 MEE(MLA)	42	H010131A.M HILL0042.D	23	4.119	2083439.9	259.2	4.819	1121788.5	250.7	5.155	8289.1	1.2
TCR-002, 012901-nadPFOS-1.4.4, 100X Dil. Method Blank, SFM 01-31-													
nadPFOS-1.4.4	01 MEE(MLA)	43	H010131A.M HILL0043.D	24	4.119	2038687.6	257.2	4.820	1106088.3	250.7	5.163	4842.2	1.0
MeOH Blank	TN-A-4740, 04 Dec 00	44	H010131A.M HILL0044.D	93	4.121	12652.4	0.0	0.000	0.0	0.0	5.158	5851.6	0.9
TCR-002, 012901-nadPFOS-1.1.1, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.1.1	100X Dil. SFM 01-31-01 MEE(MLA)	45	H010131A.M HILL0045.D	31	4.119	2044713.0	263.8	4.820	1081767.8	250.7	5.156	5915846.0	227.0
TCR-002, 012901-nadPFOS-1.1.2, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.1.2	100X Dil. SFM 01-31-01 MEE(MLA)	46	H010131A.M HILL0046.D	32	4.118	2043153.5	267.1	4.822	1067474.3	250.7	5.162	5851699.5	227.5
TCR-002, 012901-nadPFOS-1.1.3, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.1.3	100X Dil. SFM 01-31-01 MEE(MLA)	47	H010131A.M HILL0047.D	33	4.119	2047704.3	263.1	4.820	1086182.9	250.7	5.156	5827699.0	222.4
TCR-002, 012901-nadPFOS-1.1.4, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.1.4	100X Dil. SFM 01-31-01 MEE(MLA)	48	H010131A.M HILL0048.D	34	4.118	2040932.9	266.2	4.822	1070219.3	250.7	5.162	5990611.0	232.7
01003-07-09	100.2ppb PFOS, CCV	49	H010131A.M HILL0049.D	9	4.121	2112425.0	251.0	4.822	1174724.8	250.7	5.158	2899438.8	98.9
MeOH Blank	TN-A-4740, 04 Dec 00	50	H010131A.M HILL0050.D	93	4.118	11531.6	0.0	0.000	0.0	0.0	5.162	1726.1	0.9
MeOH Blank	TN-A-4740, 04 Dec 00	51	H010131A.M HILL0051.D	93	4.118	8666.6	0.0	0.000	0.0	0.0	5.161	6133.9	0.9
TCR-002, 012901-nadPFOS-1.2.1, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.2.1	100X Dil. SFM 01-31-01 MEE(MLA)	52	H010131A.M HILL0052.D	35	4.118	1995595.9	261.8	4.819	1063811.5	250.7	5.155	5222047.5	202.2
TCR-002, 012901-nadPFOS-1.2.2, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.2.2	100X Dil. SFM 01-31-01 MEE(MLA)	53	H010131A.M HILL0053.D	36	4.119	2011646.8	265.8	4.819	1056323.1	250.7	5.155	5201276.0	202.9
TCR-002, 012901-nadPFOS-1.2.3, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.2.3	100X Dil. SFM 01-31-01 MEE(MLA)	54	H010131A.M HILL0054.D	37	4.118	1994192.1	261.7	4.822	1063676.3	250.7	5.162	5192852.5	201.0
TCR-002, 012901-nadPFOS-1.2.4, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.2.4	100X Dil. SFM 01-31-01 MEE(MLA)	55	H010131A.M HILL0055.D	38	4.118	1986943.1	261.5	4.822	1060499.5	250.7	5.161	5269990.5	204.9
MeOH Blank	TN-A-4740, 04 Dec 00	56	H010131A.M HILL0056.D	94	4.120	9279.7	0.0	0.000	0.0	0.0	5.156	6002.4	0.9
TCR-002, 012901-nadPFOS-1.3.1, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.3.1	100X Dil. SFM 01-31-01 MEE(MLA)	57	H010131A.M HILL0057.D	39	4.118	1922033.9	250.0	4.819	1073103.1	250.7	5.155	4172225.8	158.1
TCR-002, 012901-nadPFOS-1.3.2, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.3.2	100X Dil. SFM 01-31-01 MEE(MLA)	58	H010131A.M HILL0058.D	40	4.118	1988949.3	258.8	4.822	1072507.3	250.7	5.161	4317565.0	164.0
TCR-002, 012901-nadPFOS-1.3.3, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.3.3	100X Dil. SFM 01-31-01 MEE(MLA)	59	H010131A.M HILL0059.D	41	4.121	1991271.4	261.3	4.821	1063607.5	250.7	5.157	4187921.5	160.2
TCR-002, 012901-nadPFOS-1.3.4, 100X Dil. SFM 01-31-01 MEE(MLA)													
nadPFOS-1.3.4	100X Dil. SFM 01-31-01 MEE(MLA)	60	H010131A.M HILL0060.D	42	4.120	1982020.0	262.7	4.821	1052995.8	250.7	5.157	4194090.0	162.2
01003-07-09	100.2ppb PFOS, CCV	61	H010131A.M HILL0061.D	9	4.121	2055678.9	249.3	4.821	1150774.1	250.7	5.157	2847996.0	99.1
MeOH Blank	TN-A-4740, 04 Dec 00	62	H010131A.M HILL0062.D	94	4.121	9943.1	0.0	0.000	0.0	0.0	0.000	0.0	0.0
MeOH Blank	TN-A-4740, 04 Dec 00	63	H010131A.M HILL0063.D	94	4.119	11681.8	0.0	0.000	0.0	0.0	5.156	6434.6	0.9
MeOH Blank	TN-A-4740, 04 Dec 00	64	H010131A.M HILL0064.D	94	4.122	11540.1	0.0	0.000	0.0	0.0	0.000	0.0	0.0
01003-07-01	0ppb PFOS	65	H010131A.M HILL0065.D	1	4.120	2041874.0	245.9	4.820	1159008.5	250.7	5.156	8746.7	1.2
01003-07-02	2.5ppb PFOS	66	H010131A.M HILL0066.D	2	4.119	2069020.8	245.7	4.819	1175189.9	250.7	5.155	80179.2	3.5
01003-07-03	5ppb PFOS	67	H010131A.M HILL0067.D	3	4.118	2081127.1	244.2	4.822	1189579.9	250.7	5.162	150817.4	5.8
01003-07-04	10ppb PFOS	68	H010131A.M HILL0068.D	4	4.119	2070209.8	244.3	4.819	1182606.9	250.7	5.156	286563.4	10.2
01003-07-05	25ppb PFOS	69	H010131A.M HILL0069.D	5	4.120	2059409.9	243.5	4.821	1180256.6	250.7	5.157	720880.0	24.6
01003-07-06	40ppb PFOS	70	H010131A.M HILL0070.D	6	4.119	2080276.8	245.4	4.820	1183280.5	250.7	5.156	1170613.4	39.4
01003-07-07	50.1ppb PFOS	71	H010131A.M HILL0071.D	7	4.118	2077642.6	244.2	4.819	1187313.3	250.7	5.155	1480991.4	49.6
01003-07-08	75.1ppb PFOS	72	H010131A.M HILL0072.D	8	4.119	2084619.8	246.0	4.819	1182561.1	250.7	5.155	2180932.8	73.5
01003-07-09	100.2ppb PFOS	73	H010131A.M HILL0073.D	9	4.119	2089444.1	245.3	4.819	1188650.3	250.7	5.155	2894335.8	97.5
01003-07-10	250.5ppb PFOS	74	H010131A.M HILL0074.D	10	4.120	2067633.0	247.4	4.821	1166542.9	250.7	5.157	6910958.0	247.4
01003-07-11	400.8ppb PFOS	75	H010131A.M HILL0075.D	11	4.115	2096577.1	255.5	4.822	1145464.9	250.7	5.159	10501242.0	402.7
01003-07-12	501.0ppb PFOS	76	H010131A.M HILL0076.D	12	4.120	2113937.3	255.7	4.820	1154010.9	250.7	5.219	12580502.0	494.2
01003-07-13	1002.0ppb PFOS	77	H010131A.M HILL0077.D	13	4.120	2036590.3	253.5	4.821	1121090.4	250.7	5.164	23094802.0	1314.3
MeOH Blank	TN-A-4740, 04 Dec 00	78	H010131A.M HILL0078.D	95	4.120	8770.0	0.0	0.000	0.0	0.0	5.157	7282.3	0.9
MeOH Blank	TN-A-4740, 04 Dec 00	79	H010131A.M HILL0079.D	95	4.119	11212.9	0.0	0.000	0.0	0.0	5.155	6371.0	0.9

-1.3

-1.1

15.3

2.3

-2.1

-1.7

-0.9

-2.2

-2.7

-1.2

0.5

-1.4

PFOS in 3.5% NaCl Solution
Shake Flask Method
Day 2

RAW DATA

Agilent 1100 HPLC/MSD "Hilary"

Acquisition Run: H010205

Raw Data

Sample ID	Sample Description	Run#	File Name	Exp File	Vol	PFOS	PFOS Amt	PFOS Amt	PFOS Amt	PFOS Amt	PFOS Amt	PFOS Amt	PFOS Amt	PFOS Amt	PFOS Amt
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	1	H010205A.M	HILL0001.D	91	4.123	41176.2	0.0	0.000	0.0	0.0	5.153	30162.1	0.0	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	2	H010205A.M	HILL0002.D	91	4.119	9599.3	0.0	0.000	0.0	0.0	5.156	3212.3	0.0	
01003-07-13	1002.0ppb PFOS	3	H010205A.M	HILL0003.D	13	4.121	1774643.6	296.4	4.821	873165.5	250.7	5.263	19985400.0	1124.4	
01003-07-13	1002.0ppb PFOS	4	H010205A.M	HILL0004.D	13	4.118	1808354.9	298.3	4.819	884255.3	250.7	5.253	20450016.0	1140.0	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	5	H010205A.M	HILL0005.D	91	4.121	9271.7	0.0	0.000	0.0	0.0	5.158	18446.0	0.0	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	6	H010205A.M	HILL0006.D	91	4.118	12252.3	0.0	0.000	0.0	0.0	5.162	4197.1	0.0	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	7	H010205A.M	HILL0007.D	91	4.121	13494.0	0.0	0.000	0.0	0.0	5.157	7831.2	0.0	
01003-07-01	0ppb PFOS	8	H010205A.M	HILL0008.D	1	4.120	1837110.1	288.1	4.820	930048.6	250.7	5.156	11402.0	0.0	
01003-07-02	2.5ppb PFOS	9	H010205A.M	HILL0009.D	2	4.119	1829781.3	281.8	4.819	947156.8	250.7	5.156	86371.4	1.7	
01003-07-03	5ppb PFOS	10	H010205A.M	HILL0010.D	3	4.122	1874887.3	286.5	4.822	954459.8	250.7	5.158	142977.5	4.1	
01003-07-04	10ppb PFOS	11	H010205A.M	HILL0011.D	4	4.119	1836290.8	278.0	4.819	963263.3	250.7	5.155	266736.0	9.1	
01003-07-05	25ppb PFOS	12	H010205A.M	HILL0012.D	5	4.118	1854312.5	281.4	4.822	960941.4	250.7	5.162	697003.3	26.9	
01003-07-06	40ppb PFOS	13	H010205A.M	HILL0013.D	6	4.120	1834995.5	275.9	4.821	969887.6	250.7	5.157	1123236.8	44.2	
01003-07-07	50.1ppb PFOS	14	H010205A.M	HILL0014.D	7	4.117	1865783.1	277.9	4.821	979183.1	250.7	5.161	1391739.1	54.8	
01003-07-08	75.1ppb PFOS	15	H010205A.M	HILL0015.D	8	4.121	1861396.1	272.6	4.821	995988.6	250.7	5.158	2083089.4	81.9	
01003-07-09	100.2ppb PFOS	16	H010205A.M	HILL0016.D	9	4.120	1811954.0	265.8	4.820	994257.7	250.7	5.157	2734341.5	108.8	
01003-07-10	250.5ppb PFOS	17	H010205A.M	HILL0017.D	10	4.121	1848057.6	268.6	4.821	1003504.8	250.7	5.157	6425435.5	263.2	
01003-07-11	400.8ppb PFOS	18	H010205A.M	HILL0018.D	11	4.118	1811635.1	270.2	4.822	977732.8	250.7	5.232	9833563.0	427.5	
01003-07-12	501.0ppb PFOS	19	H010205A.M	HILL0019.D	12	4.119	1874128.5	271.6	4.819	1006423.3	250.7	5.226	11816095.0	507.1	
01003-07-13	1002.0ppb PFOS	20	H010205A.M	HILL0020.D	13	4.118	1919553.6	283.3	4.819	988284.3	250.7	5.225	21038770.0	1022.9	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	21	H010205A.M	HILL0021.D	92	4.119	13717.4	0.0	0.000	0.0	0.0	5.155	8349.7	0.0	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	22	H010205A.M	HILL0022.D	92	4.121	12992.0	0.0	0.000	0.0	0.0	5.158	13127.0	0.0	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	23	H010205A.M	HILL0023.D	92	4.120	12346.2	0.0	0.000	0.0	0.0	5.156	9888.0	0.0	
01003-07-01	0ppb PFOS	24	H010205A.M	HILL0024.D	1	4.119	1888101.0	264.0	4.819	1042922.9	250.7	5.156	12737.3	0.0	
01003-07-02	2.5ppb PFOS	25	H010205A.M	HILL0025.D	2	4.122	1894234.6	261.4	4.822	1057020.1	250.7	5.158	91752.4	1.6	
01003-07-03	5ppb PFOS	26	H010205A.M	HILL0026.D	3	4.121	1897593.9	256.3	4.821	1079665.3	250.7	5.157	161460.5	4.0	
01003-07-04	10ppb PFOS	27	H010205A.M	HILL0027.D	4	4.117	1880741.9	256.5	4.821	1069173.0	250.7	5.161	286824.4	8.7	-12.5
01003-07-05	25ppb PFOS	28	H010205A.M	HILL0028.D	5	4.121	1869055.4	256.7	4.821	1061883.6	250.7	5.157	718901.5	25.0	-0.4
01003-07-06	40ppb PFOS	29	H010205A.M	HILL0029.D	6	4.119	1881326.5	258.3	4.820	1062443.1	250.7	5.156	1151040.3	41.2	2.8
01003-07-07	50.1ppb PFOS	30	H010205A.M	HILL0030.D	7	4.120	1896198.5	258.7	4.821	1068797.5	250.7	5.157	1455018.6	52.4	4.6
01003-07-08	75.1ppb PFOS	31	H010205A.M	HILL0031.D	8	4.121	1845975.0	250.8	4.821	1073550.4	250.7	5.157	2161271.8	78.7	4.7
01003-07-09	100.2ppb PFOS	32	H010205A.M	HILL0032.D	9	4.119	1871880.4	256.5	4.820	1064144.0	250.7	5.156	2786907.8	103.5	3.3
01003-07-10	250.5ppb PFOS	33	H010205A.M	HILL0033.D	10	4.121	1880987.0	255.7	4.821	1072810.0	250.7	5.255	6624594.0	253.3	1.1
01003-07-11	400.8ppb PFOS	34	H010205A.M	HILL0034.D	11	4.118	1871845.4	263.3	4.822	1036707.3	250.7	5.162	10062423.0	411.2	2.6
01003-07-12	501.0ppb PFOS	35	H010205A.M	HILL0035.D	12	4.118	1881822.5	260.2	4.819	1054772.8	250.7	5.204	12373037.0	506.6	1.1
01003-07-13	1002.0ppb PFOS	36	H010205A.M	HILL0036.D	13	4.118	1995748.5	268.2	4.822	1030832.6	250.7	5.162	22023792.0	1027.6	2.6
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	37	H010205A.M	HILL0037.D	92	4.121	13125.2	0.0	0.000	0.0	0.0	5.157	13229.0	0.0	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	38	H010205A.M	HILL0038.D	92	4.118	13076.4	0.0	0.000	0.0	0.0	5.162	7287.5	0.0	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	39	H010205A.M	HILL0039.D	93	4.119	8488.4	0.0	0.000	0.0	0.0	5.155	3465.8	0.0	
TCR-002, 012901-nadPFOS-2.4.1, 100X Dil. Method Blank, SFM 02-01-01															
nadPFOS-2.4.1	MEE(MLA)	40	H010205A.M	HILL0040.D	21	4.119	1943546.6	271.9	4.819	1042521.5	250.7	5.155	8906.6	0.0	
TCR-002, 012901-nadPFOS-2.4.2, 100X Dil. Method Blank, SFM 02-01-01															
nadPFOS-2.4.2	MEE(MLA)	41	H010205A.M	HILL0041.D	22	4.119	1900756.8	263.2	4.819	1053132.4	250.7	5.155	16638.0	0.0	

TCR-002, 012901-nadPFOS-2.4.3, 100X													
Dil. Method Blank, SFM 02-01-01													
nadPFOS-2.4.3	MEE(MLA)	42	H010205A.M HILL0042.D	23	4.117	1933177.9	272.1	4.821	1036265.4	250.7	5.161	5505.6	0.0
TCR-002, 012901-nadPFOS-2.4.4, 100X													
Dil. Method Blank, SFM 02-01-01													
nadPFOS-2.4.4	MEE(MLA)	43	H010205A.M HILL0043.D	24	4.120	1981495.4	276.1	4.820	1046513.6	250.7	5.156	5787.1	0.0
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	44	H010205A.M HILL0044.D	93	4.118	8589.0	0.0	0.000	0.0	0.0	5.162	1716.6	0.0
TCR-002, 012901-nadPFOS-2.1.1, 100X													
nadPFOS-2.1.1	Dil. SFM 02-01-01 MEE(MLA)	45	H010205A.M HILL0045.D	31	4.118	1954489.6	280.0	4.819	1018035.8	250.7	5.155	5360243.5	214.2
TCR-002, 012901-nadPFOS-2.1.2, 100X													
nadPFOS-2.1.2	Dil. SFM 02-01-01 MEE(MLA)	46	H010205A.M HILL0046.D	32	4.119	1995386.6	283.3	4.820	1027304.7	250.7	5.156	5515177.0	218.6
TCR-002, 012901-nadPFOS-2.1.3, 100X													
nadPFOS-2.1.3	Dil. SFM 02-01-01 MEE(MLA)	47	H010205A.M HILL0047.D	33	4.119	1973433.0	282.4	4.819	1019282.8	250.7	5.155	5645223.0	225.9
TCR-002, 012901-nadPFOS-2.1.4, 100X													
nadPFOS-2.1.4	Dil. SFM 02-01-01 MEE(MLA)	48	H010205A.M HILL0048.D	34	4.119	1916815.9	275.8	4.820	1013776.3	250.7	5.156	6158368.0	249.0
01003-07-09	100.2ppb PFOS, CCV	49	H010205A.M HILL0049.D	9	4.120	1907227.4	255.0	4.820	1090911.0	250.7	5.157	2803938.5	101.5
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	50	H010205A.M HILL0050.D	93	4.118	13009.6	0.0	0.000	0.0	0.0	5.161	2060.1	0.0
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	51	H010205A.M HILL0051.D	93	4.118	13189.6	0.0	0.000	0.0	0.0	5.162	2302.3	0.0
TCR-002, 012901-nadPFOS-2.2.1, 100X													
nadPFOS-2.2.1	Dil. SFM 02-01-01 MEE(MLA)	52	H010205A.M HILL0052.D	35	4.119	1966392.4	280.2	4.820	1023456.4	250.7	5.156	7113649.5	287.2
TCR-002, 012901-nadPFOS-2.2.2, 100X													
nadPFOS-2.2.2	Dil. SFM 02-01-01 MEE(MLA)	53	H010205A.M HILL0053.D	36	4.119	1897505.8	270.3	4.819	1023781.8	250.7	5.155	5223066.0	207.2
TCR-002, 012901-nadPFOS-2.2.3, 100X													
nadPFOS-2.2.3	Dil. SFM 02-01-01 MEE(MLA)	54	H010205A.M HILL0054.D	37	4.117	1880691.9	267.8	4.821	1024368.3	250.7	5.160	5134239.0	203.4
TCR-002, 012901-nadPFOS-2.2.4, 100X													
nadPFOS-2.2.4	Dil. SFM 02-01-01 MEE(MLA)	55	H010205A.M HILL0055.D	38	4.118	1976344.5	280.9	4.819	1026108.8	250.7	5.155	5143755.5	203.4
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	56	H010205A.M HILL0056.D	93	4.121	12975.6	0.0	0.000	0.0	0.0	5.158	7253.0	0.0
TCR-002, 012901-nadPFOS-2.3.1, 100X													
nadPFOS-2.3.1	Dil. SFM 02-01-01 MEE(MLA)	57	H010205A.M HILL0057.D	39	4.119	1949027.0	276.7	4.819	1027143.3	250.7	5.155	4691219.0	184.5
TCR-002, 012901-nadPFOS-2.3.2, 100X													
nadPFOS-2.3.2	Dil. SFM 02-01-01 MEE(MLA)	58	H010205A.M HILL0058.D	40	4.121	1899727.9	270.5	4.822	1024350.2	250.7	5.158	4632432.0	182.6
TCR-002, 012901-nadPFOS-2.3.3, 100X													
nadPFOS-2.3.3	Dil. SFM 02-01-01 MEE(MLA)	59	H010205A.M HILL0059.D	41	4.118	1893792.4	267.5	4.822	1032577.4	250.7	5.162	4642529.5	181.5
TCR-002, 012901-nadPFOS-2.3.4, 100X													
nadPFOS-2.3.4	Dil. SFM 02-01-01 MEE(MLA)	60	H010205A.M HILL0060.D	42	4.119	1925417.8	272.4	4.819	1031034.4	250.7	5.155	4582600.5	179.4
01003-07-09	100.2ppb PFOS, CCV	61	H010205A.M HILL0061.D	9	4.120	1878686.8	250.2	4.821	1095013.0	250.7	5.157	2811571.0	101.4
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	62	H010205A.M HILL0062.D	93	4.120	9121.0	0.0	0.000	0.0	0.0	5.157	7782.5	0.0
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	63	H010205A.M HILL0063.D	94	4.122	12767.6	0.0	0.000	0.0	0.0	5.158	6500.7	0.0
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	64	H010205A.M HILL0064.D	94	4.119	8712.4	0.0	0.000	0.0	0.0	5.155	5702.5	0.0
01003-07-01	0ppb PFOS	65	H010205A.M HILL0065.D	1	4.121	1859950.3	243.4	4.821	1114555.1	250.7	5.158	8455.8	0.0
01003-07-02	2.5ppb PFOS	66	H010205A.M HILL0066.D	2	4.120	1851090.0	240.7	4.820	1121590.6	250.7	5.156	88443.7	1.3
01003-07-03	5ppb PFOS	67	H010205A.M HILL0067.D	3	4.117	1863924.8	238.1	4.821	1141707.3	250.7	5.161	158514.7	3.6
01003-07-04	10ppb PFOS	68	H010205A.M HILL0068.D	4	4.122	1858022.6	240.7	4.822	1125752.9	250.7	5.158	268391.2	7.6
01003-07-05	25ppb PFOS	69	H010205A.M HILL0069.D	5	4.121	1826781.8	236.4	4.821	1126887.1	250.7	5.157	711128.3	23.2
01003-07-06	40ppb PFOS	70	H010205A.M HILL0070.D	6	4.120	1873868.3	246.3	4.821	1109607.5	250.7	5.157	1137256.0	38.9
01003-07-07	50.1ppb PFOS	71	H010205A.M HILL0071.D	7	4.123	1789390.1	230.6	4.820	1131681.9	250.7	5.152	1464379.4	49.7
01003-07-08	75.1ppb PFOS	72	H010205A.M HILL0072.D	8	4.119	1851169.0	239.7	4.820	1126242.4	250.7	5.156	2179371.0	75.6
01003-07-09	100.2ppb PFOS	73	H010205A.M HILL0073.D	9	4.121	1895202.0	246.7	4.822	1120633.3	250.7	5.158	2826115.3	99.5
01003-07-10	250.5ppb PFOS	74	H010205A.M HILL0074.D	10	4.122	1881744.3	242.8	4.822	1130160.4	250.7	5.158	6735983.0	244.0
01003-07-11	400.8ppb PFOS	75	H010205A.M HILL0075.D	11	4.120	1806523.5	240.8	4.821	1093945.6	250.7	5.220	10220093.0	394.5
01003-07-12	501.0ppb PFOS	76	H010205A.M HILL0076.D	12	4.118	1874440.5	248.7	4.822	1099099.3	250.7	5.211	12558129.0	492.0
01003-07-13	1002.0ppb PFOS	77	H010205A.M HILL0077.D	13	4.121	1874729.0	252.1	4.821	1084462.3	250.7	5.213	22272594.0	977.2
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	78	H010205A.M HILL0078.D	94	4.119	13199.4	0.0	0.000	0.0	0.0	5.156	10110.2	0.0
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	79	H010205A.M HILL0079.D	94	4.121	12154.5	0.0	0.000	0.0	0.0	5.165	4001.3	0.0

PFOS in 3.5% NaCl Solution
Shake Flask Method
Day 3

RAW DATA

Agilent 1100 HPLC/MSD "Hillary"

Acquisition Run: H010206

Raw Data

Sample ID	Sample Description	Run #	Run Method	Det. ID	Vol.	PFOS RT	PFOS Area	PFOS Amount ng/mL	THPFOS RT	THPFOS Area	THPFOS Amount ng/mL	PFOS RT	PFOS Area	PFOS Amount ng/mL	PFOS % Err. Amount
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	1	H010206A.M	HILL0001.D	95	4.102	47304.4	0.0	0.000	0.0	0.0	5.153	6427.4	0.1	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	2	H010206A.M	HILL0002.D	95	4.104	9621.8	0.0	0.000	0.0	0.0	5.162	1366.8	0.1	
01003-07-13	1002.0ppb PFOS	3	H010206A.M	HILL0003.D	13	4.108	1891799.6	299.3	4.822	967438.5	250.7	5.208	22423620.0	1538.4	
01003-07-13	1002.0ppb PFOS	4	H010206A.M	HILL0004.D	13	4.108	1887421.0	288.1	4.823	1002759.7	250.7	5.208	21458074.0	1251.5	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	5	H010206A.M	HILL0005.D	95	4.105	13291.9	0.0	0.000	0.0	0.0	5.155	10494.2	0.1	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	6	H010206A.M	HILL0006.D	95	4.105	13218.0	0.0	0.000	0.0	0.0	5.156	3099.5	0.1	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	7	H010206A.M	HILL0007.D	95	4.105	12931.8	0.0	0.000	0.0	0.0	5.155	7381.7	0.1	
01003-07-01	0ppb PFOS	8	H010206A.M	HILL0008.D	1	4.104	1808761.3	266.2	4.822	1039861.1	250.7	5.162	12818.6	0.6	
01003-07-02	2.5ppb PFOS	9	H010206A.M	HILL0009.D	2	4.106	1870716.5	268.4	4.821	1066850.1	250.7	5.157	86415.2	3.3	
01003-07-03	5ppb PFOS	10	H010206A.M	HILL0010.D	3	4.106	1907032.1	267.0	4.821	1092983.6	250.7	5.157	158469.1	5.7	
01003-07-04	10ppb PFOS	11	H010206A.M	HILL0011.D	4	4.106	1843009.6	264.8	4.820	1065255.5	250.7	5.156	273021.3	10.0	
01003-07-05	25ppb PFOS	12	H010206A.M	HILL0012.D	5	4.108	1847886.0	264.4	4.822	1069764.8	250.7	5.158	709143.3	25.8	
01003-07-06	40ppb PFOS	13	H010206A.M	HILL0013.D	6	4.105	1841950.1	262.6	4.820	1073389.1	250.7	5.156	1155762.5	42.1	
01003-07-07	50.1ppb PFOS	14	H010206A.M	HILL0014.D	7	4.104	1852270.8	262.2	4.822	1081077.6	250.7	5.162	1429296.4	51.8	
01003-07-08	75.1ppb PFOS	15	H010206A.M	HILL0015.D	8	4.106	1849483.9	263.2	4.820	1075265.0	250.7	5.156	2158808.0	79.2	
01003-07-09	100.2ppb PFOS	16	H010206A.M	HILL0016.D	9	4.106	1833357.3	261.5	4.820	1072851.6	250.7	5.156	2797833.8	103.5	
01003-07-10	250.5ppb PFOS	17	H010206A.M	HILL0017.D	10	4.106	1780889.1	251.6	4.820	1083292.4	250.7	5.240	6612530.5	252.6	
01003-07-11	400.8ppb PFOS	18	H010206A.M	HILL0018.D	11	4.104	1870953.8	272.3	4.822	1051607.1	250.7	5.183	10170355.0	420.9	
01003-07-12	501.0ppb PFOS	19	H010206A.M	HILL0019.D	12	4.104	1863719.8	267.6	4.822	1065822.9	250.7	5.211	12424400.0	523.9	
01003-07-13	1002.0ppb PFOS	20	H010206A.M	HILL0020.D	13	4.102	1820228.4	266.8	4.820	1044313.8	250.7	5.180	22065096.0	1219.6	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	21	H010206A.M	HILL0021.D	96	4.107	12848.0	0.0	0.000	0.0	0.0	5.158	3335.9	0.1	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	22	H010206A.M	HILL0022.D	96	4.105	12476.6	0.0	0.000	0.0	0.0	5.155	6796.1	0.1	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	23	H010206A.M	HILL0023.D	96	4.104	12057.4	0.0	0.000	0.0	0.0	5.162	6750.2	0.1	
01003-07-01	0ppb PFOS	24	H010206A.M	HILL0024.D	1	4.107	1840212.0	257.8	4.821	1092359.3	250.7	5.157	12639.4	0.6	0.0
01003-07-02	2.5ppb PFOS	25	H010206A.M	HILL0025.D	2	4.105	1846254.5	256.1	4.820	1103289.0	250.7	5.156	89759.2	3.3	0.0
01003-07-03	5ppb PFOS	26	H010206A.M	HILL0026.D	3	4.105	1865722.9	253.7	4.819	1125402.9	250.7	5.155	159703.8	5.6	12.3
01003-07-04	10ppb PFOS	27	H010206A.M	HILL0027.D	4	4.108	1863265.3	257.9	4.822	1105801.0	250.7	5.158	268012.8	9.5	-5.0
01003-07-05	25ppb PFOS	28	H010206A.M	HILL0028.D	5	4.104	1894610.1	262.2	4.822	1105694.1	250.7	5.162	713836.3	25.2	0.3
01003-07-06	40ppb PFOS	29	H010206A.M	HILL0029.D	6	4.105	1861333.8	258.4	4.819	1102618.5	250.7	5.155	1132725.6	40.1	0.1
01003-07-07	50.1ppb PFOS	30	H010206A.M	HILL0030.D	7	4.106	1830163.8	252.4	4.821	1109924.9	250.7	5.157	1441188.8	50.8	1.5
01003-07-08	75.1ppb PFOS	31	H010206A.M	HILL0031.D	8	4.106	1851626.5	256.3	4.820	1105607.3	250.7	5.156	2124345.5	75.7	0.7
01003-07-09	100.2ppb PFOS	32	H010206A.M	HILL0032.D	9	4.105	1833460.3	253.8	4.819	1105475.6	250.7	5.155	2806380.8	100.7	0.5
01003-07-10	250.5ppb PFOS	33	H010206A.M	HILL0033.D	10	4.106	1855395.4	256.2	4.821	1108144.0	250.7	5.157	6668410.0	248.7	-0.7
01003-07-11	400.8ppb PFOS	34	H010206A.M	HILL0034.D	11	4.106	1858516.4	262.0	4.821	1085765.9	250.7	5.241	9982846.0	397.2	-0.9
01003-07-12	501.0ppb PFOS	35	H010206A.M	HILL0035.D	12	4.104	1891111.4	265.7	4.822	1089443.1	250.7	5.197	12576619.0	517.8	3.4
01003-07-13	1002.0ppb PFOS	36	H010206A.M	HILL0036.D	13	4.104	1859519.9	272.2	4.822	1045348.4	250.7	5.169	22259990.0	1238.9	0.0
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	37	H010206A.M	HILL0037.D	96	4.107	12090.8	0.0	0.000	0.0	0.0	5.165	6525.8	0.1	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	38	H010206A.M	HILL0038.D	96	4.104	11984.1	0.0	0.000	0.0	0.0	5.161	6927.2	0.1	
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	39	H010206A.M	HILL0039.D	97	4.107	11526.1	0.0	0.000	0.0	0.0	0.000	0.0	0.0	
TCR-002, 012901-nacIPFOS-3.4.1, 100X Dil. Method Blank, SFM 02-02-01															
naclPFOS-3.4.1	MEE(MLA)	40	H010206A.M	HILL0040.D	51	4.108	1926670.8	278.3	4.823	1059642.9	250.7	5.152	8463.8	0.5	
TCR-002, 012901-nacIPFOS-3.4.2, 100X Dil. Method Blank, SFM 02-02-01															
naclPFOS-3.4.2	MEE(MLA)	41	H010206A.M	HILL0041.D	52	4.106	2009192.8	290.4	4.820	1058807.8	250.7	5.156	3730.3	0.3	

TCR-002, 012901-nadPFOS-3.4.3, 100X													
Dil. Method Blank, SFM 02-02-01													
nadPFOS-3.4.3	MEE(MLA)	42	H010206A.M HILL0042.D	53	4.106	2025762.9	292.3	4.821	1060565.4	250.7	5.157	7621.1	0.4
TCR-002, 012901-nadPFOS-3.4.4, 100X													
Dil. Method Blank, SFM 02-02-01													
nadPFOS-3.4.4	MEE(MLA)	43	H010206A.M HILL0043.D	54	4.105	1979531.4	288.6	4.819	1049618.8	250.7	5.156	3744.8	0.3
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	44	H010206A.M HILL0044.D	97	4.106	11598.8	0.0	0.000	0.0	0.0	5.157	5353.4	0.1
TCR-002, 012901-nadPFOS-3.1.1, 100X													
nadPFOS-3.1.1	Dil. SFM 02-02-01 MEE(MLA)	45	H010206A.M HILL0045.D	61	4.104	1986989.8	298.8	4.822	1017566.9	250.7	5.162	5394307.0	217.1
TCR-002, 012901-nadPFOS-3.1.2, 100X													
nadPFOS-3.1.2	Dil. SFM 02-02-01 MEE(MLA)	46	H010206A.M HILL0046.D	62	4.104	1903896.9	281.9	4.822	1033535.4	250.7	5.161	5387695.5	213.3
TCR-002, 012901-nadPFOS-3.1.3, 100X													
nadPFOS-3.1.3	Dil. SFM 02-02-01 MEE(MLA)	47	H010206A.M HILL0047.D	63	4.105	1903997.0	283.7	4.820	1027231.4	250.7	5.156	5409418.0	215.6
TCR-002, 012901-nadPFOS-3.1.4, 100X													
nadPFOS-3.1.4	Dil. SFM 02-02-01 MEE(MLA)	48	H010206A.M HILL0048.D	64	4.107	2028305.4	305.1	4.821	1017373.9	250.7	5.158	5367317.5	216.0
01003-07-09	100.2ppb PFOS, CCV	49	H010206A.M HILL0049.D	9	4.107	1828271.5	250.2	4.821	1118280.6	250.7	5.157	2840861.5	100.7
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	50	H010206A.M HILL0050.D	97	0.000	0.0	0.0	0.000	0.0	0.0	5.155	7618.2	0.1
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	51	H010206A.M HILL0051.D	97	0.000	0.0	0.0	0.000	0.0	0.0	5.162	8197.7	0.1
TCR-002, 012901-nadPFOS-3.2.1, 100X													
nadPFOS-3.2.1	Dil. SFM 02-02-01 MEE(MLA)	52	H010206A.M HILL0052.D	65	4.104	1998596.6	297.3	4.819	1028671.3	250.7	5.155	5247156.5	208.4
TCR-002, 012901-nadPFOS-3.2.2, 100X													
nadPFOS-3.2.2	Dil. SFM 02-02-01 MEE(MLA)	53	H010206A.M HILL0053.D	66	4.105	2000593.5	298.4	4.819	1025976.1	250.7	5.155	5202058.5	207.1
TCR-002, 012901-nadPFOS-3.2.3, 100X													
nadPFOS-3.2.3	Dil. SFM 02-02-01 MEE(MLA)	54	H010206A.M HILL0054.D	67	4.107	2000672.9	299.4	4.821	1022749.9	250.7	5.157	5144285.5	205.3
TCR-002, 012901-nadPFOS-3.2.4, 100X													
nadPFOS-3.2.4	Dil. SFM 02-02-01 MEE(MLA)	55	H010206A.M HILL0055.D	68	4.105	1891713.3	280.7	4.819	1031479.1	250.7	5.155	5024824.0	198.5
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	56	H010206A.M HILL0056.D	97	0.000	0.0	0.0	0.000	0.0	0.0	5.155	6650.2	0.1
TCR-002, 012901-nadPFOS-3.3.1, 100X													
nadPFOS-3.3.1	Dil. SFM 02-02-01 MEE(MLA)	57	H010206A.M HILL0057.D	69	4.106	1896904.9	285.8	4.821	1015830.9	250.7	5.157	4756814.0	190.3
TCR-002, 012901-nadPFOS-3.3.2, 100X													
nadPFOS-3.3.2	Dil. SFM 02-02-01 MEE(MLA)	58	H010206A.M HILL0058.D	70	4.104	1956381.6	295.0	4.822	1015064.3	250.7	5.162	4726155.5	189.2
TCR-002, 012901-nadPFOS-3.3.3, 100X													
nadPFOS-3.3.3	Dil. SFM 02-02-01 MEE(MLA)	59	H010206A.M HILL0059.D	71	4.106	1902701.1	283.0	4.820	1028884.6	250.7	5.156	4696616.0	185.3
TCR-002, 012901-nadPFOS-3.3.4, 100X													
nadPFOS-3.3.4	Dil. SFM 02-02-01 MEE(MLA)	60	H010206A.M HILL0060.D	72	4.105	1894214.3	284.8	4.820	1017989.3	250.7	5.156	4754382.0	189.8
01003-07-09	100.2ppb PFOS, CCV	61	H010206A.M HILL0061.D	9	4.106	1756132.8	242.7	4.820	1107496.5	250.7	5.156	2809160.5	100.6
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	62	H010206A.M HILL0062.D	98	0.000	0.0	0.0	0.000	0.0	0.0	5.155	6589.1	0.1
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	63	H010206A.M HILL0063.D	98	4.104	11876.2	0.0	0.000	0.0	0.0	0.000	0.0	0.0
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	64	H010206A.M HILL0064.D	98	0.000	0.0	0.0	0.000	0.0	0.0	5.156	5586.4	0.1
01003-07-01	0ppb PFOS	65	H010206A.M HILL0065.D	1	4.105	1780994.1	245.2	4.819	1111747.3	250.7	5.156	12076.9	0.6
01003-07-02	2.5ppb PFOS	66	H010206A.M HILL0066.D	2	4.105	1755939.4	236.5	4.819	1136281.3	250.7	5.155	88457.2	3.1
01003-07-03	5ppb PFOS	67	H010206A.M HILL0067.D	3	4.104	1814153.1	241.7	4.822	1148491.8	250.7	5.161	156107.9	5.4
01003-07-04	10ppb PFOS	68	H010206A.M HILL0068.D	4	4.104	1790839.6	239.8	4.821	1143103.6	250.7	5.161	274913.9	9.4
01003-07-05	25ppb PFOS	69	H010206A.M HILL0069.D	5	4.106	1788983.0	243.8	4.821	1122996.1	250.7	5.157	709161.8	24.6
01003-07-06	40ppb PFOS	70	H010206A.M HILL0070.D	6	4.105	1770309.1	240.0	4.820	1129011.3	250.7	5.156	1158373.6	40.1
01003-07-07	50.1ppb PFOS	71	H010206A.M HILL0071.D	7	4.106	1832118.0	248.1	4.821	1130353.5	250.7	5.157	1436545.9	49.8
01003-07-08	75.1ppb PFOS	72	H010206A.M HILL0072.D	8	4.106	1799289.3	243.7	4.820	1129984.5	250.7	5.156	2169807.0	75.6
01003-07-09	100.2ppb PFOS	73	H010206A.M HILL0073.D	9	4.104	1739000.9	235.3	4.819	1131132.6	250.7	5.155	2826039.5	99.0
01003-07-10	250.5ppb PFOS	74	H010206A.M HILL0074.D	10	4.108	1788427.6	246.5	4.822	1110432.5	250.7	5.194	6744845.5	251.3
01003-07-11	400.8ppb PFOS	75	H010206A.M HILL0075.D	11	4.104	1755005.4	248.1	4.822	1082374.3	250.7	5.162	10142755.0	406.0
01003-07-12	501.0ppb PFOS	76	H010206A.M HILL0076.D	12	4.105	1787929.6	244.5	4.819	1119100.9	250.7	5.155	12195626.0	483.6
01003-07-13	1002.0ppb PFOS	77	H010206A.M HILL0077.D	13	4.107	1830473.3	263.4	4.822	1063516.4	250.7	5.193	22142466.0	1185.0
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	78	H010206A.M HILL0078.D	98	0.000	0.0	0.0	0.000	0.0	0.0	5.156	2995.9	0.1
MeOH Blank	TN-A-4755, 05 Feb 01, exp:2005.	79	H010206A.M HILL0079.D	98	4.106	11785.3	0.0	0.000	0.0	0.0	5.156	1708.9	0.1

PFOS in 3.5% NaCl Solution

Day 1

Hillary, H010131

Sample Data, DAY 1	Sample ID	Sample Description	Run#	Rep Mid	Data file	Vial#	PFOS RT	PFOS area	ng/mL PFOS	dilution factor correction µg/mL	purity factor correction µg/mL	Average Std Dev %CV
	naclPFOS-1.1.1	TCR-002, 012901-naclPFOS-1.1.1, 100X Dil. SFM 01-31-01 MEE(MLA)	45	H010131A.M	HILL0045.D	31	5.156	5915846.0	227.0	22.7	22.2	22.3
	naclPFOS-1.1.2	TCR-002, 012901-naclPFOS-1.1.2, 100X Dil. SFM 01-31-01 MEE(MLA)	46	H010131A.M	HILL0046.D	32	5.162	5851699.5	227.5	22.8	22.3	0.45
	naclPFOS-1.1.3	TCR-002, 012901-naclPFOS-1.1.3, 100X Dil. SFM 01-31-01 MEE(MLA)	47	H010131A.M	HILL0047.D	33	5.156	5827699.0	222.4	22.2	21.7	2.0%
	naclPFOS-1.1.4	TCR-002, 012901-naclPFOS-1.1.4, 100X Dil. SFM 01-31-01 MEE(MLA)	48	H010131A.M	HILL0048.D	34	5.162	5990611.0	232.7	23.3	22.8	
	naclPFOS-1.2.1	TCR-002, 012901-naclPFOS-1.2.1, 100X Dil. SFM 01-31-01 MEE(MLA)	52	H010131A.M	HILL0052.D	35	5.155	5222047.5	202.2	20.2	19.8	19.9
	naclPFOS-1.2.2	TCR-002, 012901-naclPFOS-1.2.2, 100X Dil. SFM 01-31-01 MEE(MLA)	53	H010131A.M	HILL0053.D	36	5.155	5201276.0	202.9	20.3	19.9	0.17
	naclPFOS-1.2.3	TCR-002, 012901-naclPFOS-1.2.3, 100X Dil. SFM 01-31-01 MEE(MLA)	54	H010131A.M	HILL0054.D	37	5.162	5192852.5	201.0	20.1	19.7	0.9%
	naclPFOS-1.2.4	TCR-002, 012901-naclPFOS-1.2.4, 100X Dil. SFM 01-31-01 MEE(MLA)	55	H010131A.M	HILL0055.D	38	5.161	5269990.5	204.9	20.5	20.1	
	naclPFOS-1.3.1	TCR-002, 012901-naclPFOS-1.3.1, 100X Dil. SFM 01-31-01 MEE(MLA)	57	H010131A.M	HILL0057.D	39	5.155	4172225.8	158.1	15.8	15.5	15.8
	naclPFOS-1.3.2	TCR-002, 012901-naclPFOS-1.3.2, 100X Dil. SFM 01-31-01 MEE(MLA)	58	H010131A.M	HILL0058.D	40	5.161	4317565.0	164.0	16.4	16.1	0.26
	naclPFOS-1.3.3	TCR-002, 012901-naclPFOS-1.3.3, 100X Dil. SFM 01-31-01 MEE(MLA)	59	H010131A.M	HILL0059.D	41	5.157	4187921.5	160.2	16.0	15.7	1.6%
	naclPFOS-1.3.4	TCR-002, 012901-naclPFOS-1.3.4, 100X Dil. SFM 01-31-01 MEE(MLA)	60	H010131A.M	HILL0060.D	42	5.157	4194090.0	162.2	16.2	15.9	

* Yellowed/grayed areas indicate points that are not used.
Day 1 data was omitted from the calculations because the %CV for the day was greater than 15%.

Day 1	
average	19.3 µg/mL
std dev	3.29
%C.V.	17.0%

PFOS in 3.5% NaCl Solution

Day 2

Hillary, H010205

Sample ID	Sample Description	Run	Rep	Mid	Pilot	Vial	PFOS RT	PFOS Amt	ng/ml PFOS (ppb)	dilution factor correction µg/mL	purity factor correction µg/mL	Average	
												Std Dev	%CV
nadPFOS-2.1.1	TCR-002, 012901-nadPFOS-2.1.1, 100X Dil. SFM 02-01-01 MEE(MLA)	45	H010205A.M	HILL0045.D	31	5.155	5360243.5	214.2	21.4	21.0		22.2	
nadPFOS-2.1.2	TCR-002, 012901-nadPFOS-2.1.2, 100X Dil. SFM 02-01-01 MEE(MLA)	46	H010205A.M	HILL0046.D	32	5.156	5515177.0	218.6	21.9	21.4		1.52	
nadPFOS-2.1.3	TCR-002, 012901-nadPFOS-2.1.3, 100X Dil. SFM 02-01-01 MEE(MLA)	47	H010205A.M	HILL0047.D	33	5.155	5645223.0	225.9	22.6	22.1		6.8%	
nadPFOS-2.1.4	TCR-002, 012901-nadPFOS-2.1.4, 100X Dil. SFM 02-01-01 MEE(MLA)	48	H010205A.M	HILL0048.D	34	5.156	6158368.0	249.0	24.9	24.4			
nadPFOS-2.2.1	TCR-002, 012901-nadPFOS-2.2.1, 100X Dil. SFM 02-01-01 MEE(MLA)	52	H010205A.M	HILL0052.D	35	5.156	7113649.5	287.2	28.7	28.1		20.0	
nadPFOS-2.2.2	TCR-002, 012901-nadPFOS-2.2.2, 100X Dil. SFM 02-01-01 MEE(MLA)	53	H010205A.M	HILL0053.D	36	5.155	5223066.0	207.2	20.7	20.3		0.23	
nadPFOS-2.2.3	TCR-002, 012901-nadPFOS-2.2.3, 100X Dil. SFM 02-01-01 MEE(MLA)	54	H010205A.M	HILL0054.D	37	5.160	5134239.0	203.4	20.3	19.9		1.2%	
nadPFOS-2.2.4	TCR-002, 012901-nadPFOS-2.2.4, 100X Dil. SFM 02-01-01 MEE(MLA)	55	H010205A.M	HILL0055.D	38	5.155	5143755.5	203.4	20.3	19.9			
nadPFOS-2.3.1	TCR-002, 012901-nadPFOS-2.3.1, 100X Dil. SFM 02-01-01 MEE(MLA)	57	H010205A.M	HILL0057.D	39	5.155	4691219.0	184.5	18.5	18.1		17.8	
nadPFOS-2.3.2	TCR-002, 012901-nadPFOS-2.3.2, 100X Dil. SFM 02-01-01 MEE(MLA)	58	H010205A.M	HILL0058.D	40	5.158	4632432.0	182.6	18.3	17.9		0.25	
nadPFOS-2.3.3	TCR-002, 012901-nadPFOS-2.3.3, 100X Dil. SFM 02-01-01 MEE(MLA)	59	H010205A.M	HILL0059.D	41	5.182	4642529.5	181.5	18.2	17.8		1.4%	
nadPFOS-2.3.4	TCR-002, 012901-nadPFOS-2.3.4, 100X Dil. SFM 02-01-01 MEE(MLA)	60	H010205A.M	HILL0060.D	42	5.155	4582600.5	179.4	17.9	17.5			

* Yellowed/grayed areas indicate points that are not used.

Day 2
 average: 20.0 µg/mL
 std dev: 2.20
 %C.V.: 11.0%

PFOS in 3.5% NaCl Solution

Day 3

Hillary, 010206

Sample ID	Sample Description	Run#	Rep. No.	Batch#	Vol.	PFOS (ng)	PFOS (ng)	ng/mL PFOS (ppb)	dilution factor correction	purification factor correction	Average
naclPFOS-3.1.1	TCR-002, 012901-naclPFOS-3.1.1, 100X Dil. SFM 02-02-01 MEE(MLA)	45	H010206A.M	HILL0045.D	61	5.162	5394307.0	217.1	21.7	21.2	21.1
naclPFOS-3.1.2	TCR-002, 012901-naclPFOS-3.1.2, 100X Dil. SFM 02-02-01 MEE(MLA)	46	H010206A.M	HILL0046.D	62	5.161	5387695.5	213.3	21.3	20.9	0.13
naclPFOS-3.1.3	TCR-002, 012901-naclPFOS-3.1.3, 100X Dil. SFM 02-02-01 MEE(MLA)	47	H010206A.M	HILL0047.D	63	5.156	5409419.0	215.6	21.6	21.1	0.6%
naclPFOS-3.1.4	TCR-002, 012901-naclPFOS-3.1.4, 100X Dil. SFM 02-02-01 MEE(MLA)	48	H010206A.M	HILL0048.D	64	5.158	5367317.5	216.0	21.6	21.1	
naclPFOS-3.2.1	TCR-002, 012901-naclPFOS-3.2.1, 100X Dil. SFM 02-02-01 MEE(MLA)	52	H010206A.M	HILL0052.D	65	5.155	5247156.5	208.4	20.8	20.4	20.1
naclPFOS-3.2.2	TCR-002, 012901-naclPFOS-3.2.2, 100X Dil. SFM 02-02-01 MEE(MLA)	53	H010206A.M	HILL0053.D	66	5.155	5202058.5	207.1	20.7	20.3	0.45
naclPFOS-3.2.3	TCR-002, 012901-naclPFOS-3.2.3, 100X Dil. SFM 02-02-01 MEE(MLA)	54	H010206A.M	HILL0054.D	67	5.157	5144285.5	205.3	20.5	20.1	2.2%
naclPFOS-3.2.4	TCR-002, 012901-naclPFOS-3.2.4, 100X Dil. SFM 02-02-01 MEE(MLA)	55	H010206A.M	HILL0055.D	68	5.155	5024824.0	198.5	19.8	19.4	
naclPFOS-3.3.1	TCR-002, 012901-naclPFOS-3.3.1, 100X Dil. SFM 02-02-01 MEE(MLA)	57	H010206A.M	HILL0057.D	69	5.157	4756814.0	190.3	19.0	18.6	18.5
naclPFOS-3.3.2	TCR-002, 012901-naclPFOS-3.3.2, 100X Dil. SFM 02-02-01 MEE(MLA)	58	H010206A.M	HILL0058.D	70	5.162	4726155.5	189.2	18.9	18.5	0.24
naclPFOS-3.3.3	TCR-002, 012901-naclPFOS-3.3.3, 100X Dil. SFM 02-02-01 MEE(MLA)	59	H010206A.M	HILL0059.D	71	5.156	4696616.0	185.3	18.5	18.1	1.3%
naclPFOS-3.3.4	TCR-002, 012901-naclPFOS-3.3.4, 100X Dil. SFM 02-02-01 MEE(MLA)	60	H010206A.M	HILL0060.D	72	5.156	4754382.0	189.8	19.0	18.6	

Yellowed/grayed areas indicate points that are not used.

Day 3
 average: 19.9 µg/mL µg/mL
 std dev: 1.31
 %C.V.: 6.6%

FACT-TCR002 PFOS Solubility of TCR-00017-46 in 3.5% NaCl Solution

Day 1 data source acquisition instrument/sequence: Agilent 1100 HPLC/MSD "Hillary"/H010131.s

Day 2 data source acquisition instrument/sequence: Agilent 1100 HPLC/MSD "Hillary"/H010205.s

Day 3 data source acquisition instrument/sequence: Agilent 1100 HPLC/MSD "Hillary"/H010206.s

DATA SUMMARY FOR INDIVIDUAL SAMPLES

Sample ID	Day 1 ppm	Day 2 ppm	Day 3 ppm
nacPFOS-1.1.1	22.7	22.2	22.3
nacPFOS-1.1.2	22.6	22.3	0.45
nacPFOS-1.1.3	22.2	21.7	2.0%
nacPFOS-1.1.4	23.3	22.8	
nacPFOS-1.2.1	20.2	19.8	18.9
nacPFOS-1.2.2	20.3	19.9	0.17
nacPFOS-1.2.3	20.1	19.7	0.3%
nacPFOS-1.2.4	20.5	20.1	
nacPFOS-1.3.1	15.8	15.5	15.8
nacPFOS-1.3.2	16.4	16.1	0.26
nacPFOS-1.3.3	16.0	15.7	1.6%
nacPFOS-1.3.4	16.2	15.9	
nacPFOS-2.1.1	21.4	21.0	22.2
nacPFOS-2.1.2	21.9	21.4	1.52
nacPFOS-2.1.3	22.6	22.1	6.8%
nacPFOS-2.1.4	24.9	24.4	
nacPFOS-2.2.1	28.7	28.1	20.0
nacPFOS-2.2.2	20.7	20.3	0.23
nacPFOS-2.2.3	20.3	19.9	1.2%
nacPFOS-2.2.4	20.3	19.9	
nacPFOS-2.3.1	18.5	18.1	17.8
nacPFOS-2.3.2	18.3	17.9	0.25
nacPFOS-2.3.3	18.2	17.8	1.4%
nacPFOS-2.3.4	17.9	17.5	
nacPFOS-3.1.1	21.7	21.2	21.1
nacPFOS-3.1.2	21.3	20.9	0.13
nacPFOS-3.1.3	21.6	21.1	0.6%
nacPFOS-3.1.4	21.6	21.1	
nacPFOS-3.2.1	20.8	20.4	20.1
nacPFOS-3.2.2	20.7	20.3	0.45
nacPFOS-3.2.3	20.5	20.1	2.2%
nacPFOS-3.2.4	19.8	19.4	
nacPFOS-3.3.1	19.0	18.6	18.5
nacPFOS-3.3.2	18.9	18.5	0.24
nacPFOS-3.3.3	18.5	18.1	1.3%
nacPFOS-3.3.4	19.0	18.6	

FACT-TCR002 PFOS Solubility of TCR-00017-46 in 3.5% NaCl Solution.**DAILY AND TOTAL DATA SUMMARIES****Day 1 data summary**

Average: 19.9 ppm

Std Dev: 4.99

%CV: 17.0%

(One standard greater than 1% CV)

Day 2 data summary

Average: 20.0 ppm

Std Dev: 2.20

%CV: 11.0%

(Sample 2.2.1 excluded via Q-Test)

Day 3 data summary

Average: 19.9 ppm

Std Dev: 1.31

%CV: 6.6%

TOTAL data summary

Average: 20.0 ppm

Std Dev: 0.07

%CV: 0.4%

Data from Days 2 and 3 are used.

Day 1 has been omitted from the calculations.

* Greyed / yellowed areas have been omitted from the calculations.

PFOS in 3.5% NaCl Solution Shake Flask Method

Q-TEST Data Calculations:

Sample ID:	naclPFOS-2.2.1 naclPFOS-2.2.2 naclPFOS-2.2.3 naclPFOS-2.2.4
Sample Conc., Rep1	28.1
Sample Conc., Rep2	20.3
Sample Conc., Rep3	19.9
Sample Conc., Rep4	19.9
Range:	8.20
Gap:	7.80
Qobs:	0.95
No. observations:	4
Retain?	no

Exclude rep 1!

Qobserved = gap/range where "gap" is the difference between the questionable data point and the closest value of the data set, and "range" is the difference between the highest and lowest value of the data set. If Q-observed is > Q-tabulated, then the data point may be excluded with 90% confidence. Q-tabulated for 4 observations (or data points) is 0.68. The samples highlighted in yellow in the above table have Q-observed > 0.68, and were therefore excluded from the reported data.

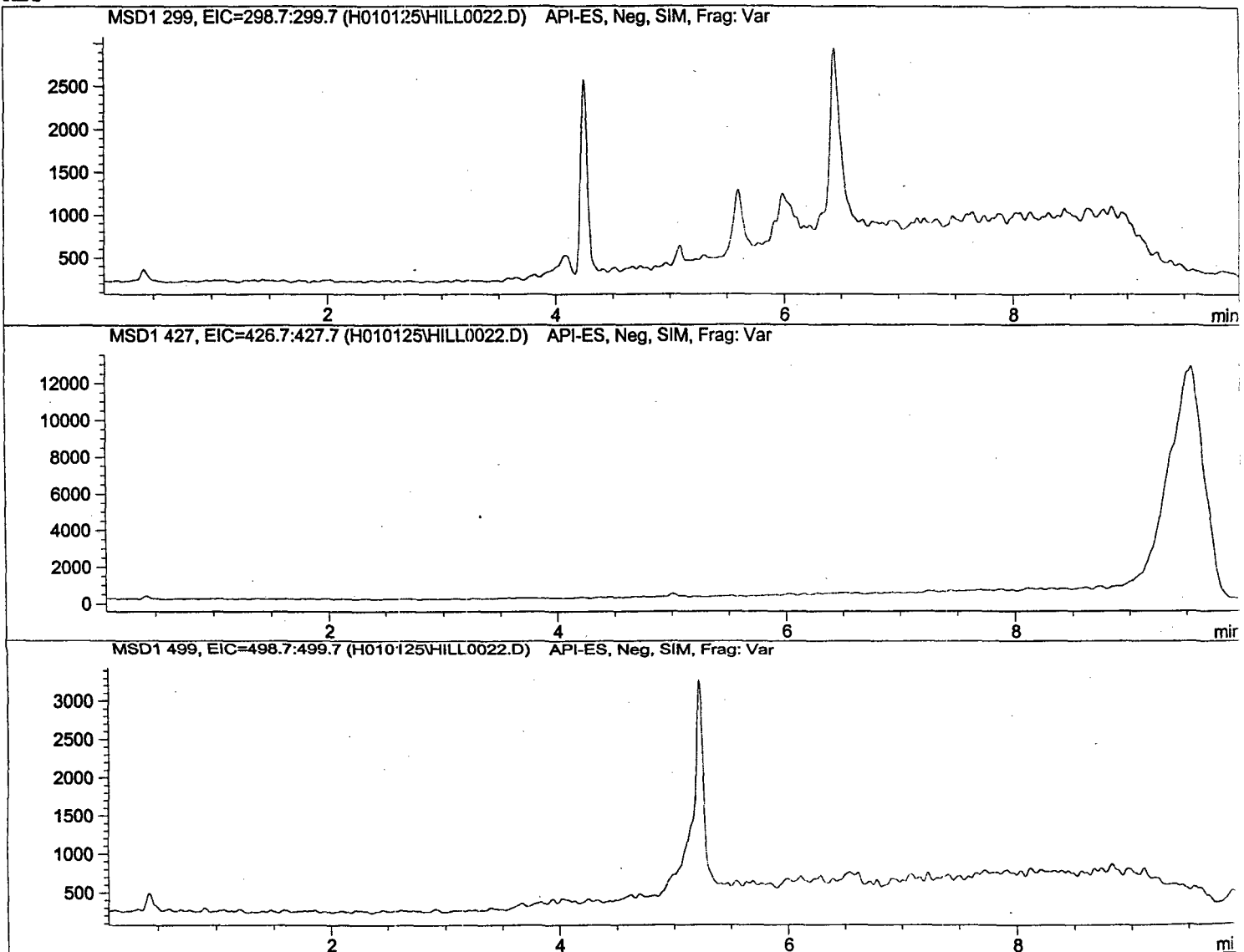
ATTACHMENT C: SAMPLE CHROMATOGRAMS

TN-A-4740, 04 Dec 00

=====

Injection Date	: 1/25/01 1:13:34 PM	Seq. Line	: 22
Sample Name	: MeOH Blank	Vial	: 92
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H0125A1.M		
Last changed	: 1/29/01 2:28:35 PM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil 5um 50X2 mm
Eluents: Methanol and 2 mm Ammonium Acetate
Reprocessed on "Dioana"
KLT



Internal Standard Report

Exact Copy of Original

Sorted By : Signal
Calib. Data Modified : Monday, January 29, 2001 2:15:31 PM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name

Initial

Date

#	[ng/mL]	
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.111	-	-	-	-	-	PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.864	-	-	-	-	-	THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.144	-	-	-	-	-	PFOS

Totals without ISTD(s) : 0.00000

1 Warnings or Errors :

Warning : ISTD compound(s) not found

=====
=====
Area Percent Report
=====

Sorted By : Signal
Calib. Data Modified : Monday, January 29, 2001 2:15:31 PM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 299, EIC=298.7:299.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	4.111	I	0.0000	0.00000	0.0000	PFBS

Totals : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	4.864	I	0.0000	0.00000	0.0000	THPFOS

Totals : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	5.144		0.0000	0.00000	0.0000	PFOS

Totals : 0.00000

1 Warnings or Errors :

Warning : ISTD compound(s) not found

=====
*** End of Report ***

TN-A-4740, 04 Dec 00

=====

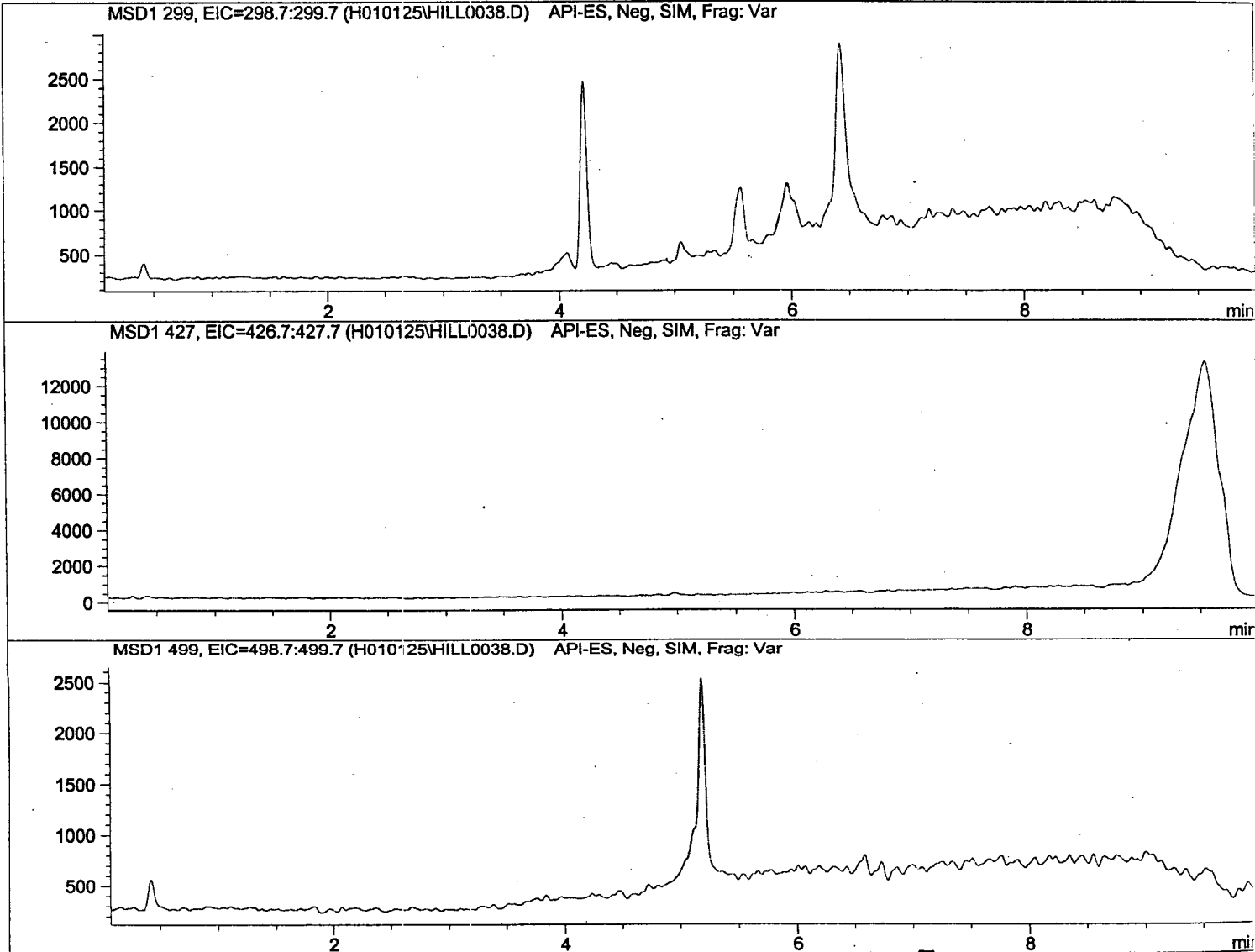
Injection Date	: 1/25/01 4:11:20 PM	Seq. Line	: 38
Sample Name	: MeOH Blank	Vial	: 92
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H0125A1.M		
Last changed	: 1/29/01 2:30:01 PM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

Internal Standard Report

MLA
Initial02 Apr 01
Date
page 1 of 3

Sorted By : Signal
Calib. Data Modified : Monday, January 29, 2001 2:15:31 PM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name

#	[ng/mL]	
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.111	-	-	-	-	-	PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.864	-	-	-	-	-	THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.144	-	-	-	-	-	PFOS

Totals without ISTD(s) : 0.00000

1 Warnings or Errors :

Warning : ISTD compound(s) not found

=====
 Area Percent Report
 =====

Sorted By : Signal
 Calib. Data Modified : Monday, January 29, 2001 2:15:31 PM
 Multiplier : 1.0000
 Dilution : 1.0000

Signal 1: MSD1 299, EIC=298.7:299.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	4.111	I	0.0000	0.00000	0.0000	PFBS

Totals : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	4.864	I	0.0000	0.00000	0.0000	THPFOS

Totals : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	5.144		0.0000	0.00000	0.0000	PFOS

Totals : 0.00000

1 Warnings or Errors :

Warning : ISTD compound(s) not found

=====
*** End of Report ***

TN-A-4740, 04 Dec 00

=====

Injection Date	: 1/30/01 12:10:30 PM	Seq. Line	: 122
Sample Name	: MeOH Blank	Vial	: 97
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010129.M		
Last changed	: 1/29/01 1:35:12 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010129B.M		
Last changed	: 1/31/01 11:08:44 AM by KLT/MLA		

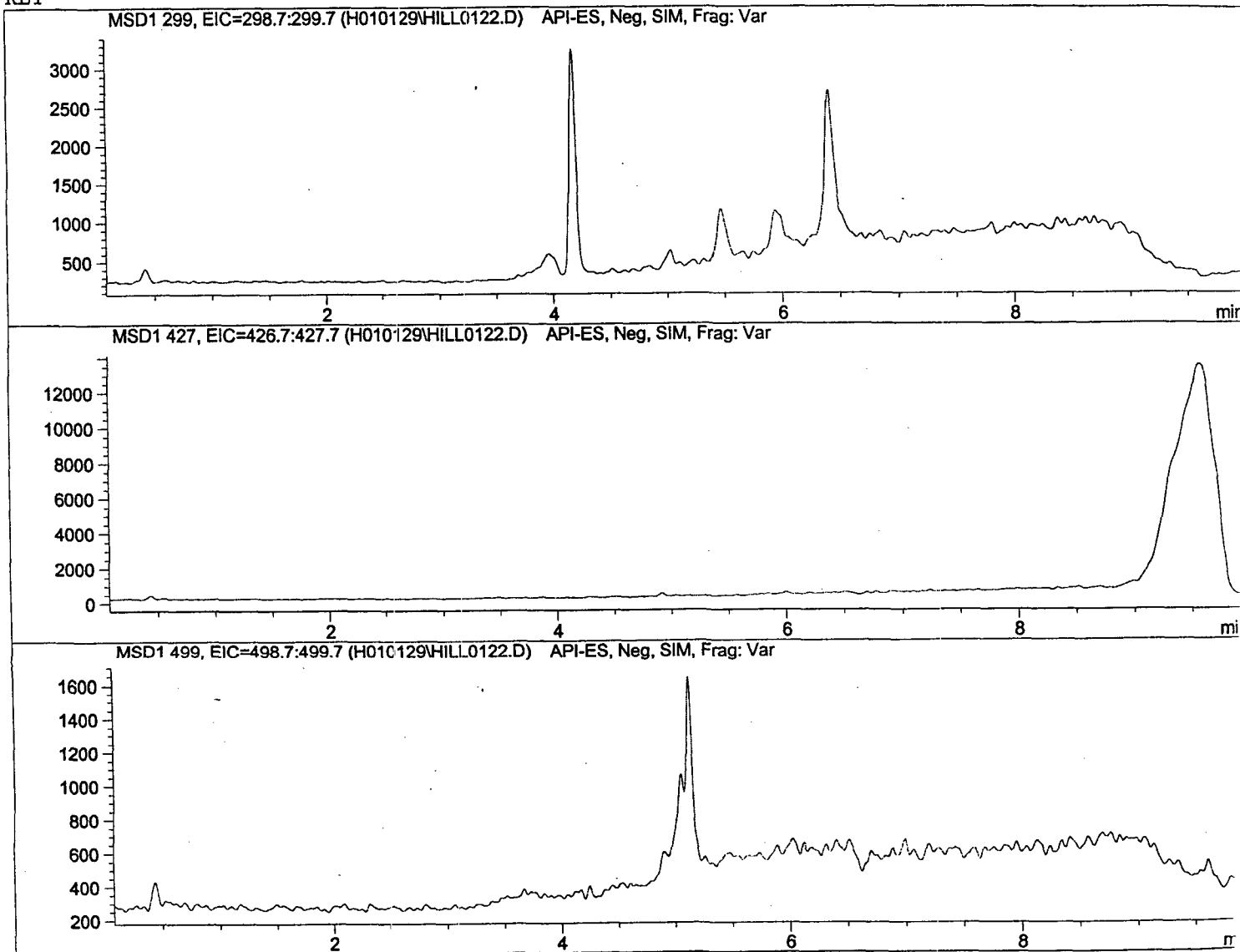
(modified after loading)

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



=====
Internal Standard Report
=====

Exact Copy of Original

Sorted By	:	Signal
Calib. Data Modified	:	1/31/01 11:08:42 AM
Multiplier	:	1.0000
Dilution	:	1.0000
Sample ISTD Information:		
ISTD ISTD Amount	Name	

MLA
Initial

03 Apr 01
Date

page 1 of 3

#	[ng/mL]	
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.111	-	-	-	-	-	PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.909	-	-	-	-	-	THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.144	-	-	-	-	-	PFOS

Totals without ISTD(s) : 0.00000

1 Warnings or Errors :

Warning : ISTD compound(s) not found

Area Percent Report

Sorted By : Signal
Calib. Data Modified : 1/31/01 11:08:42 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: MSD1 299, EIC=298.7:299.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	4.111	I	0.0000	0.00000	0.0000	PFBS

Totals : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	4.909	I	0.0000	0.00000	0.0000	THPFOS

Totals : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

Peak #	RetTime [min]	Type	Width [min]	Area	Area %	Name
1	5.144		0.0000	0.00000	0.0000	PFOS

Totals : 0.00000

1 Warnings or Errors :

Warning : ISTD compound(s) not found

=====
*** End of Report ***

TCR-002, 012201-swPFOS-1.4.2, 100X Dil.

Method Blank, SFM 01-24-01 MEE(MLA)

=====

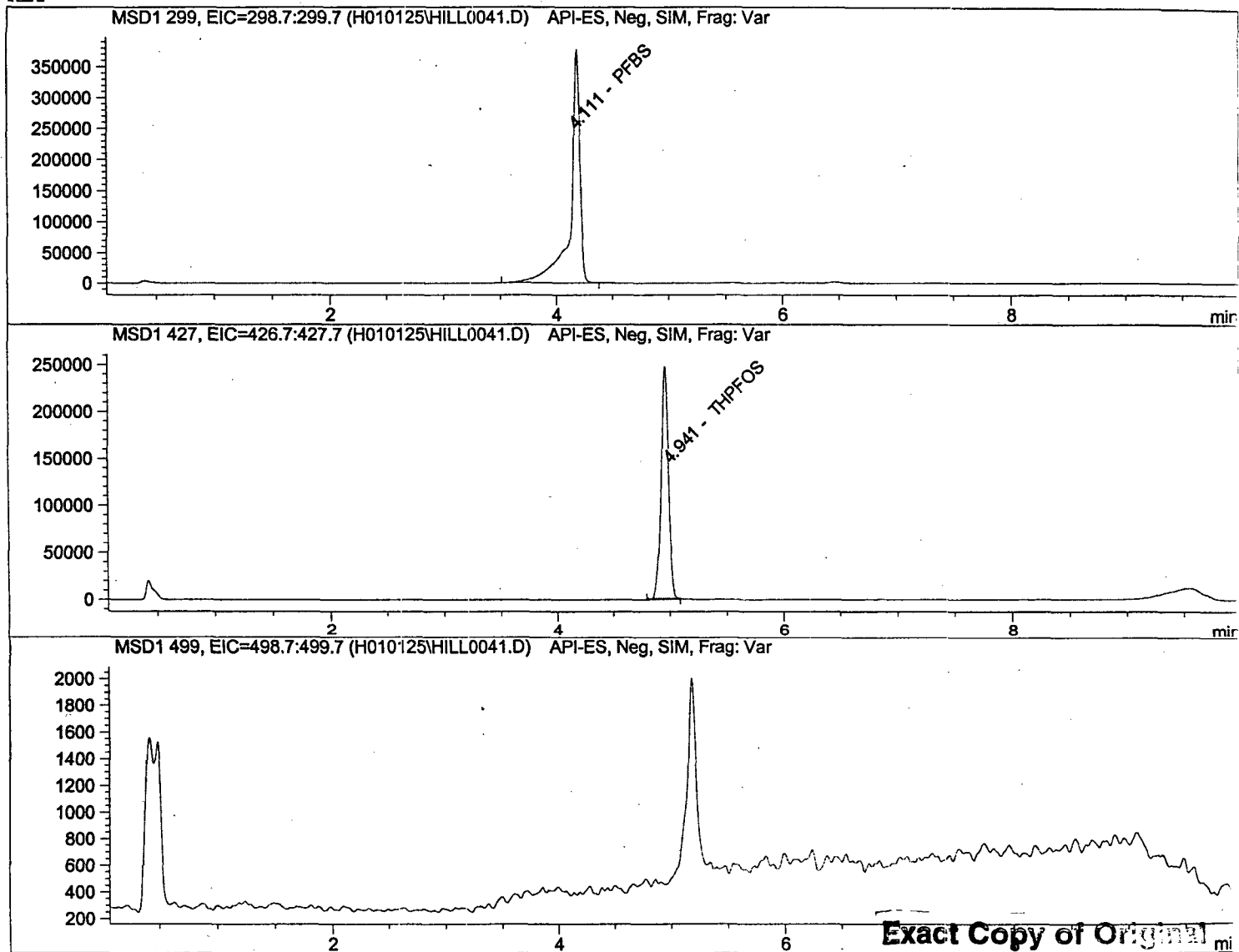
Injection Date	: 1/25/01 4:44:42 PM	Seq. Line	: 41
Sample Name	: swPFOS-1.4.2	Vial	: 22
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H0125A1.M		
Last changed	: 1/29/01 2:30:17 PM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

MLA
Initial02 Apr 01
Date

page 1 of 2

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : Monday, January 29, 2001 2:15:31 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD #	ISTD Amount [ng/mL]	Name
--------	---------------------	------

1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.111	VBA+ I	2.23757e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.941	PBA+ I	1.13828e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.144		-	-	-		PFOS

Totals without ISTD(s) : 0.00000

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-1.4.3, 100X Dil.

Method Blank, SFM 01-25-01 MEE (MLA)

=====

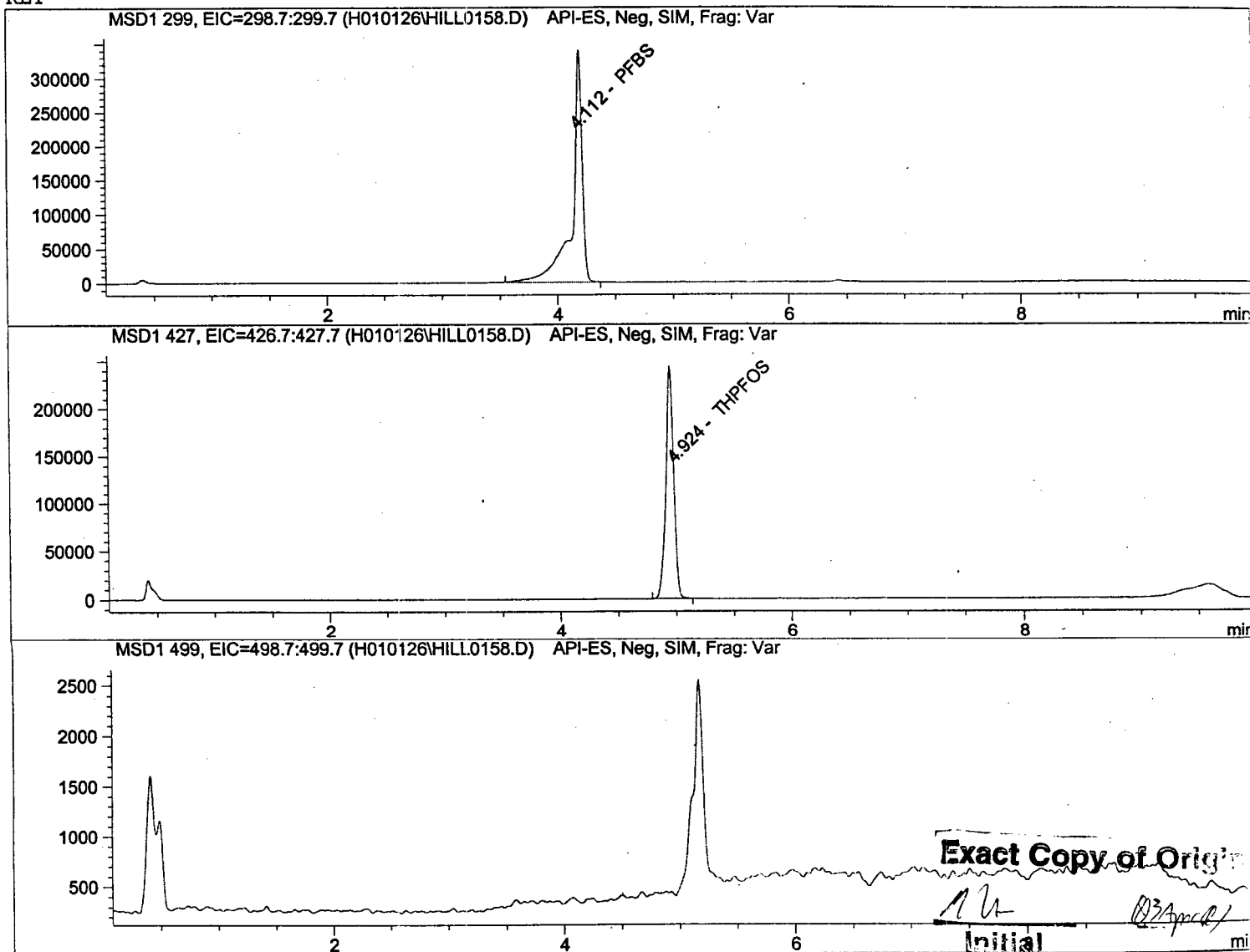
Injection Date	: 1/27/01 7:11:01 PM	Seq. Line	: 158
Sample Name	: swPFOS-2.4.3	Vial	: 61
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010126C.M		
Last changed	: 1/30/01 2:52:45 PM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : Tuesday, January 30, 2001 2:27:34 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD #	ISTD Amount [ng/mL]	Name
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.112	VBA+ I	2.05076e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.924	PBA+ I	1.12624e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.142		-	-	-		PFOS

Totals without ISTD(s) : 0.00000

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-3.4.3, 100X Dil.

Method Blank, SFM 01-26-01 MEE (MLA)

=====

Injection Date	: 1/30/01 8:16:24 AM	Seq. Line	: 101
Sample Name	: swPFOS-3.4.3	Vial	: 53
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010129.M		
Last changed	: 1/29/01 1:35:12 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010129B.M		
Last changed	: 1/31/01 11:08:44 AM by KLT/MLA		

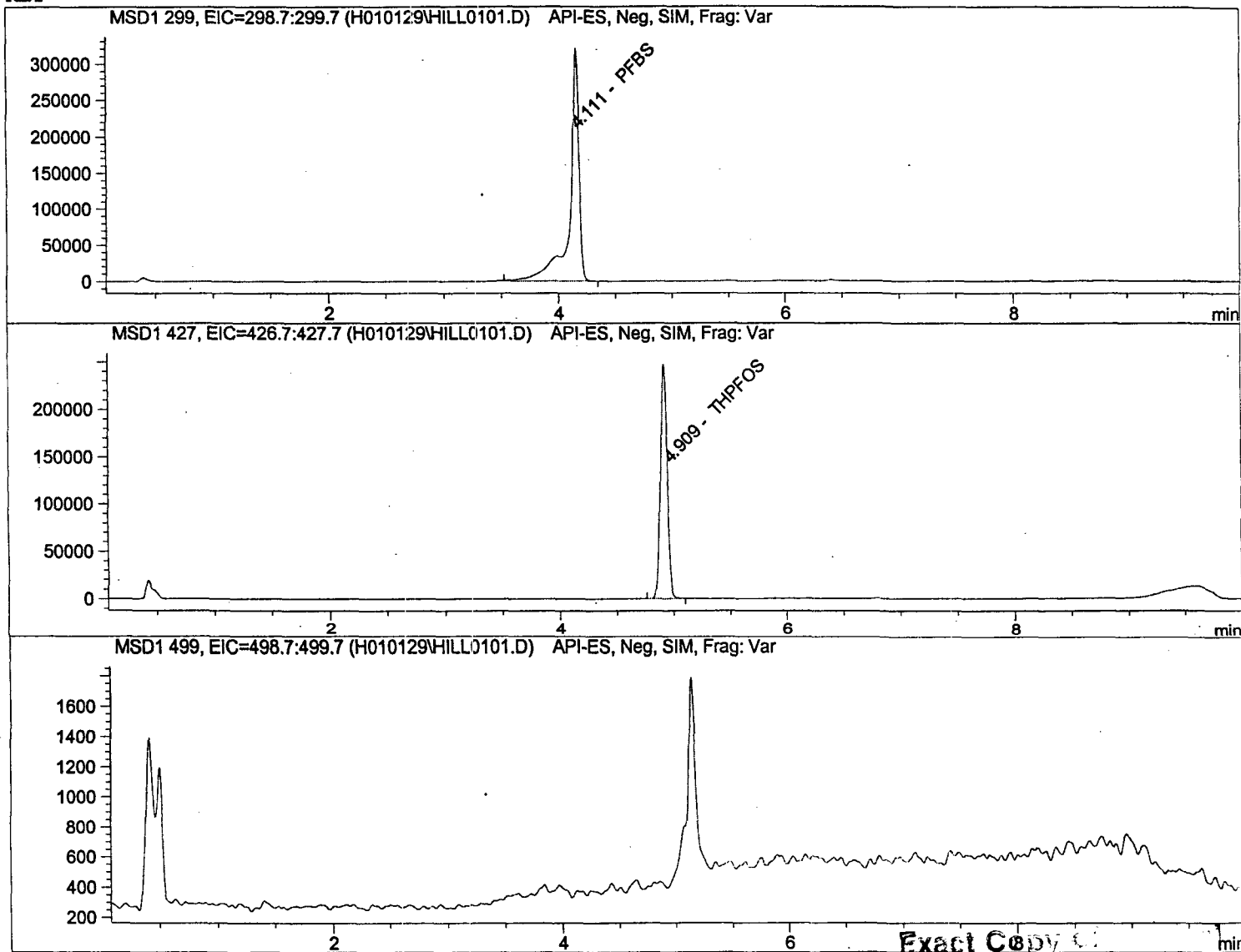
(modified after loading)

PFOS Analysis Column: C18 Betasil Sum 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT

MLA
Initial02 Apr 01
L. (initials)
page 1 of 1

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : 1/31/01 11:08:42 AM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name
[ng/mL]

#	ISTD	Amount [ng/mL]	Name
1	254.00000	PFBS	
2	254.40000	THPFOS	

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.111	VBA+ I	1.81230e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.909	PBA+ I	1.00441e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.144		-	-	-		PFOS

Totals without ISTD(s) : 0.00000

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-1.1.2, 100X Dil.

SFM 01-24-01 MEE (MLA)

=====

Injection Date	: 1/25/01 5:40:19 PM	Seq. Line	: 46
Sample Name	: swPFOS-1.1.2	Vial	: 32
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H0125A1.M		
Last changed	: 1/29/01 2:30:45 PM by KLT/MLA		

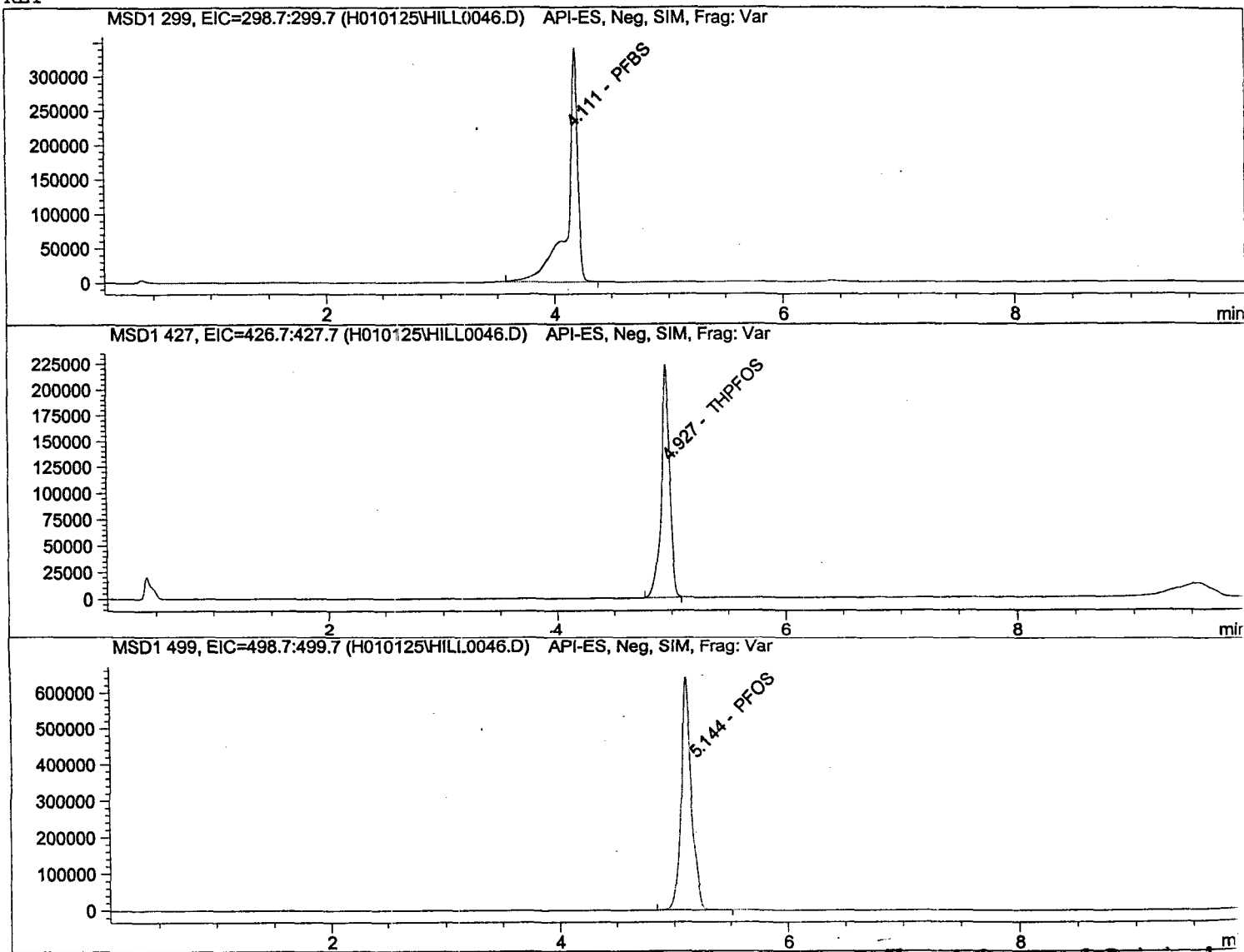
(modified after loading)

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

MLA
Initial03 Apr 01
Date

page 1 of 2

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : Monday, January 29, 2001 2:15:31 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name
[ng/mL]

#	ISTD Amount [ng/mL]	Name
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.111	VBA+ I	2.15890e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.927	VBA+ I	1.11676e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.144	VBA+	3.91905e6	1.45546e-1	129.93889		PFOS

Totals without ISTD(s) : 129.93889

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-1.2.2, 100X Dil.

SFM 01-24-01 MEE(MLA)

=====

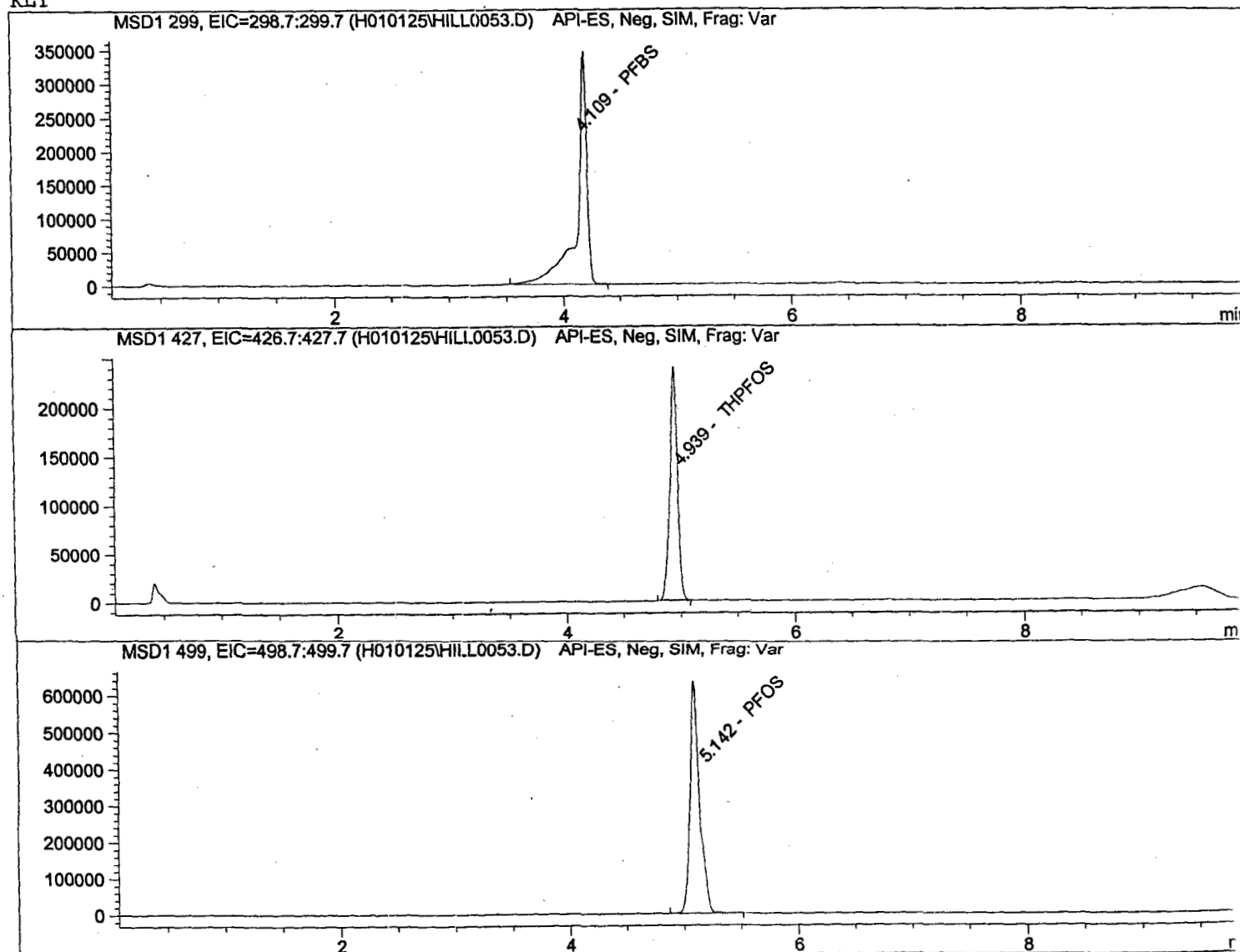
Injection Date	: 1/25/01 6:58:14 PM	Seq. Line	: 53
Sample Name	: swPFOS-1.2.2	Vial	: 36
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H0125A1.M		
Last changed	: 1/29/01 2:31:22 PM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil Sum 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT

**Exact Copy of Original**

MLA

initial

03 Apr 01

page 1 of 2

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : Monday, January 29, 2001 2:15:31 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name
[ng/mL]

#	ISTD Amount [ng/mL]	Name
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.109	VBA+ I	2.14016e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.939	VBA+ I	1.11329e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.142	PVA+	3.70294e6	1.45288e-1	122.93840		PFOS

Totals without ISTD(s) : 122.93840

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-1.3.1, 100X Dil.

SFM 01-24-01 MEE (MLA)

=====

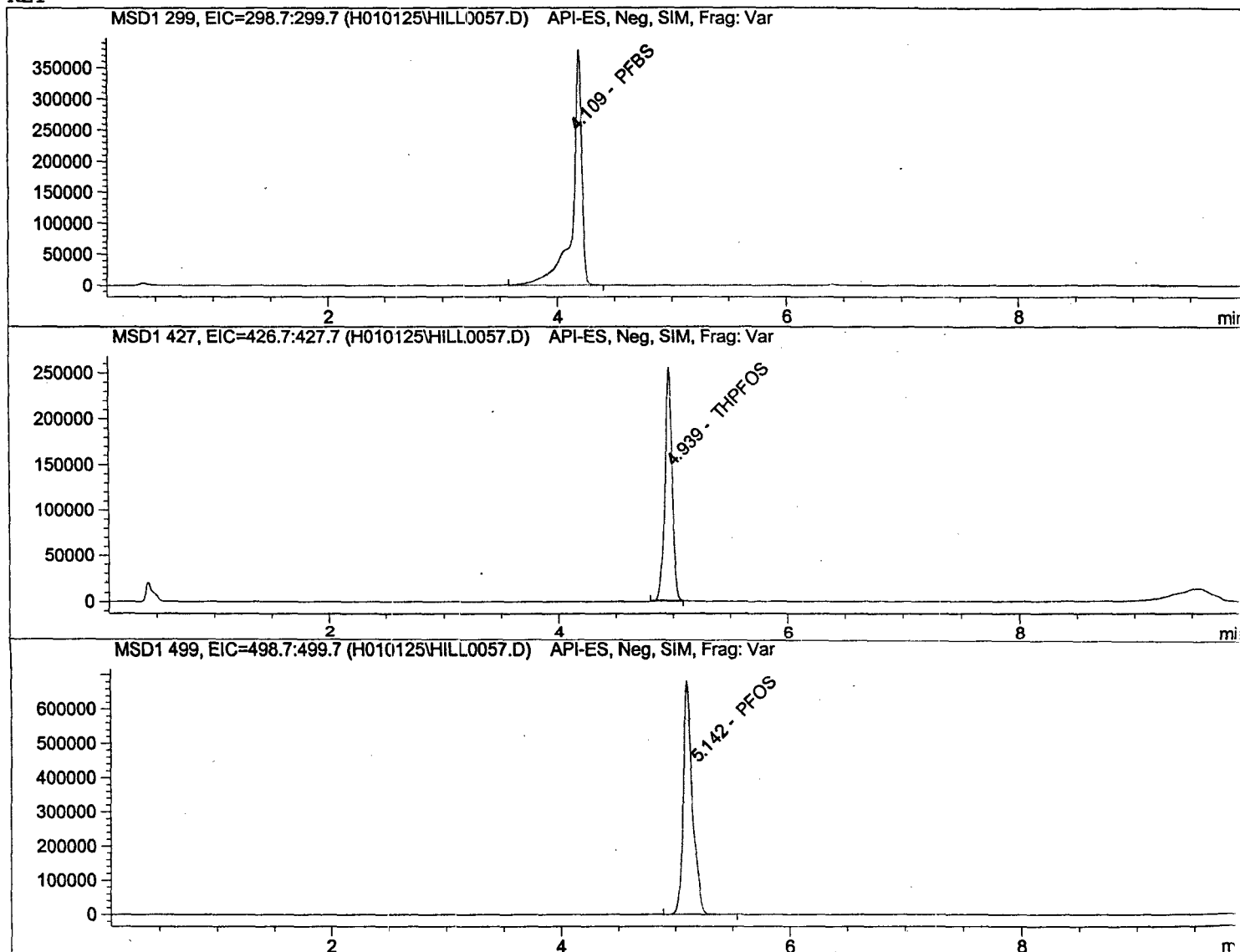
Injection Date	: 1/25/01 7:42:55 PM	Seq. Line	: 57
Sample Name	: swPFOS-1.3.1	Vial	: 39
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H0125A1.M		
Last changed	: 1/29/01 2:31:44 PM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

MLA
Initial03 Apr 01
per/10

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : Monday, January 29, 2001 2:15:31 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD #	ISTD Amount [ng/mL]	Name
--------	---------------------	------

1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.109	VBA+ I	2.17183e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.939	VBA+ I	1.12792e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.142	PVA+	3.79790e6	1.45346e-1	124.50482		PFOS

Totals without ISTD(s) : 124.50482

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-2.1.1, 100X Dil.

SFM 01-25-01 MEE (MLA)

=====

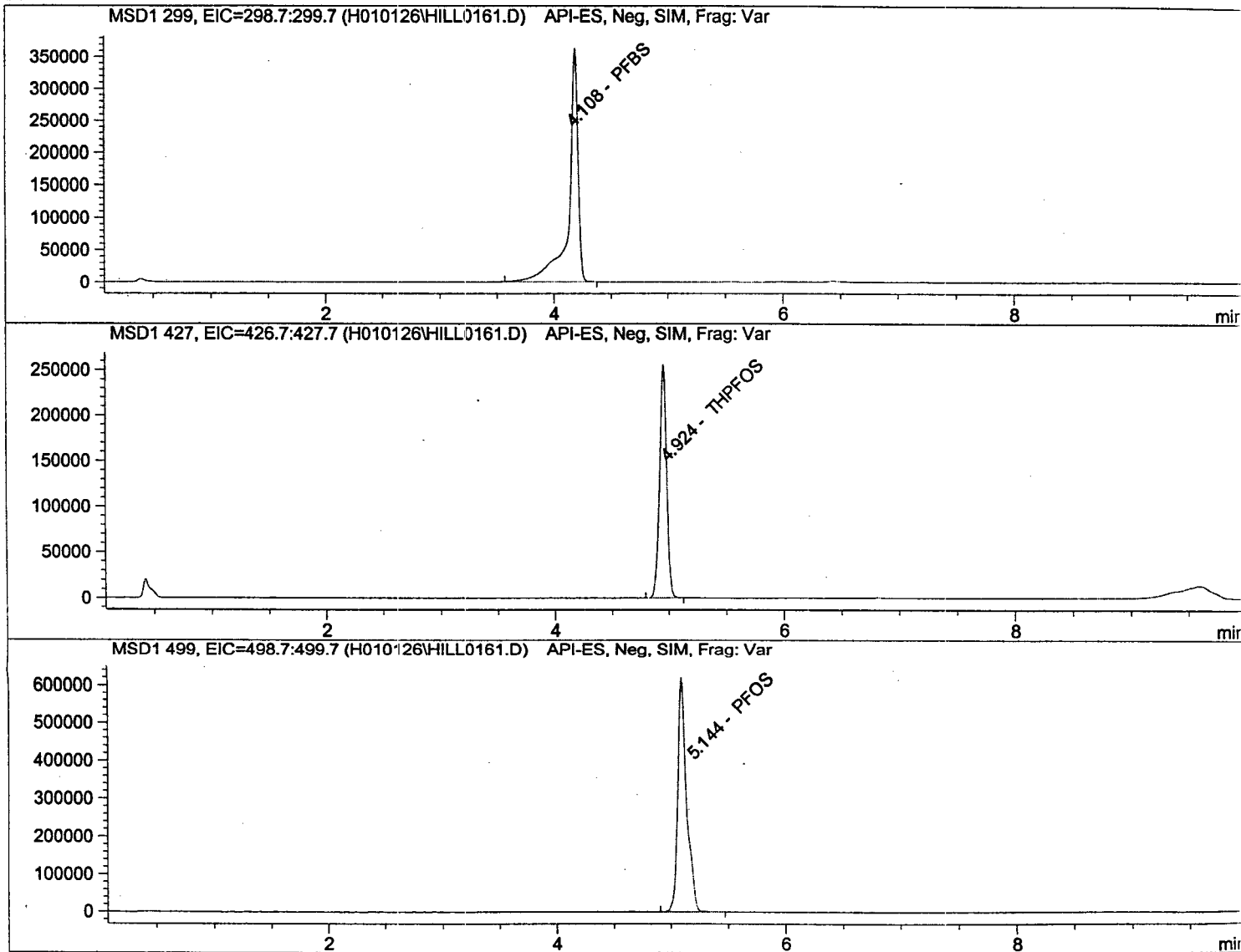
Injection Date	: 1/27/01 7:44:28 PM	Seq. Line	: 161
Sample Name	: swPFOS-2.1.1	Vial	: 63
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010126C.M		
Last changed	: 1/30/01 2:53:00 PM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mM Ammonium Acetate

Reprocessed on "Dioana"

KLT

**Exact Copy of Original**

MLA
Initial

03 Apr 01
post 1 of 2

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : Tuesday, January 30, 2001 2:27:34 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name

[ng/mL]

1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.108	VBA+ I	2.07348e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.924	PBA+ I	1.11620e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.144	VVA+	3.37958e6	1.58477e-1	122.06875		PFOS

Totals without ISTD(s) : 122.06875

1 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-2.1.4, 100X Dil.

SFM 01-25-01 MEE (MLA)

=====

Injection Date	: 1/27/01 8:17:52 PM	Seq. Line	: 164
Sample Name	: swPFOS-2.1.4	Vial	: 66
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010126C.M		
Last changed	: 1/30/01 2:53:16 PM by KLT/MLA		

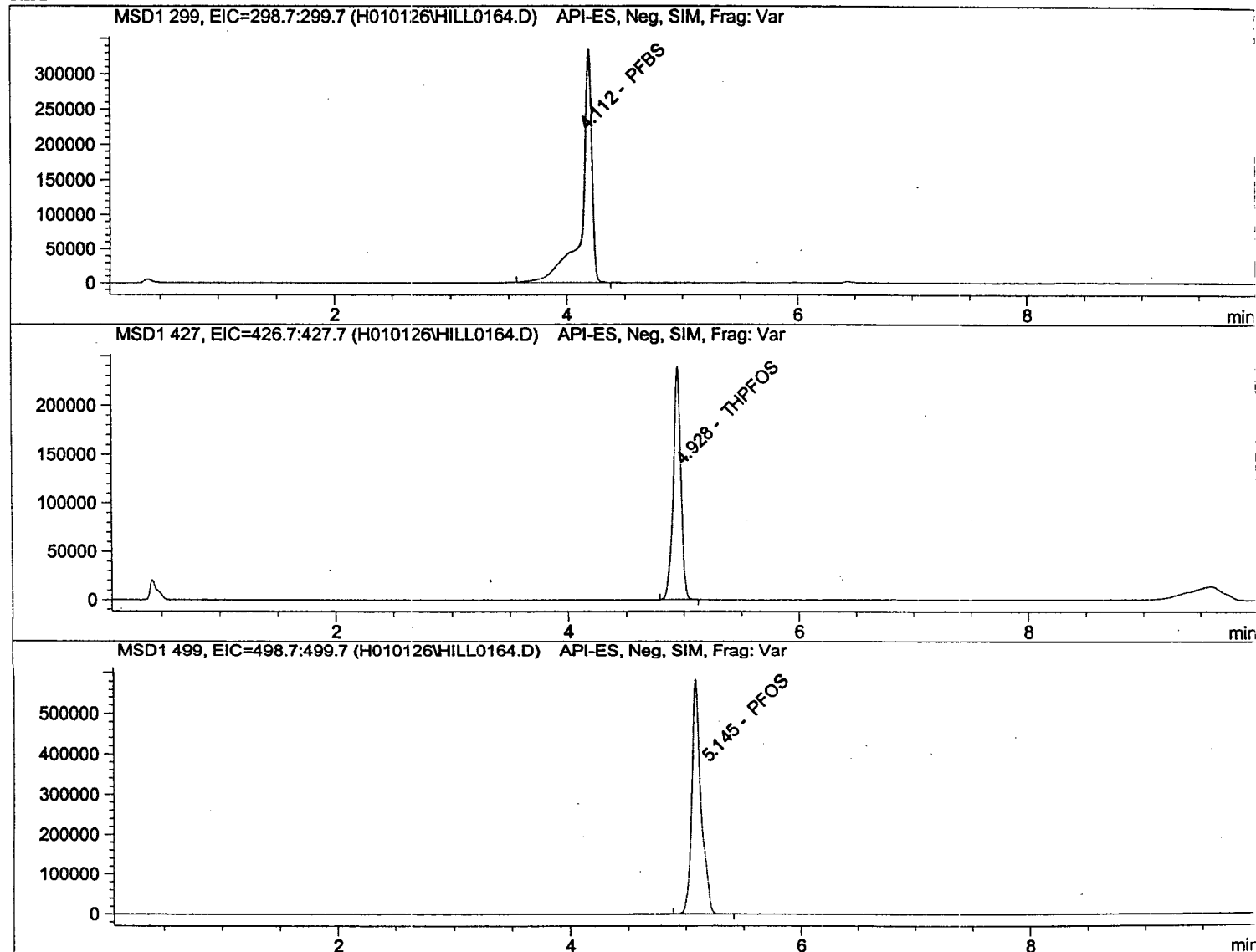
(modified after loading)

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

MLA
Initial03 April 2001
Ray Top

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : Tuesday, January 30, 2001 2:27:34 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name
[ng/mL]

-----|-----|-----
1 254.00000 PFBS
2 254.40000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.112	VBA+ I	2.03714e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.928	VBA+ I	1.10630e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.145	VVA+	3.33404e6	1.58453e-1	121.48330		PFOS

Totals without ISTD(s) : 121.48330

1 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

=====
*** End of Report ***

TCR-002, 012201-swPFOS-2.2.2, 100X Dil.

SFM 01-25-01 MEE (MLA)

=====

Injection Date	: 1/27/01 9:13:46 PM	Seq. Line	: 169
Sample Name	: swPFOS-2.2.2	Vial	: 68
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010125.M		
Last changed	: 1/25/01 9:13:04 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010126C.M		
Last changed	: 1/30/01 2:53:42 PM by KLT/MLA		

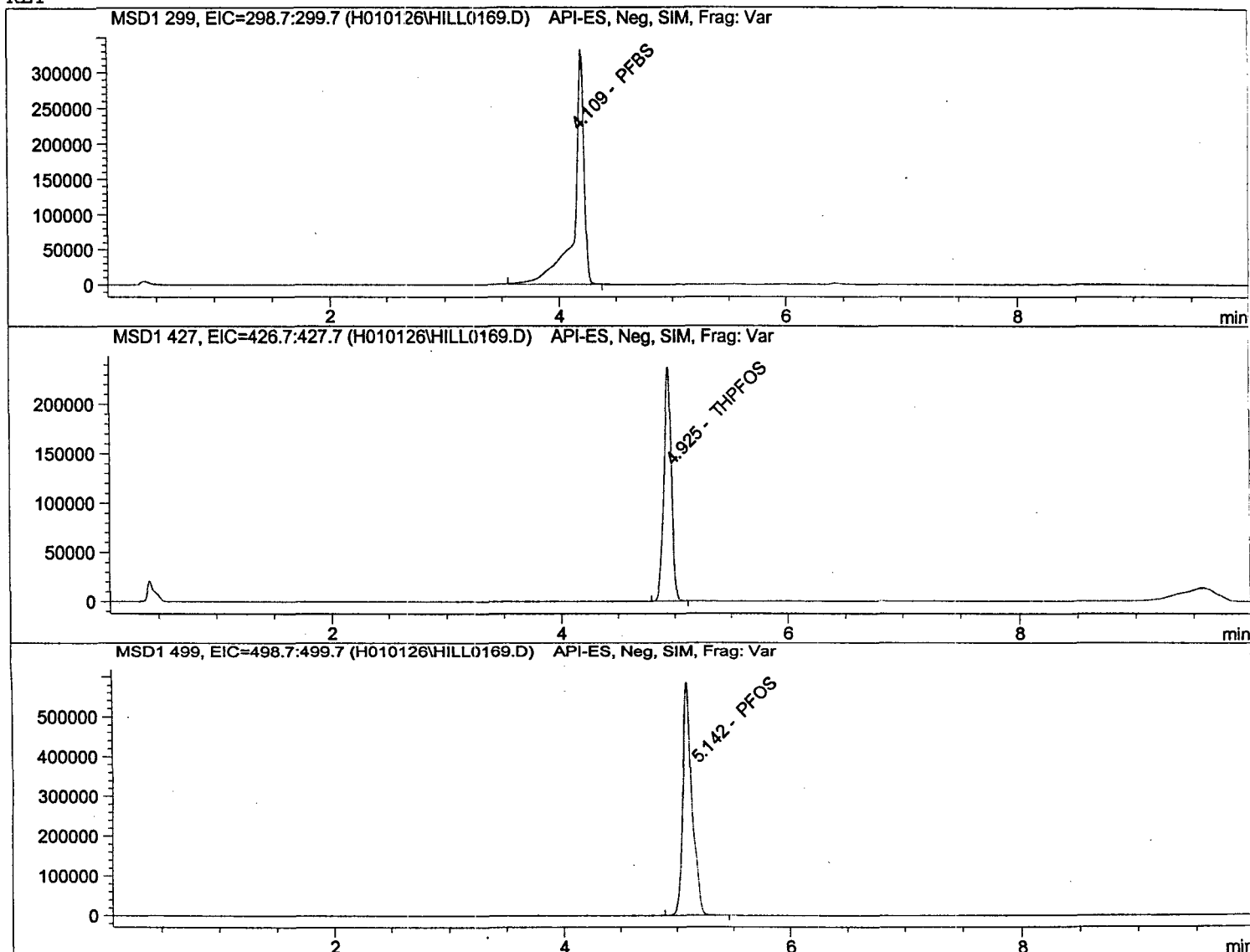
(modified after loading)

PFOS Analysis Column: C18 Betasil 5µm 50X2 mm

Eluents: Methanol and 2 mM Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

MLA
Initial

03 Apr 01
L. 10

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : Tuesday, January 30, 2001 2:27:34 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name

[ng/mL]

#	ISTD Amount [ng/mL]	Name
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.109	VBA+ I	2.01151e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.925	PBA+ I	1.10297e6	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.142	VBA+	3.38154e6	1.58541e-1	123.65462		PFOS

Totals without ISTD(s) : 123.65462

1 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-3.1.1, 100X Dil.

SFM 01-26-01 MEE (MLA)

=====

Injection Date	: 1/30/01 8:49:51 AM	Seq. Line	: 104
Sample Name	: swPFOS-3.1.1	Vial	: 61
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010129.M		
Last changed	: 1/29/01 1:35:12 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010129B.M		
Last changed	: 1/31/01 11:08:44 AM by KLT/MLA		

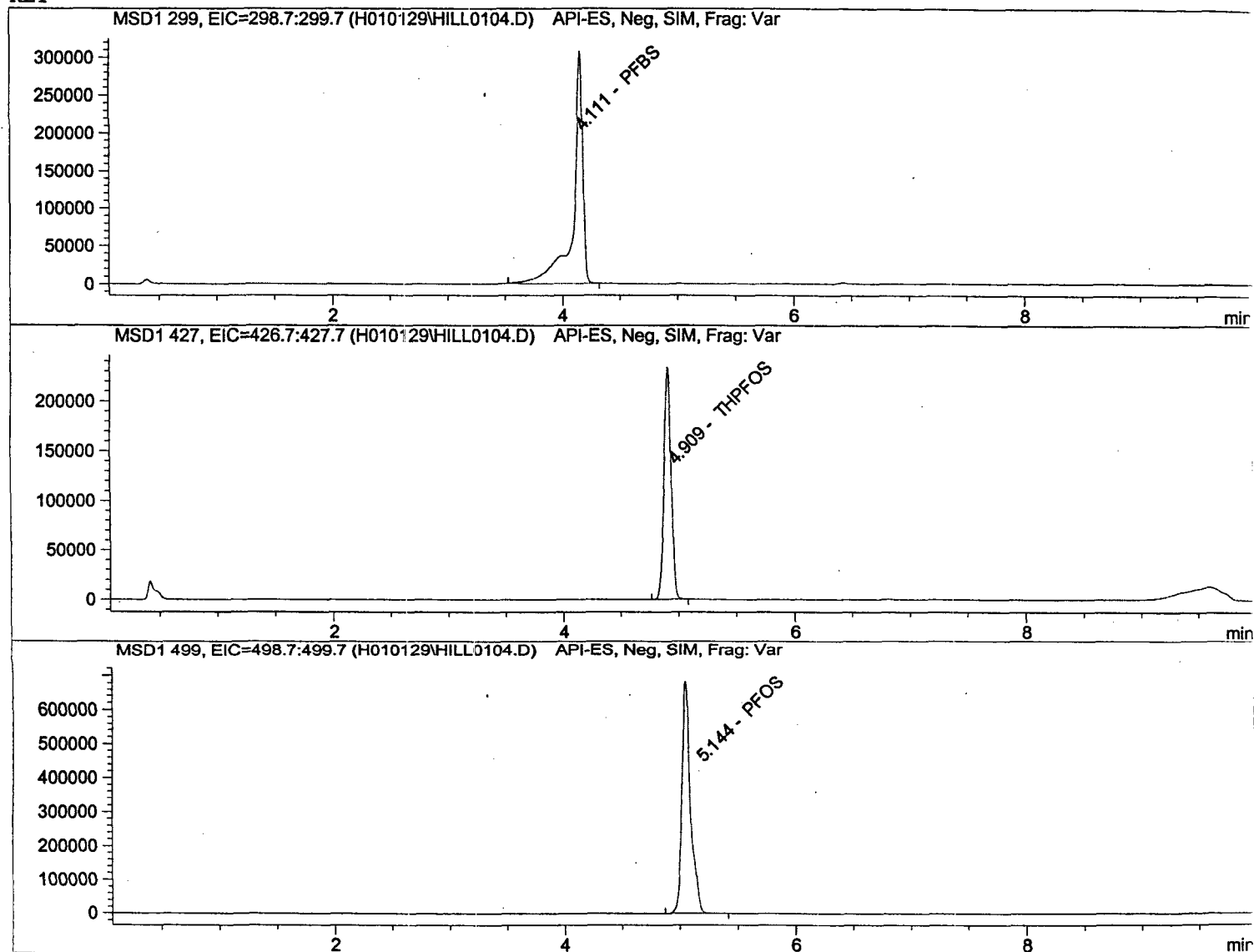
(modified after loading)

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

Initial

Date

1/31/01

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : 1/31/01 11:08:42 AM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name
[ng/mL]

-----|-----|-----
1 254.00000 PFBS
2 254.40000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.111	VBA+ I	1.79360e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.909	PBA+ I	9.89058e5	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.144	VVA+	3.60619e6	1.59134e-1	147.60707		PFOS

Totals without ISTD(s) : 147.60707

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-3.1.4, 100X Dil.

SFM 01-26-01 MEE (MLA)

=====

Injection Date	: 1/30/01 9:23:14 AM	Seq. Line	: 107
Sample Name	: swPFOS-3.1.4	Vial	: 64
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010129.M		
Last changed	: 1/29/01 1:35:12 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010129B.M		
Last changed	: 1/31/01 11:08:44 AM by KLT/MLA		

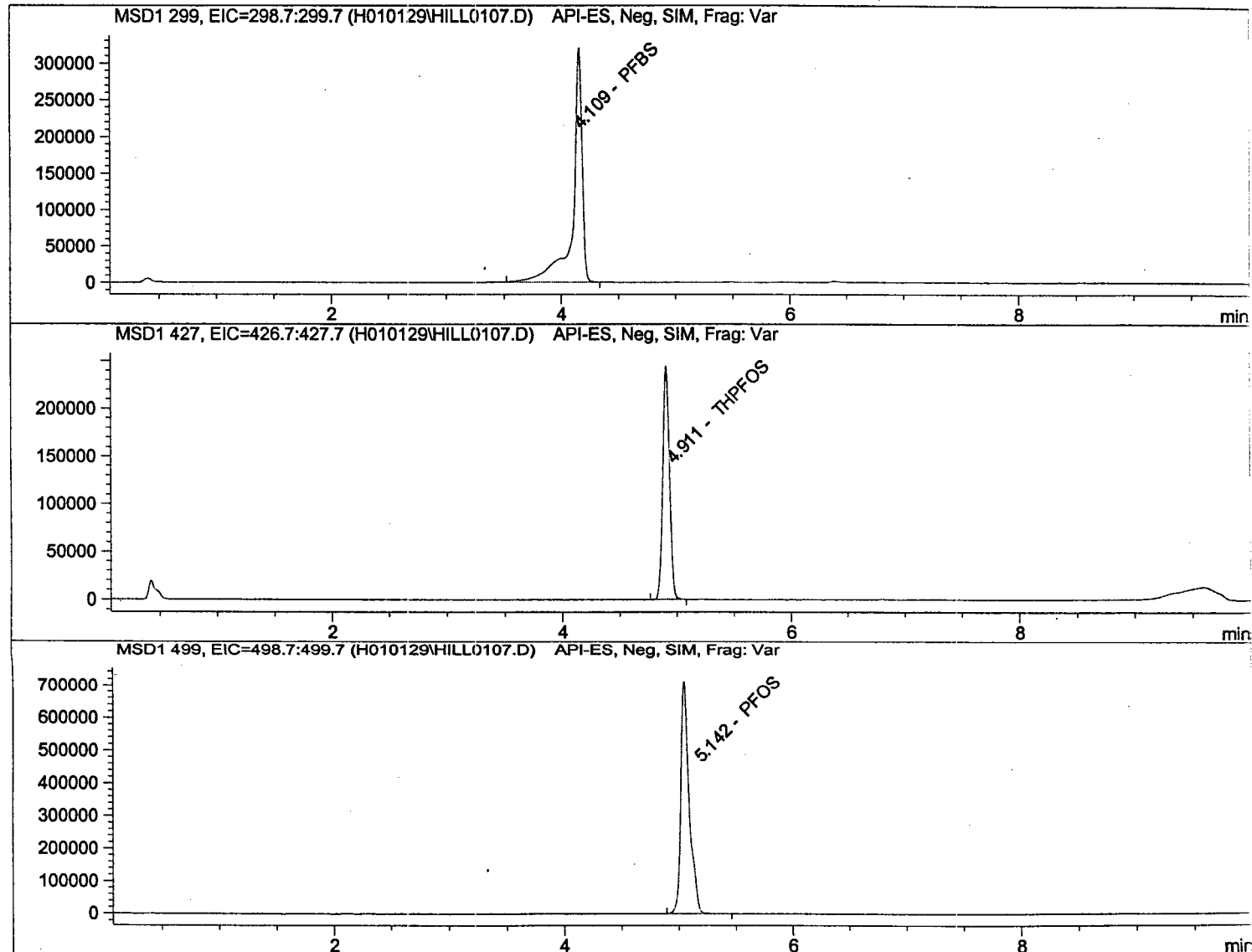
(modified after loading)

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

MLA
Initial03 Apr 01
Page 1 of 2

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : 1/31/01 11:08:42 AM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD #	ISTD Amount [ng/mL]	Name
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.109	VBA+ I	1.82894e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.911	PBA+ I	9.98892e5	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.142	VVA+	3.65623e6	1.59152e-1	148.19823		PFOS

Totals without ISTD(s) : 148.19823

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-3.2.1, 100X Dil.

SFM 01-26-01 MEE (MLA)

=====

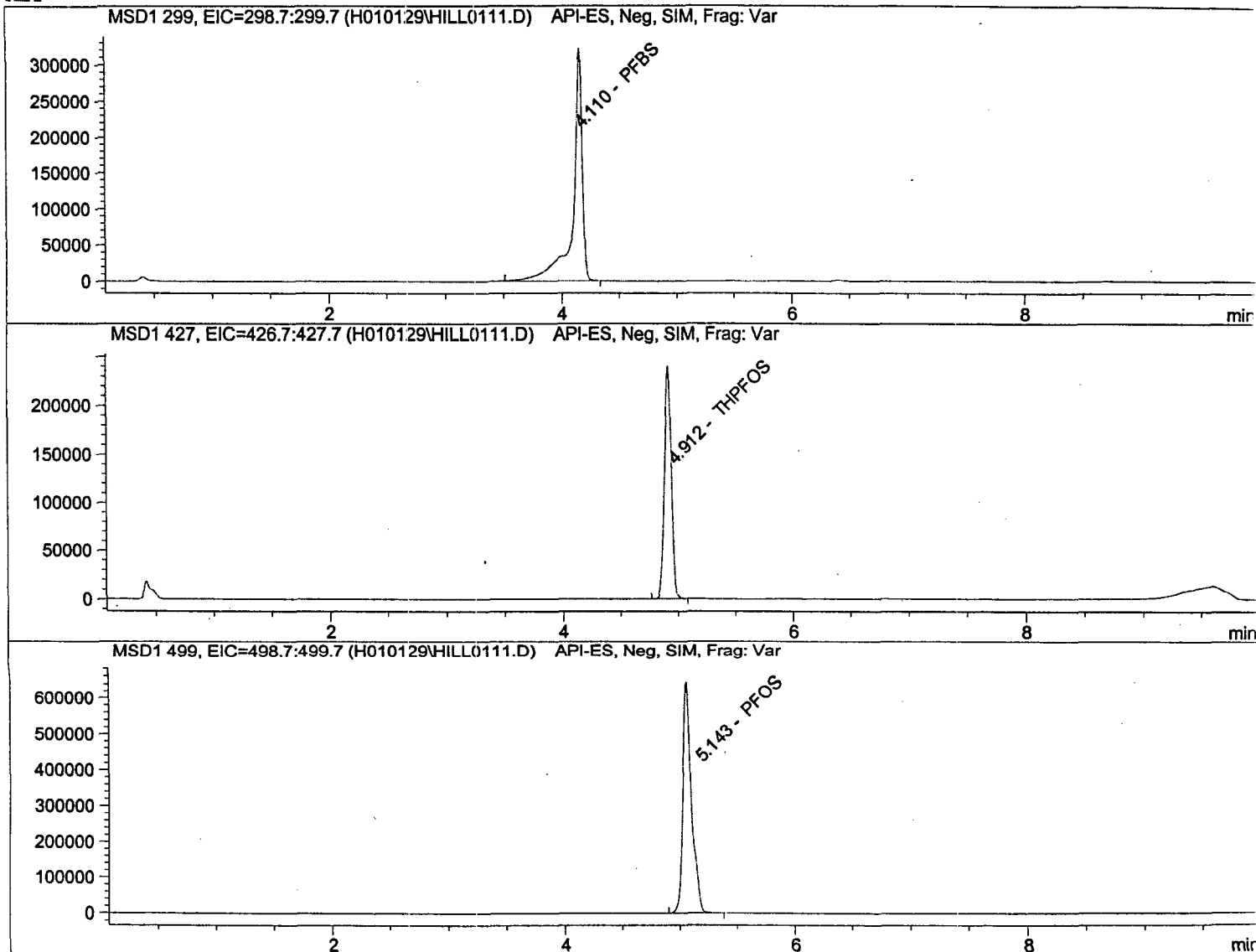
Injection Date	: 1/30/01 10:07:52 AM	Seq. Line	: 111
Sample Name	: swPFOS.3.2.1	Vial	: 65
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010129.M		
Last changed	: 1/29/01 1:35:12 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010129B.M		
Last changed	: 1/31/01 11:08:44 AM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil Sum 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



Exact Copy of Original

MLA
Initial

03 Apr 01
Date

Page 1 of 2

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : 1/31/01 11:08:42 AM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name

[ng/mL]

#	ISTD Amount [ng/mL]	Name
1	254.00000	PFBS
2	254.40000	THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.110	VBA+ I	1.82761e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.912	PBA+ I	9.96184e5	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.143	VVA+	3.37134e6	1.58803e-1	136.72210		PFOS

Totals without ISTD(s) : 136.72210

=====
*** End of Report ***
=====

TCR-002, 012201-swPFOS-3.3.1, 100X Dil.

SFM 01-26-01 MEE(MLA)

=====

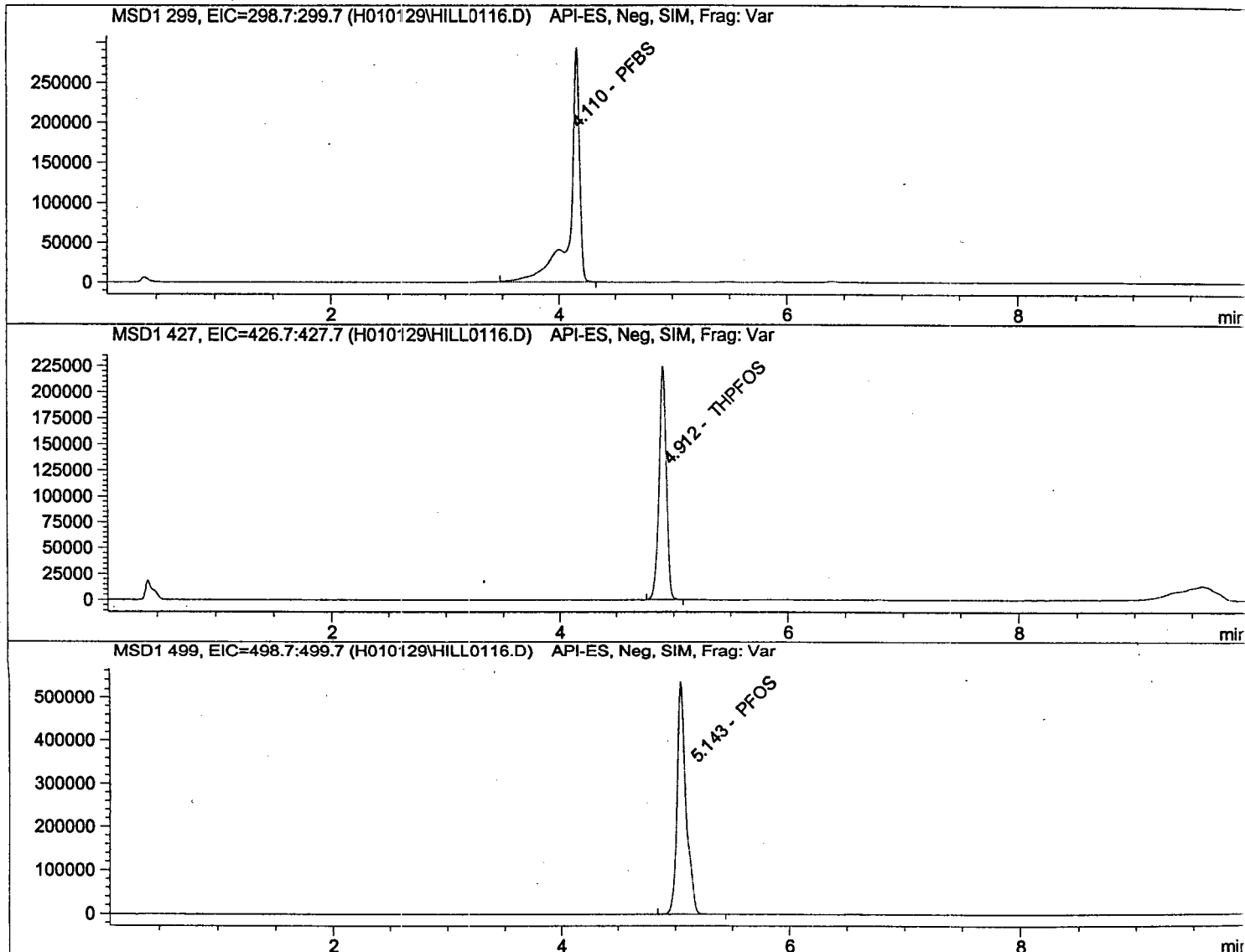
Injection Date	: 1/30/01 11:03:41 AM	Seq. Line	: 116
Sample Name	: swPFOS-3.3.1	Vial	: 69
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010129.M		
Last changed	: 1/29/01 1:35:12 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010129B.M		
Last changed	: 1/31/01 11:08:44 AM by KLT/MLA		
	(modified after loading)		

PFOS Analysis Column: C18 Betasil 5um 50X2 mm

Eluents: Methanol and 2 mm Ammonium Acetate

Reprocessed on "Dioana"

KLT



E-1 Copy of Original

MLA 03 Apr 01
Initial Date

page 1 of 1

=====
Internal Standard Report
=====

Sorted By : Signal
Calib. Data Modified : 1/31/01 11:08:42 AM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name

[ng/mL]

#	ISTD	Amount	Name
1	254.00000	PFBS	
2	254.40000	THPFOS	

Signal 1: MSD1 299, EIC=298.7:299.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.110	VBA+ I	1.76373e6	1.00000	254.00000		PFBS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
4.912	VBA+ I	9.87376e5	1.00000	254.40000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ng/mL]	Grp	Name
5.143	VVA+	2.90941e6	1.58231e-1	118.61261		PFOS

Totals without ISTD(s) : 118.61261

=====
*** End of Report ***
=====

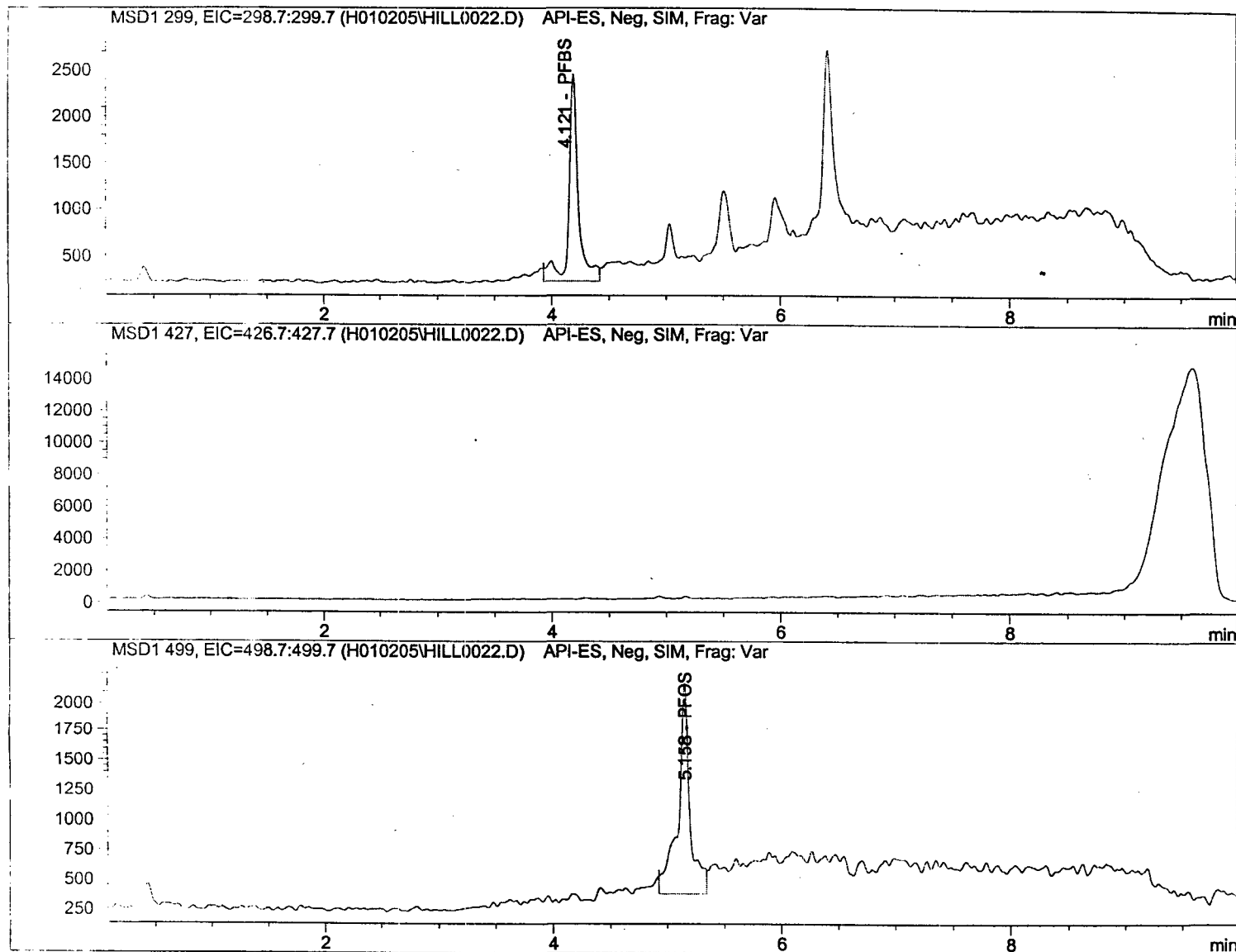
TN-A-4755, 05 Feb 01, exp:2005.

=====

Injection Date	: 2/5/01 6:07:45 PM	Seq. Line	: 22
Sample Name	: MeOH Blank	Vial	: 92
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010205.M		
Last changed	: 2/5/01 2:09:33 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010205A.M		
Last changed	: 2/9/01 3:37:04 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



=====

Internal Standard Report

=====

Sorted By	:	Retention Time
Calib. Data Modified	:	Friday, February 09, 2001 3:22:24 PM
Multiplier	:	1.0000
Dilution	:	1.0000
Sample ISTD Information:		
ISTD	ISTD Amount	Name
#	[ppb]	

Exact Copy of O. J. Hill

Initial

Date

TM-A-4755, 05 Feb 01, exp:2005.

```
=====
Injection Date   : 2/5/01 6:07:45 PM           Seq. Line :   22
Sample Name      : MeOH Blank                   Vial       :   92
Acq. Operator    : MLA                         Inj        :    1
Acq. Instrument  : Hillary                     Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:37:04 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluoronated carboxylic acids

```
=====
1      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.121	1	VVA+	1.29920e4	0.00000	#####		PFBS
4.821	2		-	-	-		THPFOS
5.158	3	VVA+	1.31270e4	0.00000	0.00000		PFOS

Totals without ISTD(s) : 2.53242e-12

2 Warnings or Errors :

Warning : ISTD compound(s) not found

Warning : Negative results set to zero (cal. curve intercept), (PFOS)

```
=====
*** End of Report ***
=====
```

TMA-4755, 05 Feb 01, exp:2005.

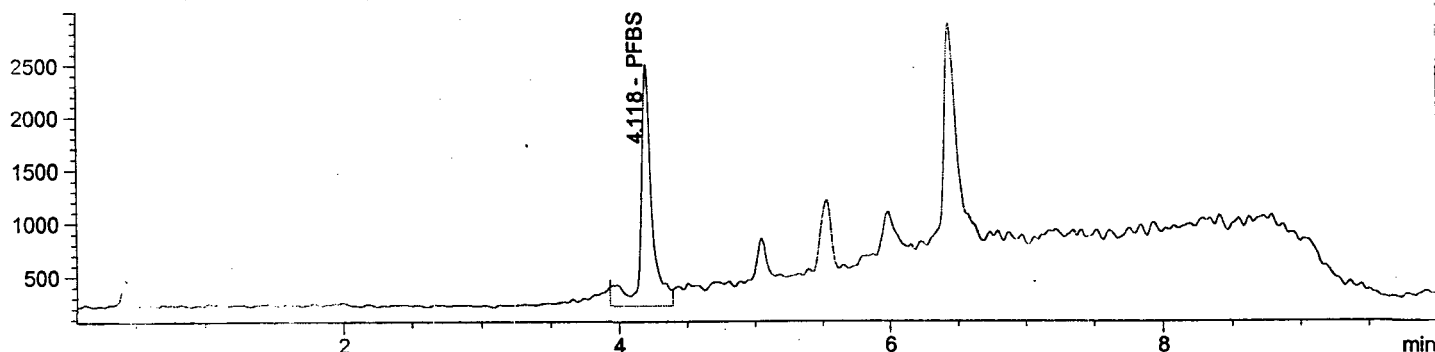
=====

Injection Date	: 2/5/01 11:19:11 PM	Seq. Line	: 50
Sample Name	: MeOH Blank	Vial	: 93
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010205.M		
Last changed	: 2/5/01 2:09:33 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010205A.M		
Last changed	: 2/9/01 3:39:25 PM by MLA		

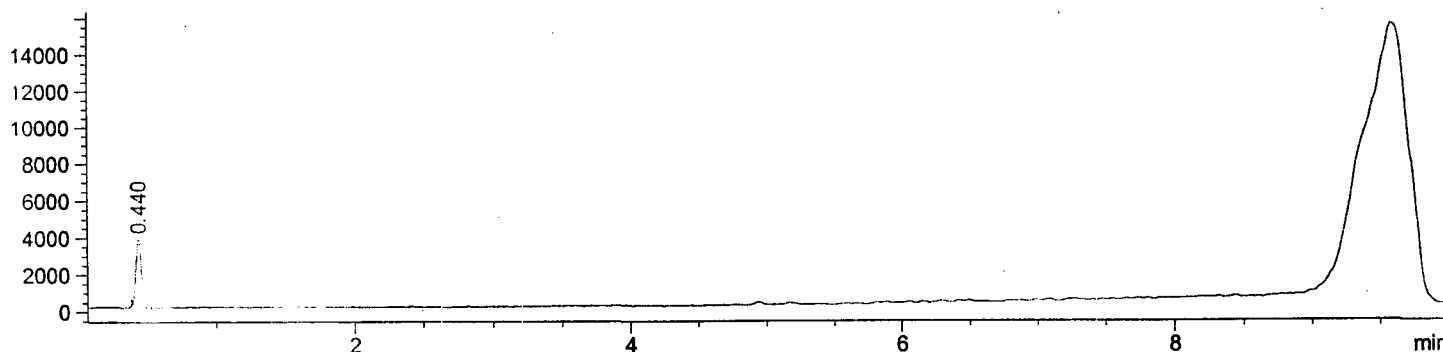
(modified after loading)

Sim run C2, c3, c4, c5, c7 and C8 perfluorinated carboxylic acids

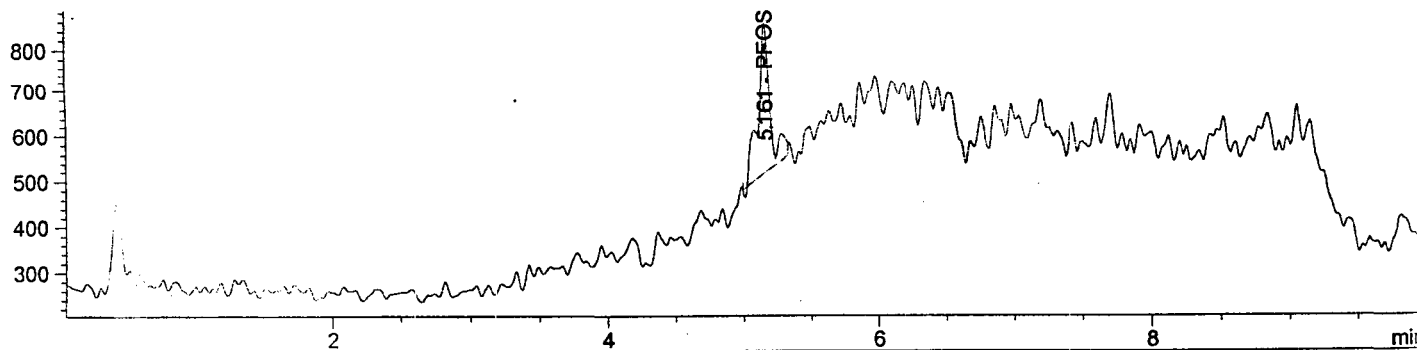
MSD1 299, EIC=298.7:299.7 (H010205\HILL0050.D) API-ES, Neg, SIM, Frag: Var



MSD1 427, EIC=426.7:427.7 (H010205\HILL0050.D) API-ES, Neg, SIM, Frag: Var



MSD1 499, EIC=498.7:499.7 (H010205\HILL0050.D) API-ES, Neg, SIM, Frag: Var



Internal Standard Report

Exact Copy of Original

Sorted By : Retention Time
Calib. Data Modified : Friday, February 09, 2001 3:22:24 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD ISTD Amount Name
[ppb]

Initial *MLA*
per/ly

TH-A-4755, 05 Feb 01, exp:2005.

```
=====
Injection Date   : 2/5/01 11:19:11 PM           Seq. Line :   50
Sample Name      : MeOH Blank                     Vial       :   93
Acq. Operator    : MLA                           Inj        :    1
Acq. Instrument  : Hillary                       Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:39:25 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

```
=====
1      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.118	1	VVA+	1.30096e4	0.00000	#####		PFBS
4.821	2		-	-	-		THPFOS
5.161	3	PBA+	2060.08472	0.00000	0.00000		PFOS

Totals without ISTD(s) : 2.53242e-12

2 Warnings or Errors :

Warning : ISTD compound(s) not found

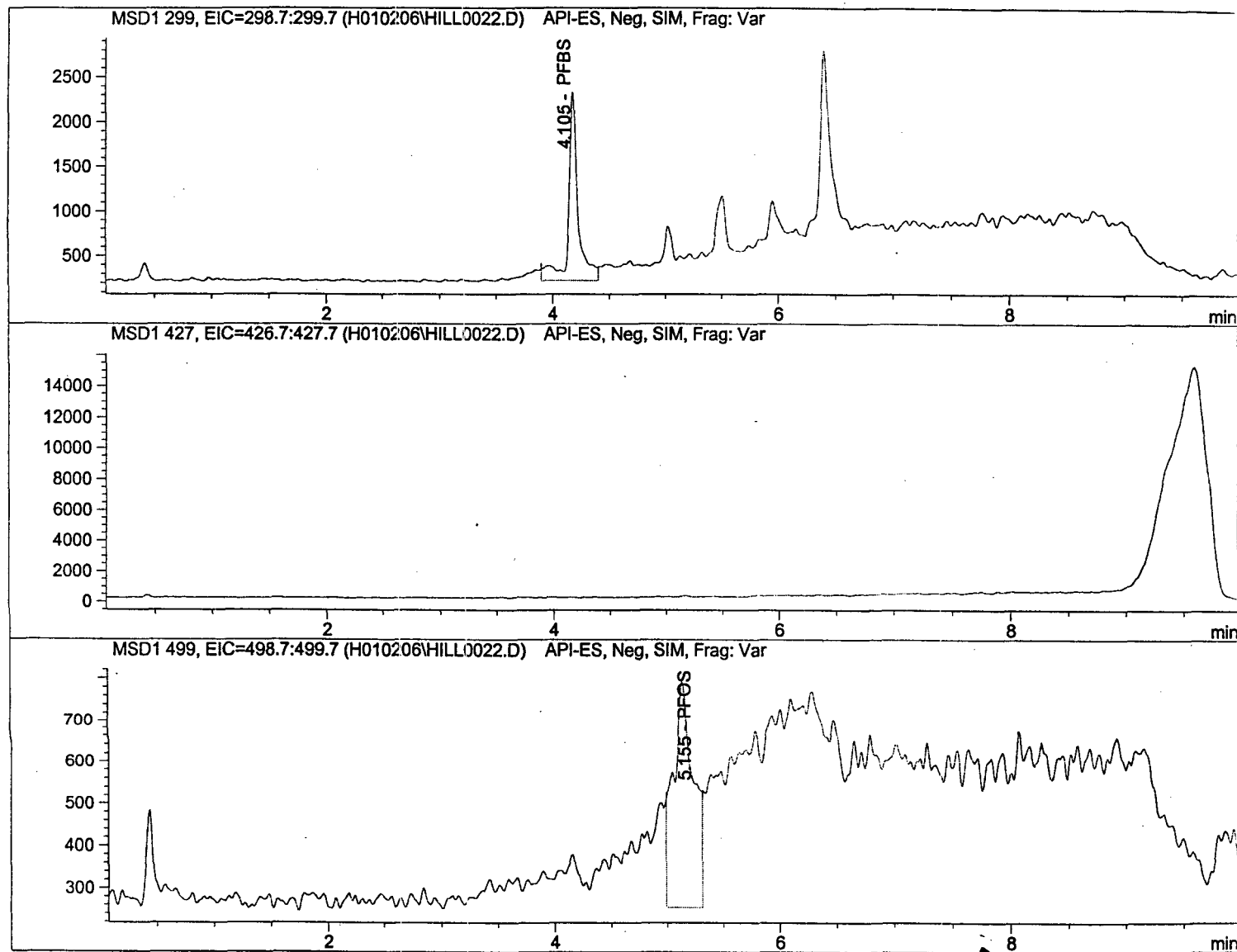
Warning : Negative results set to zero (cal. curve intercept), (PFOS)

```
=====
*** End of Report ***
=====
```

TN-A-4755, 05 Feb 01, exp:2005.

```
=====
Injection Date   : 2/6/01 3:46:36 PM           Seq. Line :   22
Sample Name      : MeOH Blank                   Vial       :   96
Acq. Operator    : MLA                         Inj        :    1
Acq. Instrument  : Hillary                     Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:14:17 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



Internal Standard Report

```
=====
Sorted By      : Retention Time
Calib. Data Modified : Wednesday, February 07, 2001 12:53:59 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name
# [ppb]
-----|-----|-----
```

Exact Copy of Original
MLA
Initial

02/07/01
MLA
rev 1/12

TN-A-4755, 05 Feb 01, exp:2005.

```
=====
Injection Date   : 2/6/01 3:46:36 PM           Seq. Line :   22
Sample Name      : MeOH Blank                   Vial       :   96
Acq. Operator    : MLA                         Inj        :    1
Acq. Instrument  : Hillary                     Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:14:17 PM by MLA
                  (modified after loading)
Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids
=====
```

1 250.70000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.105	1	VVA+	1.24766e4	0.00000	#####		PFBS
4.819	2		-	-	-		THPFOS
5.155	3	VVA+	6796.12695	0.00000	1.42633e-1		PFOS

Totals without ISTD(s) : 1.42633e-1

1 Warnings or Errors :

Warning : ISTD compound(s) not found

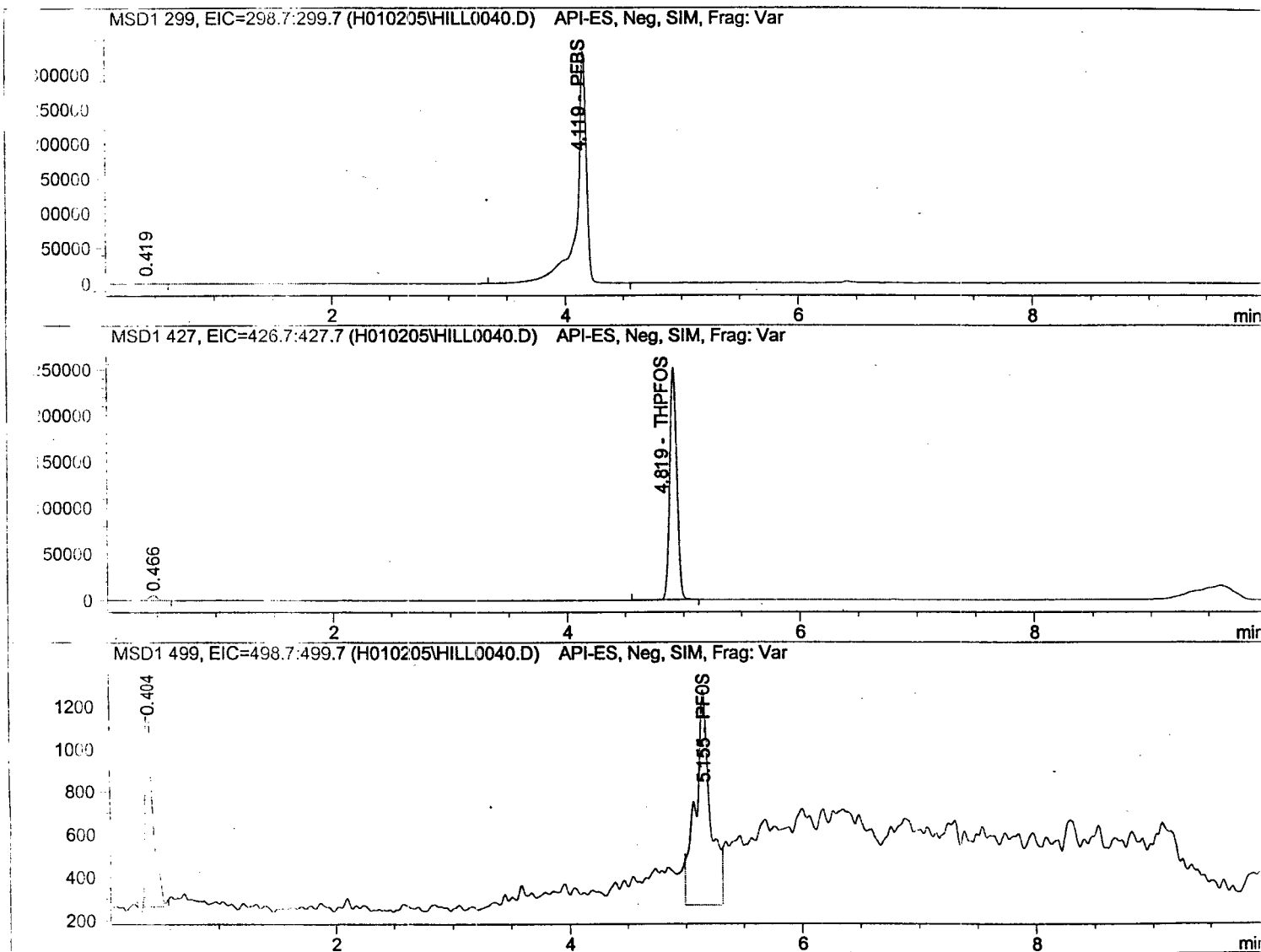
```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-2.4.1, 100X Dil.

Method Blank, SFM 02-01-01 MEE(MLA)

```
=====
Injection Date   : 2/5/01 9:27:53 PM           Seq. Line :   40
Sample Name      : naclPFOS-2.4.1             Vial       :   21
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:38:35 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



Internal Standard Report

```
=====
Sorted By           :      Retention Time
Calib. Data Modified :      Friday, February 09, 2001 3:22:24 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Sample ISTD Information:
ISTD ISTD Amount   Name
# [ppb]
=====
```

Exact Copy of Original

03 Apr 02
page 1 of 2

MLA
Initial

TMR-002, 012901-naclPFOS-2.4.1, 100X Dil.

Method Blank, SFM 02-01-01 MEE(MLA)

```
=====
Injection Date   : 2/5/01 9:27:53 PM           Seq. Line :   40
Sample Name      : naclPFOS-2.4.1              Vial       :   21
Anal. Operator   : MLA                        Inj        :    1
Anal. Instrument : Hillary                   Inj Volume  : 10 µl
Anal. Method     : C:\HPCHEM\1\METHODS\H010205.M
Last changed    : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed    : 2/9/01 3:38:35 PM by MLA
                  (modified after loading)
=====
```

Scan ran C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

```
=====
1      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
1.119	1	VVA+	1.94355e6	5.81750e-1	271.89463		PFBS
1.819	2	PVA+ I	1.04252e6	1.00000	250.70000		THPFOS
5.155	3	VVA+	8906.63379	0.00000	0.00000		PFOS

Totals without ISTD(s) : 271.89463

1 Warnings or Errors :

Warning : Negative results set to zero (cal. curve intercept), (PFOS)

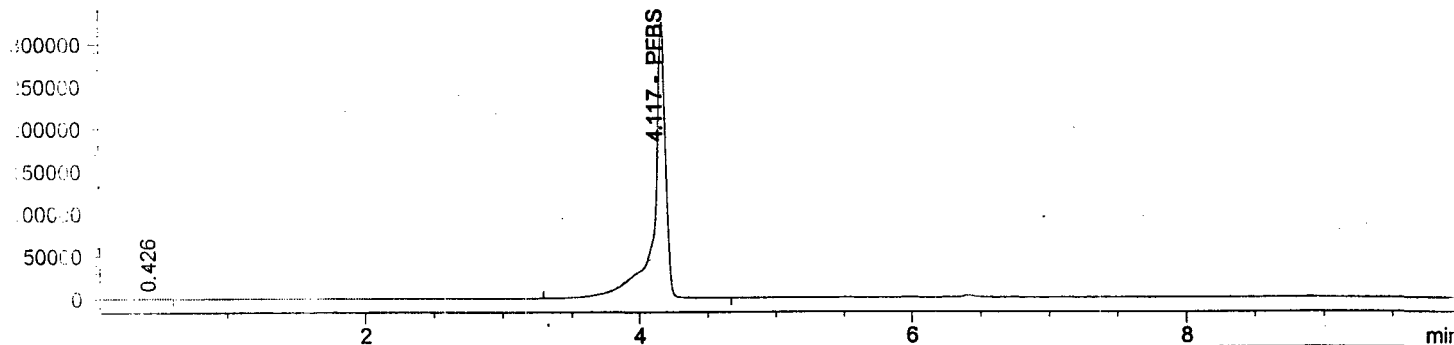
```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-2.4.3, 100X Dil.

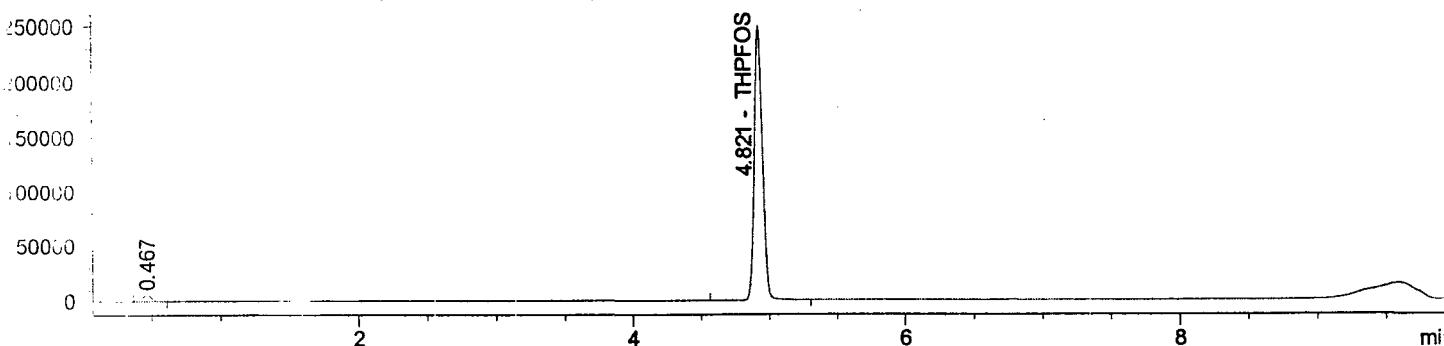
Method Blank, SFM 02-01-01 MEE (MLA)

```
=====
Injection Date   : 2/5/01 9:50:09 PM           Seq. Line :   42
Sample Name      : naclPFOS-2.4.3              Vial       :   23
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:38:45 PM by MLA
                  (modified after loading)
Sample run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids
=====
```

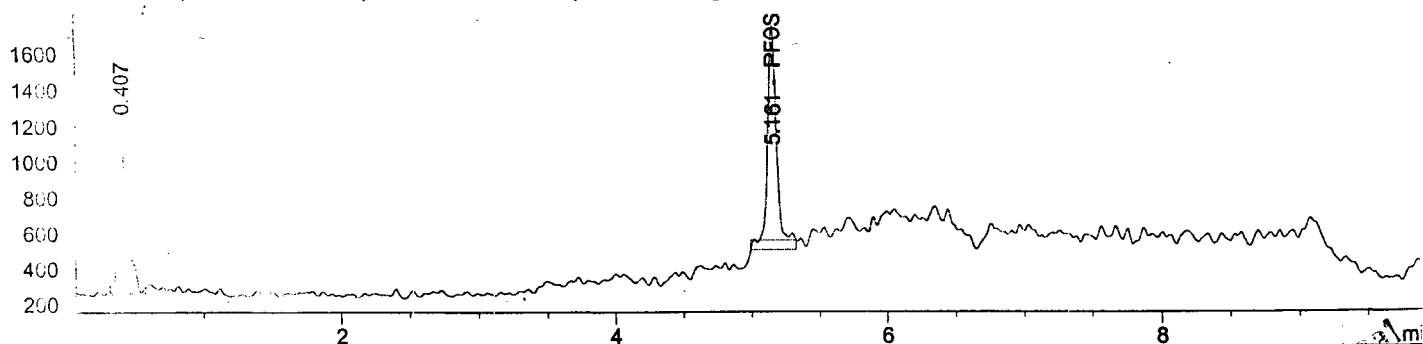
MSD1 299, EIC=298.7:299.7 (H010205\HILL0042.D) API-ES, Neg, SIM, Frag: Var



MSD1 427, EIC=426.7:427.7 (H010205\HILL0042.D) API-ES, Neg, SIM, Frag: Var



MSD1 499, EIC=498.7:499.7 (H010205\HILL0042.D) API-ES, Neg, SIM, Frag: Var



Internal Standard Report

```
=====
Started By      : Retention Time
Calib. Data Modified : Friday, February 09, 2001 3:22:24 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name
# [ppb]
=====
```

Exact Copy of Original
MLA
Initial
Date
2/9/01

TMR-002, 012901-naclPFOS-2.4.3, 100X Dil.

Method Blank, SFM 02-01-01 MEE (MLA)

```
=====
Injection Date   : 2/5/01 9:50:09 PM           Seq. Line :   42
Sample Name      : naclPFOS-2.4.3              Vial       :   23
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:38:45 PM by MLA
                  (modified after loading)
=====
```

Scan run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

1 250.70000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
1.117	1	VVA+	1.93318e6	5.81750e-1	272.07681		PFBS
1.821	2	PBA+ I	1.03627e6	1.00000	250.70000		THPFOS
5.151	3	PPA+	5505.63037	0.00000	0.00000		PFOS

Totals without ISTD(s) : 272.07681

1 Warnings or Errors :

Warning : Negative results set to zero (cal. curve intercept), (PFOS)

```
=====
*** End of Report ***
=====
```

TCP-002, 012901-naclPFOS-3.4.2, 100X Dil.

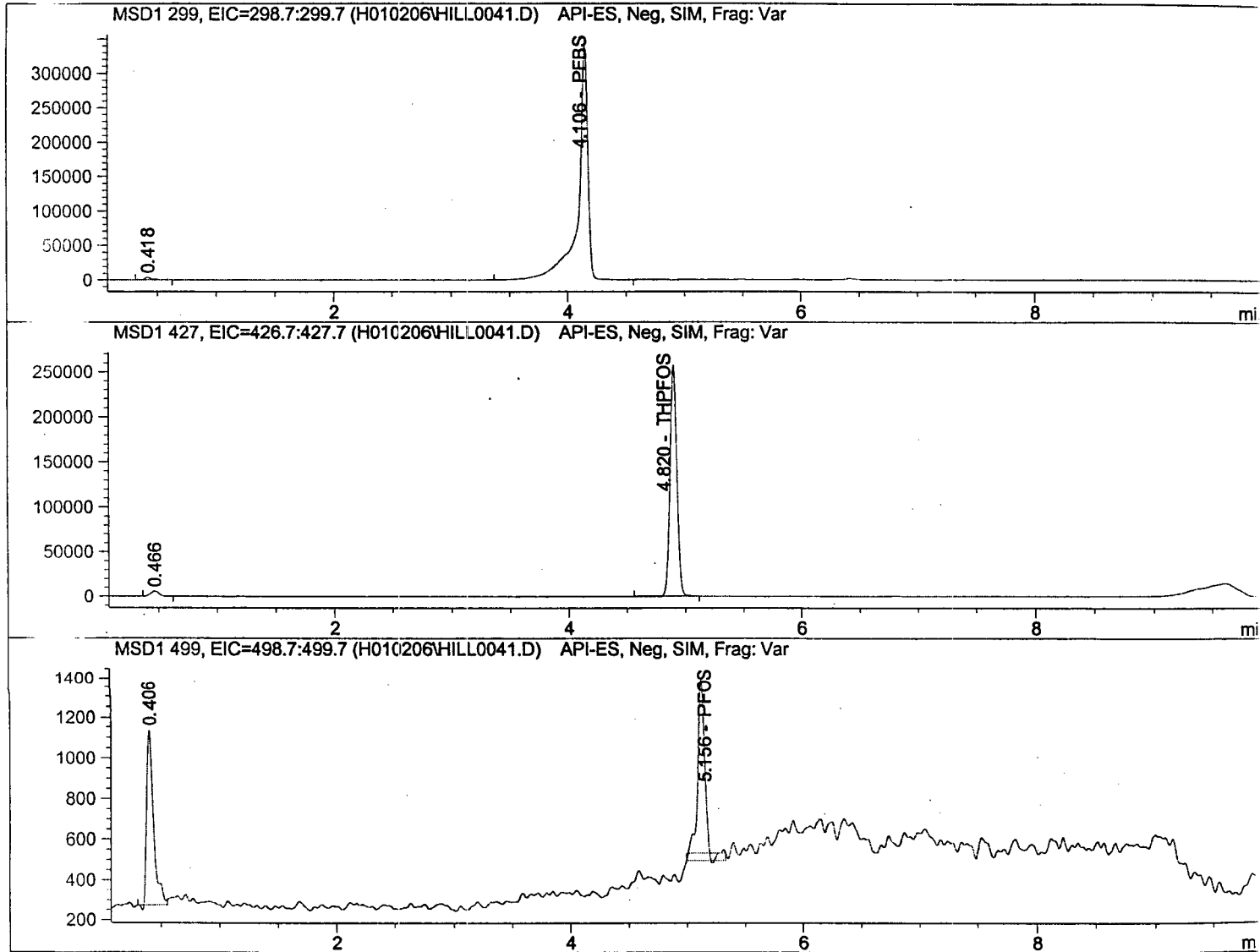
Method Blank, SFM 02-02-01 MEE (MLA)

=====

Injection Date	: 2/6/01 7:17:53 PM	Seq. Line	: 41
Sample Name	: naclPFOS-3.4.2	Vial	: 52
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010206.M		
Last changed	: 2/6/01 11:47:54 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010206A.M		
Last changed	: 2/7/01 1:15:46 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



=====

Internal Standard Report

=====

Sorted By	:	Retention Time
Calib. Data Modified	:	Wednesday, February 07, 2001 12:53:59 PM
Multiplier	:	1.0000
Dilution	:	1.0000

Sample ISTD Information:

ISTD #	ISTD Amount	Name
	[ppb]	

-----|-----|-----

Exact Copy of Original

MLA Initial *02/07/01* Date *page 1 of 2*

TCR-002, 012901-naclPFOS-3.4.2, 100X Dil.

Method Blank, SFM 02-02-01 MEE(MLA)

```
=====
Injection Date   : 2/6/01 7:17:53 PM           Seq. Line :   41
Sample Name      : naclPFOS-3.4.2              Vial       :   52
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:15:46 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

```
=====
1      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.106	1	VVA+	2.00919e6	6.10461e-1	290.41323		PFBS
4.820	2	PVA+ I	1.05881e6	1.00000	250.70000		THPFOS
5.156	3	PPA+	3730.26538	3.15106e-1	2.78313e-1		PFOS

Totals without ISTD(s) : 290.69154

```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-3.4.4, 100X Dil.

Method Blank, SFM 02-02-01 MEE (MLA)

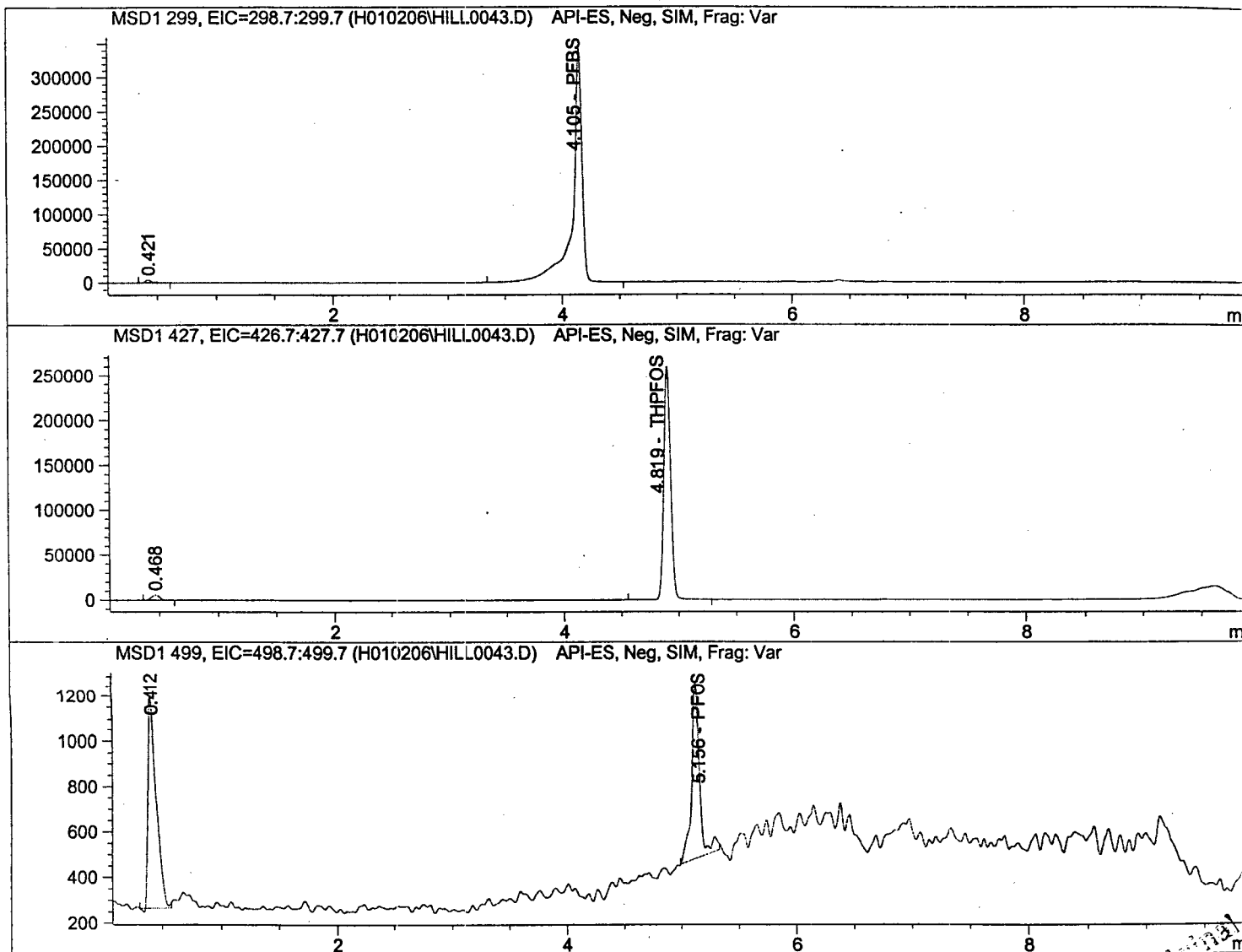
=====

Injection Date	: 2/6/01 7:40:11 PM	Seq. Line	: 43
Sample Name	: naclPFOS-3.4.4	Vial	: 54
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010206.M		
Last changed	: 2/6/01 11:47:54 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010206A.M		
Last changed	: 2/7/01 1:15:55 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

=====



=====

Internal Standard Report

=====

Sorted By	:	Retention Time
Calib. Data Modified	:	Wednesday, February 07, 2001 12:53:59 PM
Multiplier	:	1.0000
Dilution	:	1.0000

Sample ISTD Information:

ISTD	ISTD Amount	Name
#	[ppb]	

-----|-----|-----

Exact Copy of Original

MLA Initial

03/07/01

10/1/01

TCR-002, 012901-naclPFOS-3.4.4, 100X Dil.

Method Blank, SFM 02-02-01 MEE (MLA)

```
=====
Injection Date   : 2/6/01 7:40:11 PM           Seq. Line :   43
Sample Name      : naclPFOS-3.4.4              Vial       :   54
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:15:55 PM by MLA
                  (modified after loading)
Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids
=====
```

1 250.70000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.105	1	VVA+	1.97953e6	6.10461e-1	288.63083		PFBS
4.819	2	PBA+ I	1.04962e6	1.00000	250.70000		THPFOS
5.156	3	PBA+	3744.80615	3.13083e-1	2.80034e-1		PFOS

Totals without ISTD(s) : 288.91086

```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-2.1.1, 100X Dil.

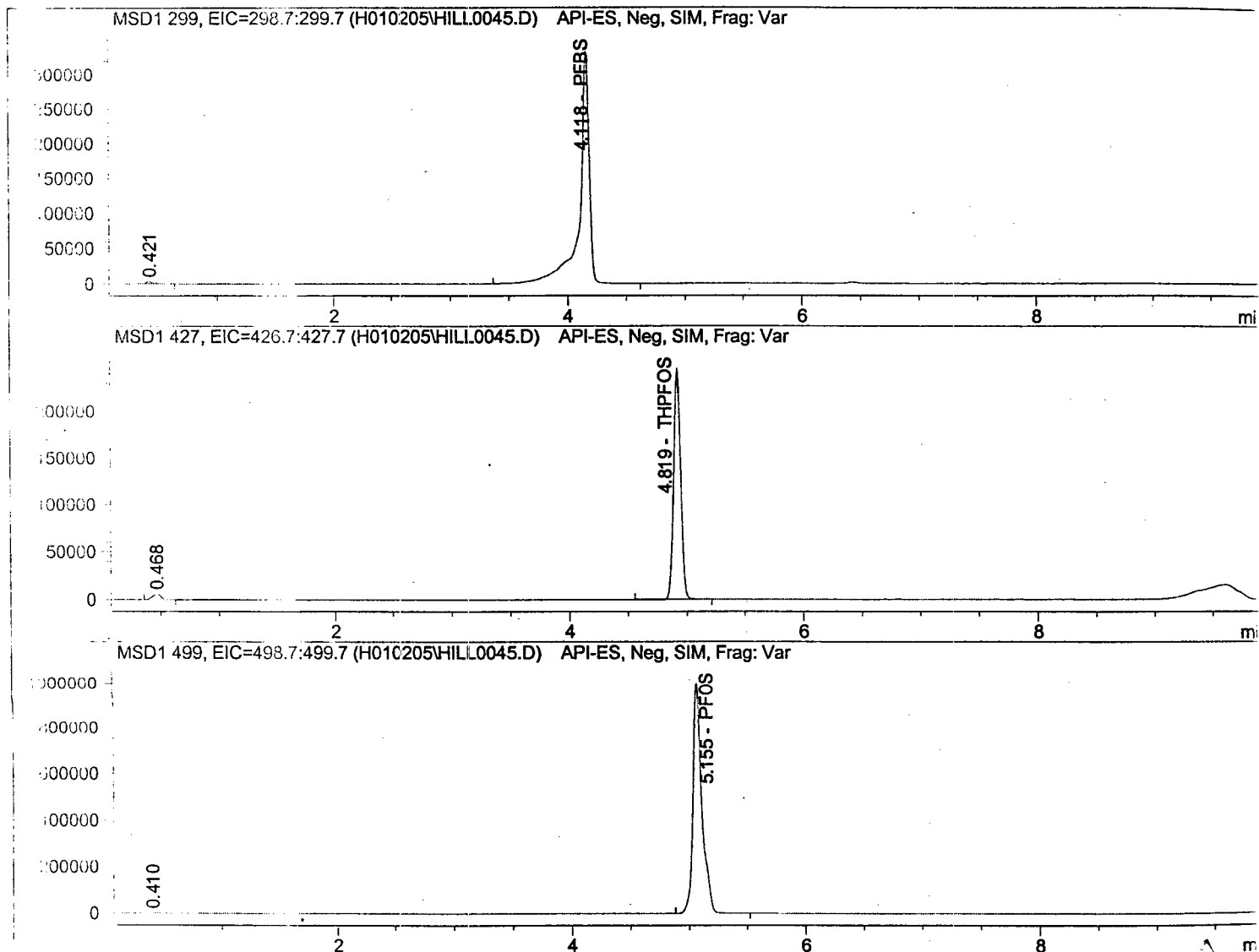
SEM 02-01-01 MEE (MLA)

=====

Injection Date	: 2/5/01 10:23:33 PM	Seq. Line	: 45
Sample Name	: naclPFOS-2.1.1	Vial	: 31
Anal. Operator	: MLA	Inj	: 1
Anal. Instrument	: Hillary	Inj Volume	: 10 µl
Anal. Method	: C:\HPCHEM\1\METHODS\H010205.M		
Last changed	: 2/5/01 2:09:33 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010205A.M		
Last changed	: 2/9/01 3:39:00 PM by MLA		
	(modified after loading)		

=====

Sem run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



=====

Internal Standard Report

=====

Sorted By	:	Retention Time
Calib. Data Modified	:	Friday, February 09, 2001 3:22:24 PM
Multiplier	:	1.0000
Dilution	:	1.0000

Sample ISTD Information:

ISTD	ISTD Amount	Name
#	[ppb]	

TCR-002, 012901-naclPFOS-2.1.1, 100X Dil.

SMA 02-01-01 MEE (MLA)

=====

Injection Date	: 2/5/01 10:23:33 PM	Seq. Line	: 45
Sample Name	: naclPFOS-2.1.1	Vial	: 31
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010205.M		
Last changed	: 2/5/01 2:09:33 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010205A.M		
Last changed	: 2/9/01 3:39:00 PM by MLA		
	(modified after loading)		

Sum Run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

=====

250.70000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime	Sig	Type	Area	Amt/Area	Amount	Grp	Name
[min]				ratio	[ppb]		
4.118	1	VVA+	1.95449e6	5.81750e-1	280.00194		PFBS
4.819	2	PBA+ I	1.01804e6	1.00000	250.70000		THPFOS
5.155	3	VVA+	5.36024e6	1.62253e-1	214.17458		PFOS

Totals without ISTD(s) : 494.17652

=====

*** End of Report ***

TIC-002, 012901-naclPFOS-2.1.2, 100X Dil.

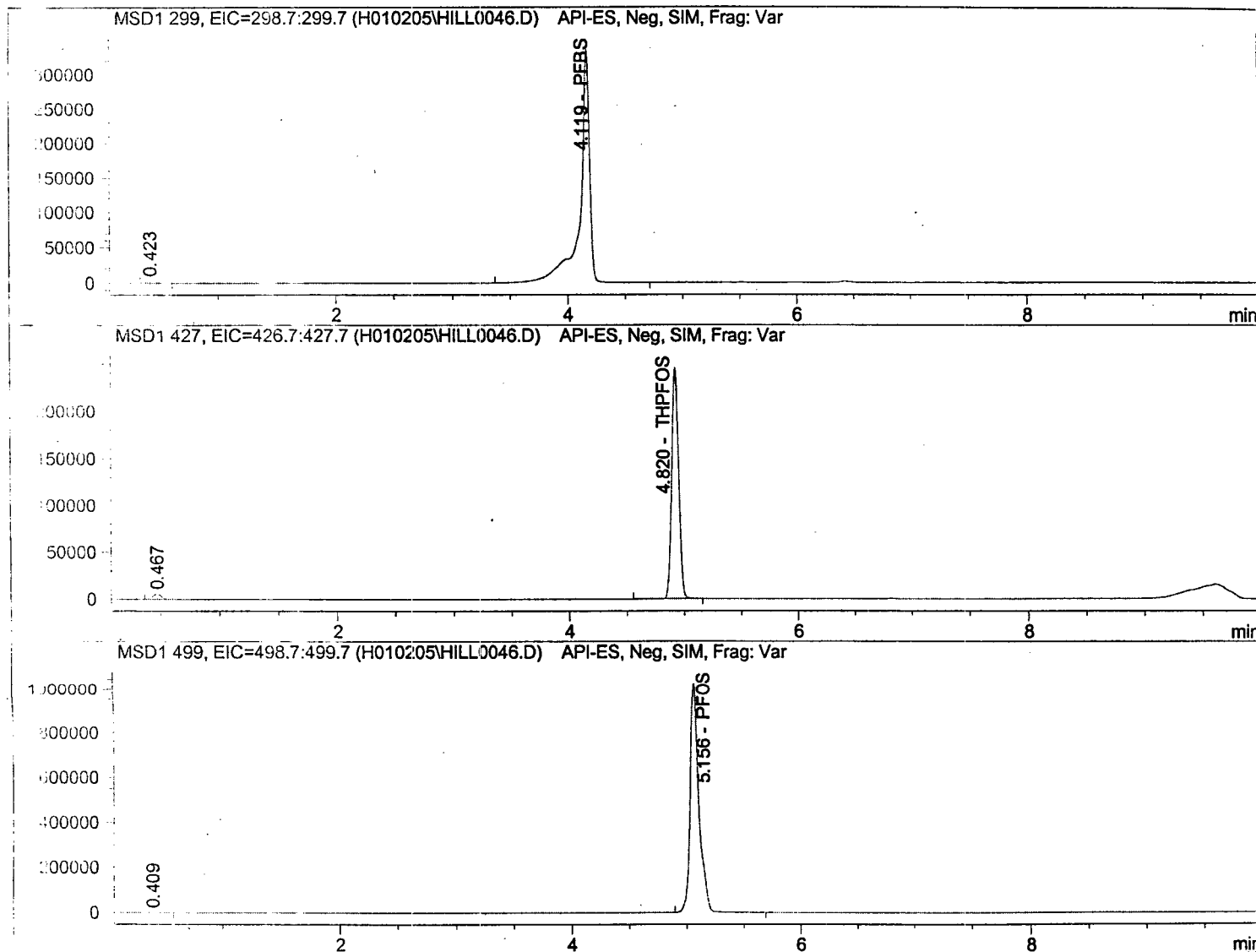
SIM 02-01-01 MEE(MLA)

=====

Injection Date	: 2/5/01 10:34:39 PM	Seq. Line	: 46
Sample Name	: naclPFOS-2.1.2	Vial	: 32
Anal. Operator	: MLA	Inj	: 1
Anal. Instrument	: Hillary	Inj Volume	: 10 µl
Anal. Method	: C:\HPCHEM\1\METHODS\H010205.M		
Last changed	: 2/5/01 2:09:33 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010205A.M		
Last changed	: 2/9/01 3:39:05 PM by MLA		

(modified after loading)

Sum run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Friday, February 09, 2001 3:22:24 PM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name
[ppb]

Exact Copy of Original
MLA Initial Date
02/09/01
page 1/2

TCR-002, 012901-naclPFOS-2.1.2, 100X Dil.

SFM 02-01-01 MEE (MLA)

```
=====
Injection Date   : 2/5/01 10:34:39 PM          Seq. Line :   46
Sample Name      : naclPFOS-2.1.2              Vial       :   32
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:39:05 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

```
=====
      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.119	1	VVA+	1.99539e6	5.81750e-1	283.28167		PFBS
4.820	2	PVA+ I	1.02730e6	1.00000	250.70000		THPFOS
5.156	3	VVA+	5.51518e6	1.62412e-1	218.59113		PFOS

Totals without ISTD(s) : 501.87280

```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-2.1.4, 100X Dil.

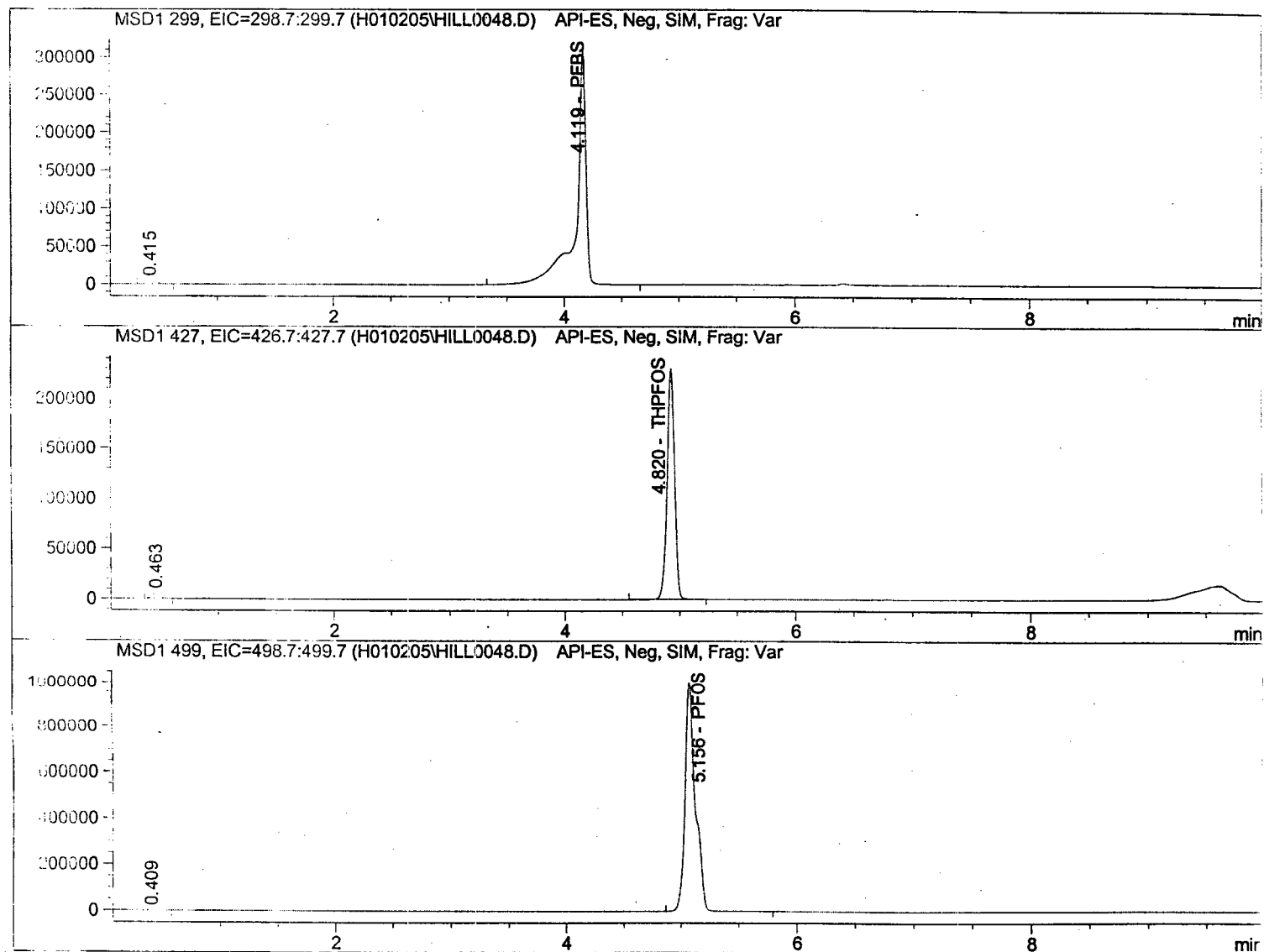
SFM 02-01-01 MEE (MLA)

=====

Injection Date	: 2/5/01 10:56:54 PM	Seq. Line	: 48
Sample Name	: naclPFOS-2.1.4	Vial	: 34
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010205.M		
Last changed	: 2/5/01 2:09:33 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010205A.M		
Last changed	: 2/9/01 3:39:15 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



=====

Internal Standard Report

=====

Sorted By	:	Retention Time
Calib. Data Modified	:	Friday, February 09, 2001 3:22:24 PM
Multiplier	:	1.0000
Dilution	:	1.0000
Sample ISTD Information:		
ISTD ISTD Amount Name		
# [ppb]		

Exact Copy of Original

MLA Initial

Date 03 Apr 02

Page 1 of 2

TCR-002, 012901-naclPFOS-2.1.4, 100X Dil.

SP4-02-01-01 MEE(MLA)

```
=====
Injection Date   : 2/5/01 10:56:54 PM          Seq. Line :   48
Sample Name      : naclPFOS-2.1.4              Vial       :   34
Acq. Operator    : MLA                          Inj        :    1
Acq. Instrument  : Hillary                      Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:39:15 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluoronated carboxylic acids

=====

1 250.70000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
1.119	1	VBA+	1.91682e6	5.81750e-1	275.75855		PFBS
1.820	2	PVA+ I	1.01378e6	1.00000	250.70000		THPFOS
5.156	3	PBA+	6.15837e6	1.63487e-1	248.97829		PFOS

Totals without ISTD(s) : 524.73683

=====

*** End of Report ***

TCR-002, 012901-naclPFOS-2.2.2, 100X Dil.

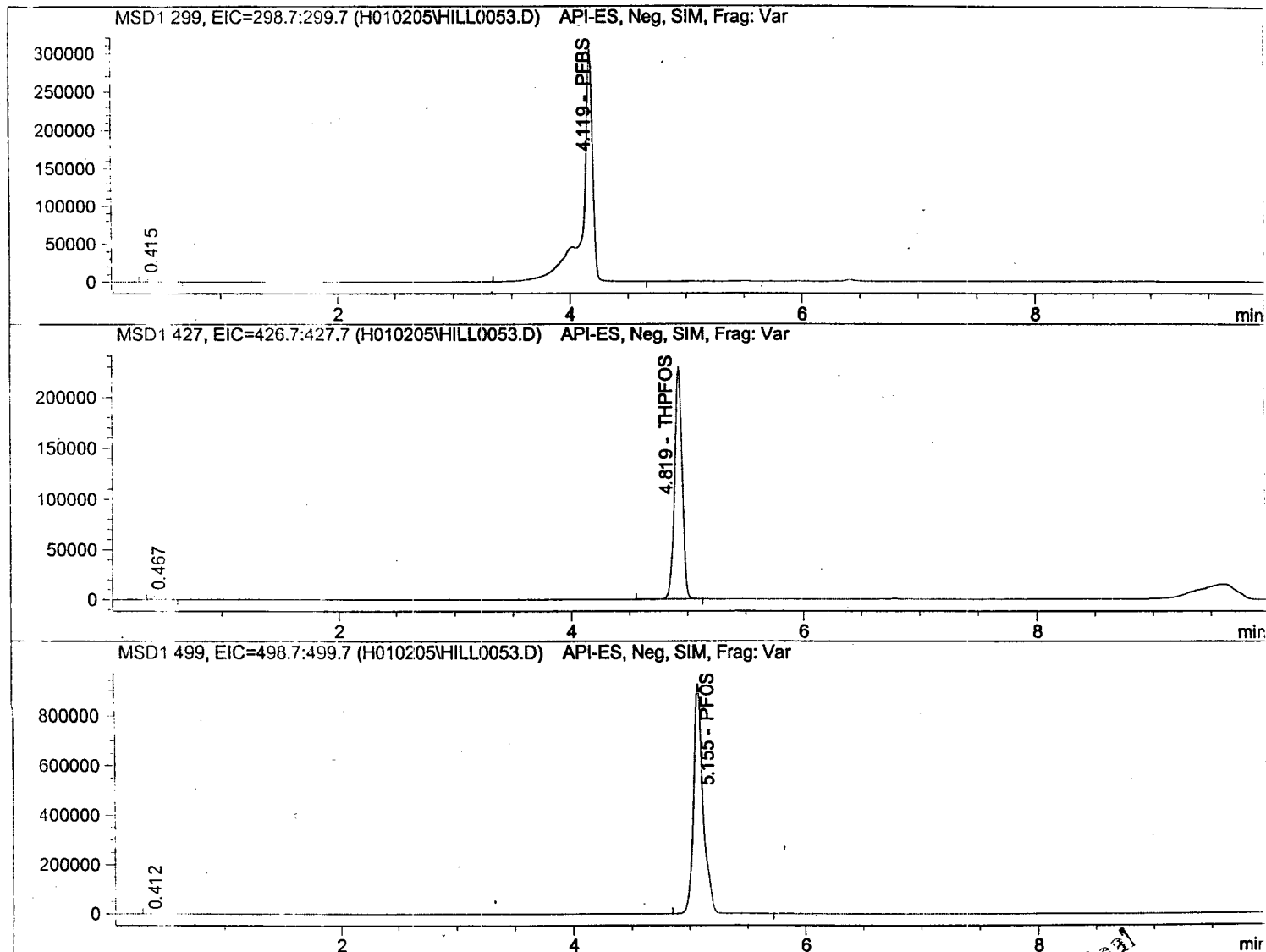
SFM 02-01-01 MEE (MLA)

=====

Injection Date	: 2/5/01 11:52:35 PM	Seq. Line	: 53
Sample Name	: naclPFOS-2.2.2	Vial	: 36
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010205.M		
Last changed	: 2/5/01 2:09:33 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010205A.M		
Last changed	: 2/9/01 3:39:40 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Friday, February 09, 2001 3:22:24 PM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name
[ppb]

Exact Copy of Original
MLA Initial
03 Apr 01
Liao
Page 1 of 2

TCR-002, 012901-naclPFOS-2.2.2, 100X Dil.

SFM 02-01-01 MEE(MLA)

```
=====
Injection Date   : 2/5/01 11:52:35 PM          Seq. Line :   53
Sample Name      : naclPFOS-2.2.2              Vial       :   36
Acq. Operator    : MLA                          Inj         :    1
Acq. Instrument  : Hillary                      Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:39:40 PM by MLA
                  (modified after loading)
Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids
=====
```

1 250.70000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.119	1	VVA+	1.89751e6	5.81750e-1	270.31266		PFBS
4.819	2	PVA+ I	1.02378e6	1.00000	250.70000		THPFOS
5.155	3	VVA+	5.22307e6	1.61999e-1	207.19812		PFOS

Totals without ISTD(s) : 477.51078

```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-2.2.3, 100X Dil.

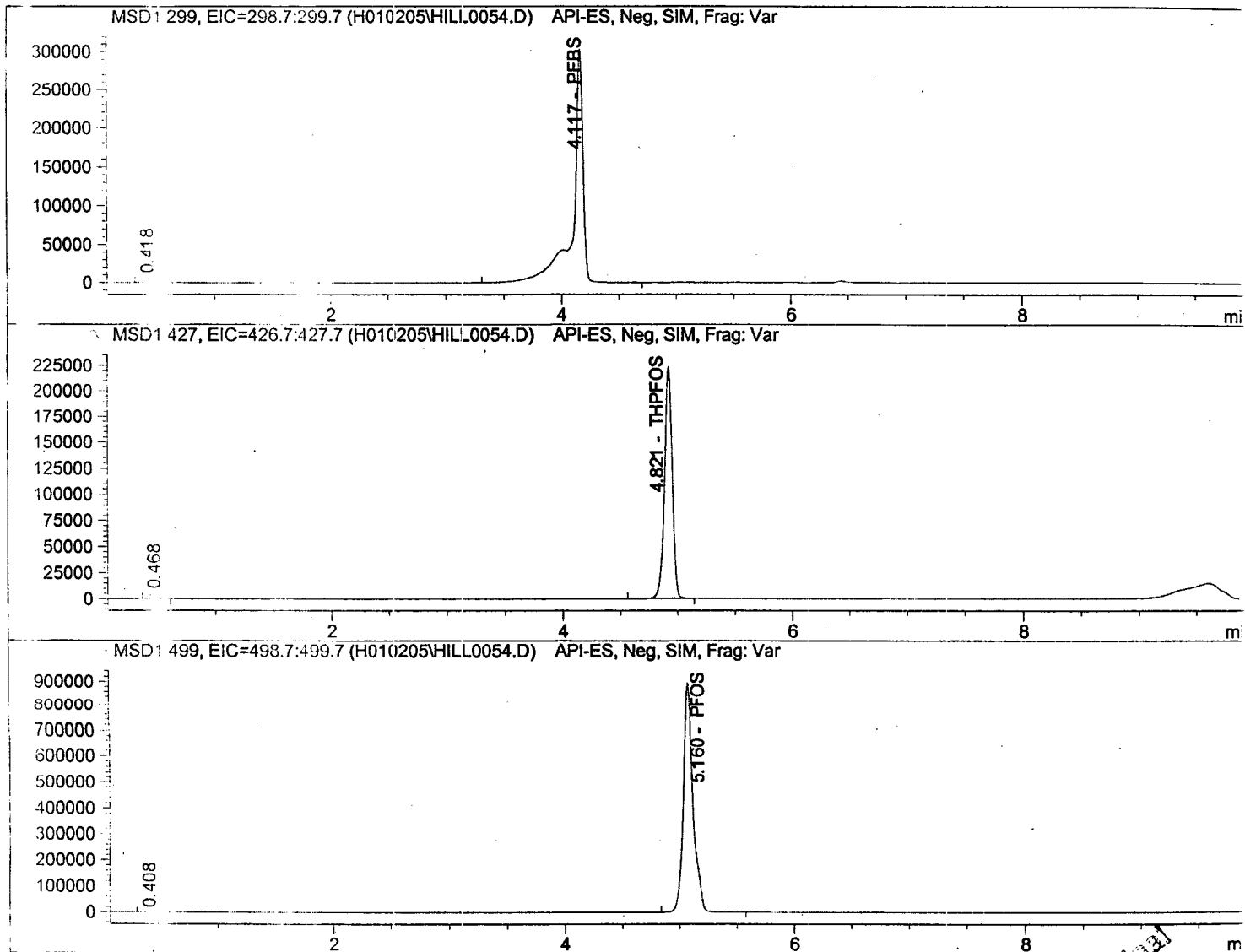
SPM 02-01-01 MEE (MLA)

=====

Injection Date	: 2/6/01 12:03:44 AM	Seq. Line	: 54
Sample Name	: naclPFOS-2.2.3	Vial	: 37
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010205.M		
Last changed	: 2/5/01 2:09:33 PM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010205A.M		
Last changed	: 2/9/01 3:39:45 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Friday, February 09, 2001 3:22:24 PM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD #	ISTD Amount	Name
[ppb]		

Exact Copy of Original
MLA Initial
Date 03/01/01
Page 1 of 2

TCR-002, 012901-naclPFOS-2.2.3, 100X Dil.

SEM 02-01-01 MEE (MLA)

```
=====
Injection Date   : 2/6/01 12:03:44 AM          Seq. Line :   54
Sample Name      : naclPFOS-2.2.3              Vial       :   37
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010205.M
Last changed     : 2/5/01 2:09:33 PM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010205A.M
Last changed     : 2/9/01 3:39:45 PM by MLA
                  (modified after loading)
Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids
=====
```

1 250.70000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.117	1	VBA+	1.88069e6	5.81750e-1	267.76401		PFBS
4.821	2	VVA+ I	1.02437e6	1.00000	250.70000		THPFOS
5.160	3	VVA+	5.13424e6	1.61860e-1	203.38226		PFOS

Totals without ISTD(s) : 471.14627

```
=====
*** End of Report ***
```

TCR-002, 012901-naclPFOS-3.1.2, 100X Dil.

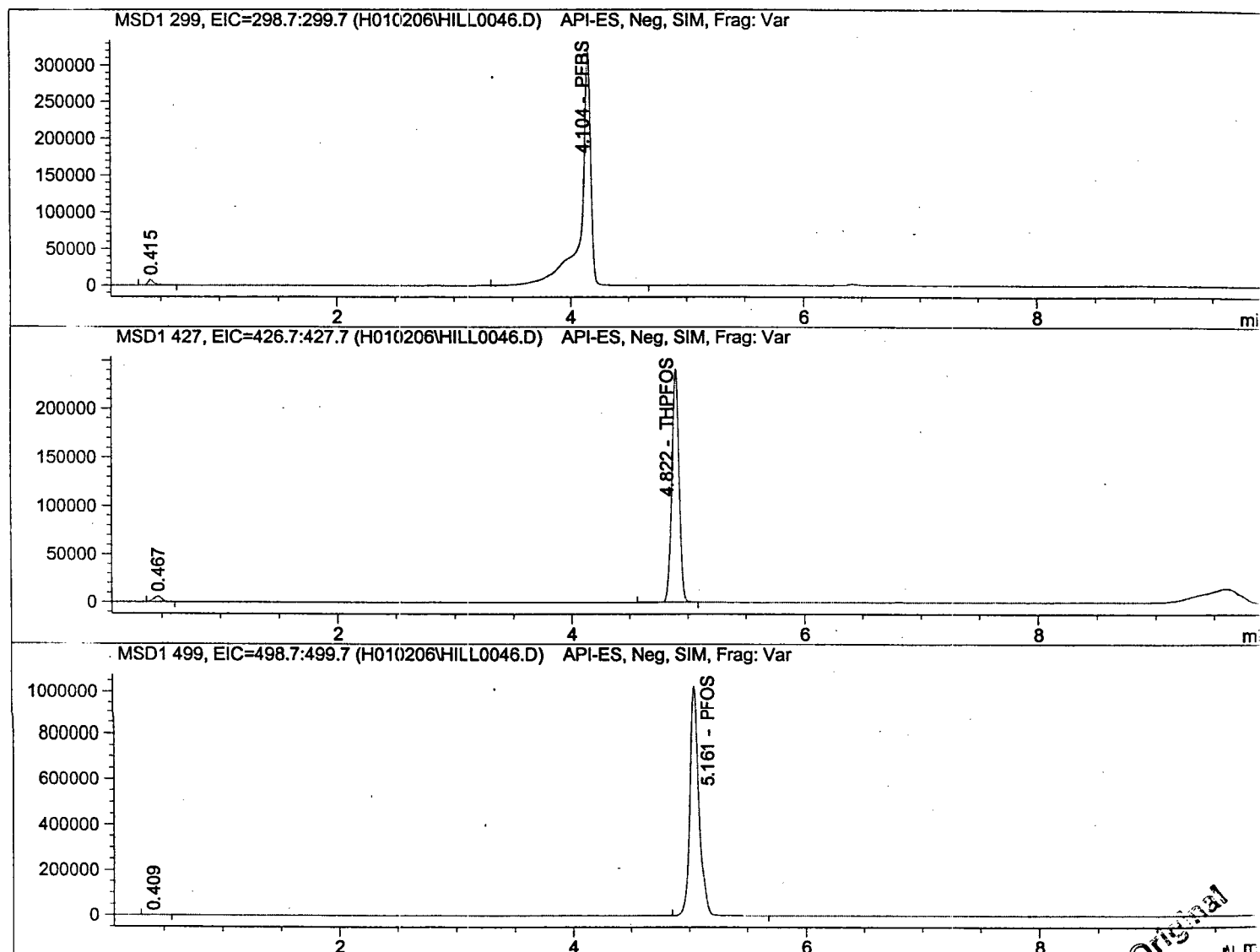
SFM 02-02-01 MEE (MLA)

=====

Injection Date	: 2/6/01 8:13:35 PM	Seq. Line	: 46
Sample Name	: naclPFOS-3.1.2	Vial	: 62
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010206.M		
Last changed	: 2/6/01 11:47:54 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010206A.M		
Last changed	: 2/7/01 1:16:10 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



=====

Internal Standard Report

=====

Sorted By	:	Retention Time
Calib. Data Modified	:	Wednesday, February 07, 2001 12:53:59 PM
Multiplier	:	1.0000
Dilution	:	1.0000

Sample ISTD Information:

ISTD	ISTD Amount	Name
#	[ppb]	

-----|-----|-----

Exact Copy of Original

MLA Initial

Date

Page 1 of 2

TCR-002, 012901-naclPFOS-3.1.2, 100X Dil.

SFM 02-02-01 MEE(MLA)

```
=====
Injection Date   : 2/6/01 8:13:35 PM           Seq. Line :   46
Sample Name      : naclPFOS-3.1.2              Vial       :   62
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:16:10 PM by MLA
                  (modified after loading)
Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids
=====
```

1 250.70000 THPFOS

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.104	1	VBA+	1.90390e6	6.10461e-1	281.92266		PFBS
4.822	2	PVA+ I	1.03354e6	1.00000	250.70000		THPFOS
5.161	3	VVA+	5.38770e6	1.63206e-1	213.28895		PFOS

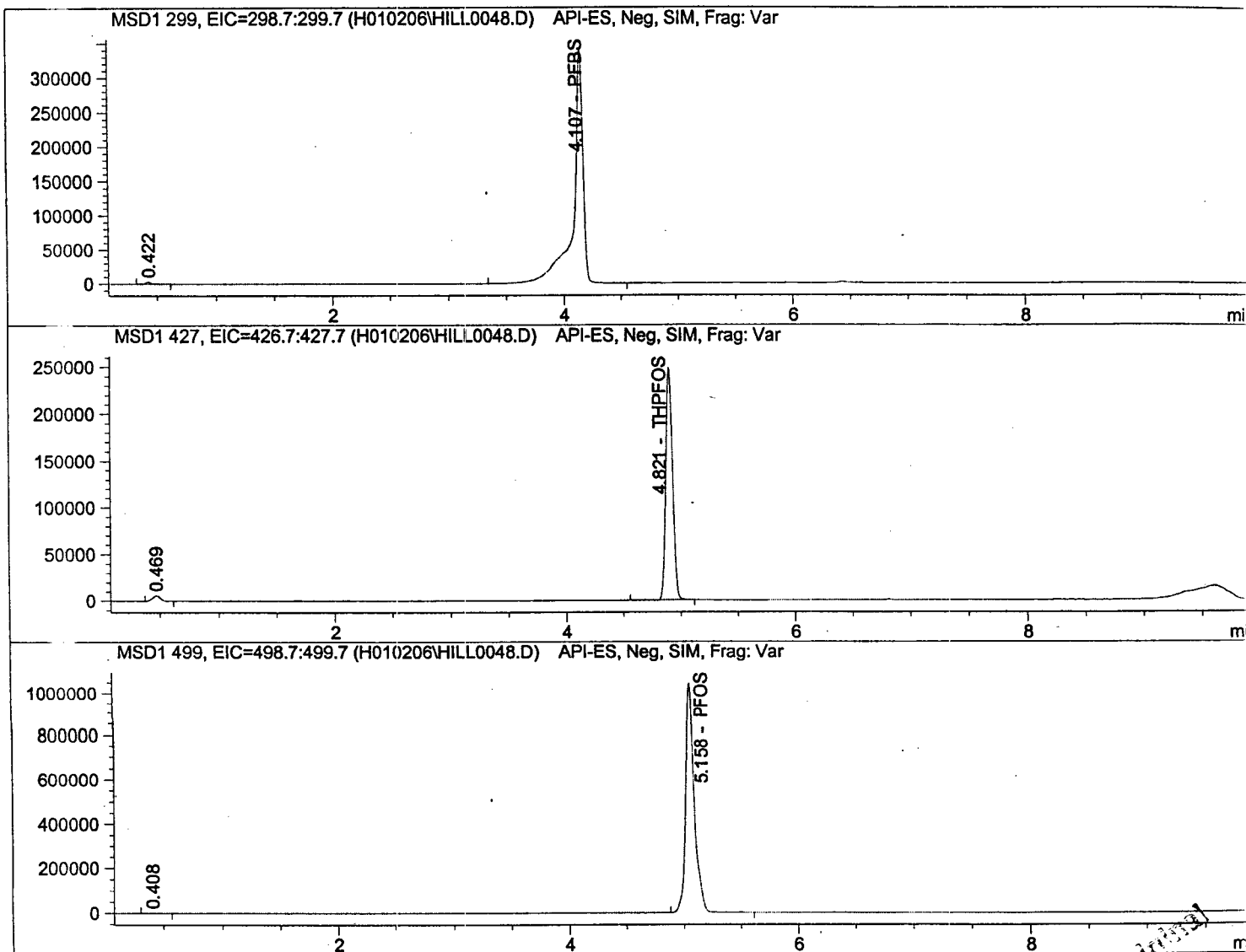
Totals without ISTD(s) : 495.21161

```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-3.1.4, 100X Dil.

SFM 02-02-01 MEE (MLA)

```
=====
Injection Date   : 2/6/01 8:35:51 PM           Seq. Line :   48
Sample Name      : naclPFOS-3.1.4              Vial       :   64
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:16:19 PM by MLA
                  (modified after loading)
Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids
=====
```



=====
Internal Standard Report
=====

```
Sorted By           :      Retention Time
Calib. Data Modified :      Wednesday, February 07, 2001 12:53:59 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Sample ISTD Information:
ISTD  ISTD Amount  Name
#      [ppb]
-----|-----|-----
```

Exact Copy of Original
MLA Initial
02/07/01
page 1/2

TCR-002, 012901-naclPFOS-3.1.4, 100X Dil.

SFM 02-02-01 MEE (MLA)

```
=====
Injection Date   : 2/6/01 8:35:51 PM      Seq. Line :   48
Sample Name      : naclPFOS-3.1.4         Vial       :   64
Acq. Operator    : MLA                   Inj        :    1
Acq. Instrument  : Hillary                Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:16:19 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

```
=====
1      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.107	1	VVA+	2.02831e6	6.10461e-1	305.11576		PFBS
4.821	2	PVA+ I	1.01737e6	1.00000	250.70000		THPFOS
5.158	3	VVA+	5.36732e6	1.63334e-1	216.02714		PFOS

Totals without ISTD(s) : 521.14289

```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-3.2.1, 100X Dil.

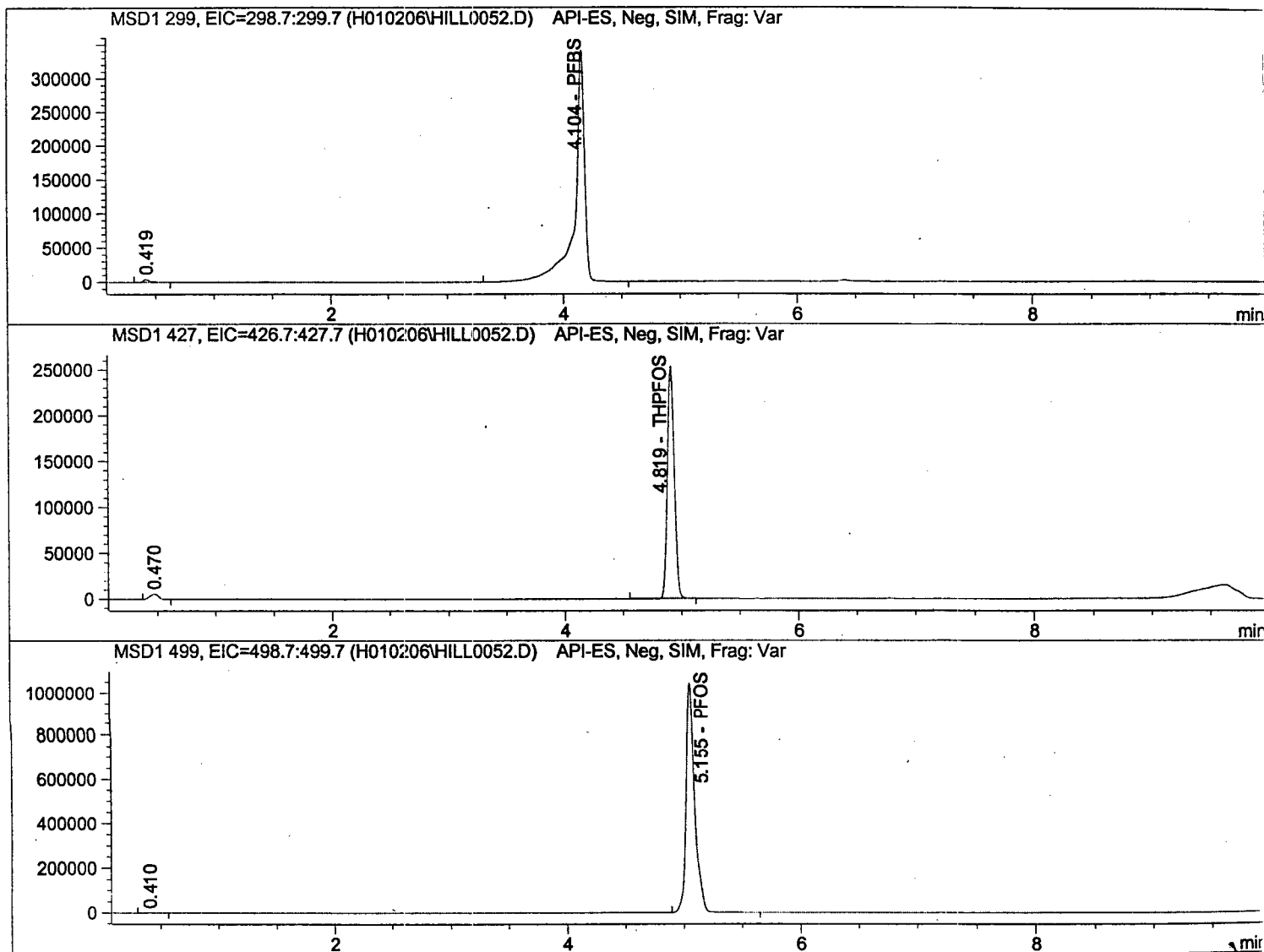
SFM 02-02-01 MEE (MLA)

=====

Injection Date	: 2/6/01 9:20:29 PM	Seq. Line	: 52
Sample Name	: naclPFOS-3.2.1	Vial	: 65
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010206.M		
Last changed	: 2/6/01 11:47:54 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010206A.M		
Last changed	: 2/7/01 1:16:38 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Wednesday, February 07, 2001 12:53:59 PM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name
[ppb]

Exact Copy of Original
Date
Initial
page 1/2

TCR-002, 012901-naclPFOS-3.2.1, 100X Dil.

SFM 02-02-01 MEE (MLA)

```
=====
Injection Date   : 2/6/01 9:20:29 PM           Seq. Line :   52
Sample Name      : naclPFOS-3.2.1              Vial       :   65
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:16:38 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

```
=====
1      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.104	1	VVA+	1.99860e6	6.10461e-1	297.34485		PFBS
4.819	2	PVA+ I	1.02867e6	1.00000	250.70000		THPFOS
5.155	3	VVA+	5.24716e6	1.62979e-1	208.41657		PFOS

Totals without ISTD(s) : 505.76142

```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-3.2.3, 100X Dil.

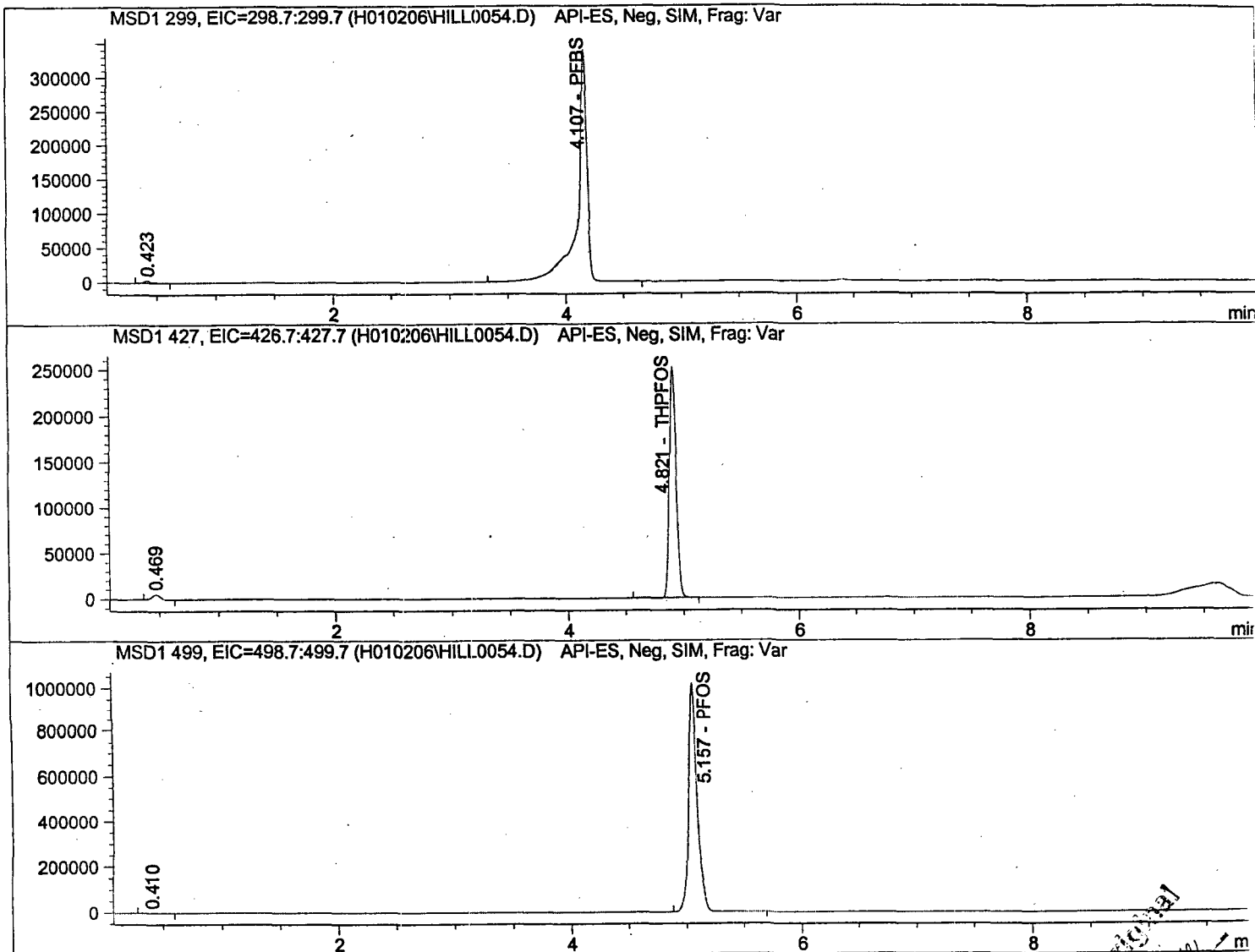
SFM 02-02-01 MEE (MLA)

=====

Injection Date	: 2/6/01 9:42:48 PM	Seq. Line	: 54
Sample Name	: naclPFOS-3.2.3	Vial	: 67
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010206.M		
Last changed	: 2/6/01 11:47:54 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010206A.M		
Last changed	: 2/7/01 1:16:47 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluoronated carboxylic acids



Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Wednesday, February 07, 2001 12:53:59 PM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name
[ppb]

Exact Copy of Original
MLA Initial
02/07/01
paw/lyl

TCR-002, 012901-naclPFOS-3.2.3, 100X Dil.

SFM 02-02-01 MEE (MLA)

```
=====
Injection Date   : 2/6/01 9:42:48 PM           Seq. Line :   54
Sample Name      : naclPFOS-3.2.3              Vial       :   67
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:16:47 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C3 perfluorinated carboxylic acids

```
=====
1      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.107	1	VVA+	2.00067e6	6.10461e-1	299.37708		PFBS
4.821	2	PVA+ I	1.02275e6	1.00000	250.70000		THPFOS
5.157	3	VVA+	5.14429e6	1.62835e-1	205.33247		PFOS

Totals without ISTD(s) : 504.70955

```
=====
*** End of Report ***
=====
```

TCR-002, 012901-naclPFOS-3.3.3, 100X Dil.

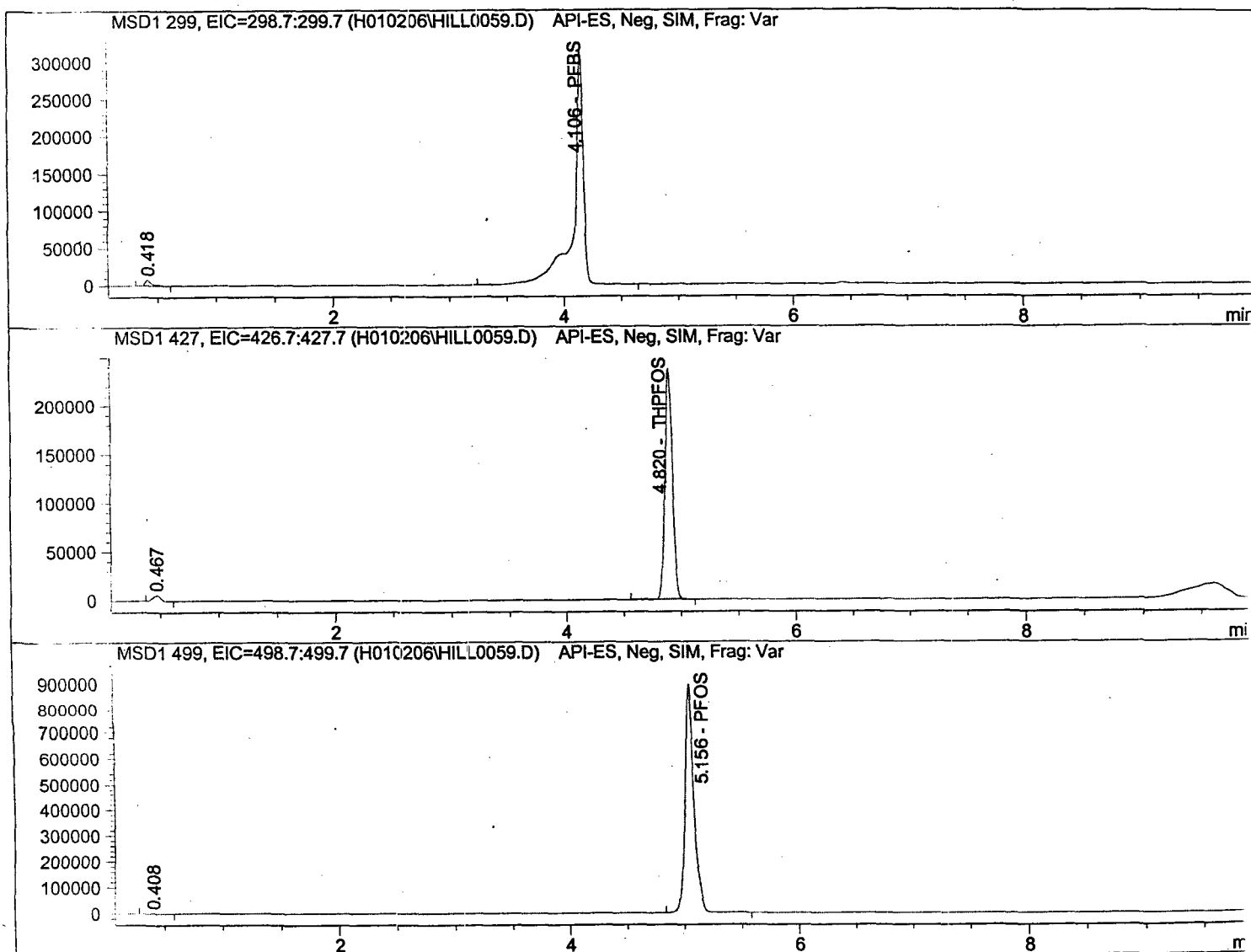
SFM 02-02-01 MEE(MLA)

=====

Injection Date	: 2/6/01 10:38:35 PM	Seq. Line	: 59
Sample Name	: naclPFOS-3.3.3	Vial	: 71
Acq. Operator	: MLA	Inj	: 1
Acq. Instrument	: Hillary	Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\H010206.M		
Last changed	: 2/6/01 11:47:54 AM by MLA		
Analysis Method	: C:\HPCHEM\1\METHODS\H010206A.M		
Last changed	: 2/7/01 1:17:11 PM by MLA		

(modified after loading)

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids



Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Wednesday, February 07, 2001 12:53:59 PM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:
ISTD ISTD Amount Name
[ppb]

Exact Copy of OIR

MLA
InitialB2A
Di
Page 1 of 2

TCR-002, 012901-naclPFOS-3.3.3, 100X Dil.

SFM 02-02-01 MEE(MLA)

```
=====
Injection Date   : 2/6/01 10:38:35 PM          Seq. Line :   59
Sample Name      : naclPFOS-3.3.3              Vial       :   71
Acq. Operator    : MLA                        Inj        :    1
Acq. Instrument  : Hillary                    Inj Volume  : 10 µl
Acq. Method      : C:\HPCHEM\1\METHODS\H010206.M
Last changed     : 2/6/01 11:47:54 AM by MLA
Analysis Method  : C:\HPCHEM\1\METHODS\H010206A.M
Last changed     : 2/7/01 1:17:11 PM by MLA
                  (modified after loading)
=====
```

Sim run C2,c3,c4,c5, c7 and C8 perfluorinated carboxylic acids

```
=====
1      250.70000    THPFOS
=====
```

Signal 1: MSD1 299, EIC=298.7:299.7

Signal 2: MSD1 427, EIC=426.7:427.7

Signal 3: MSD1 499, EIC=498.7:499.7

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
4.106	1	VBA+	1.90270e6	6.10461e-1	283.01915		PFBS
4.820	2	PVA+ I	1.02888e6	1.00000	250.70000		THPFOS
5.156	3	VVA+	4.69662e6	1.61909e-1	185.28658		PFOS

Totals without ISTD(s) : 468.30573

```
=====
*** End of Report ***
=====
```

ATTACHMENT D: DEVIATIONS FROM THE PROTOCOL

Record of Deviation

I. Identification

Study / Project No.

TCR-002

Deviation Type:

(Check one)

☐ SOP☒ Method☐ Equipment Procedure☐ Protocol☐ Other:

Document Number: ETS-8-155.0

Date(s) of occurrence: 01 Jan 01 to 07 Feb 01

II. Description:

Required Procedure/process: ETS-8-155.0 section 10.2.1 states, "Matrix spikes are prepared for each sample set and analyzed to determine the matrix effect on the recovery efficiency." Section 10.2.3 states, "analyze the matrix spike and the matrix spike duplicate (if prepared) in the same run as the original sample."

Actual Procedure/process: The matrix spikes were not prepared, therefore, they were not analyzed.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)

The ETS-8-155.0 method has been revised so that matrix spikes do not need to be prepared when there is no need.

Recorded By:

Date:



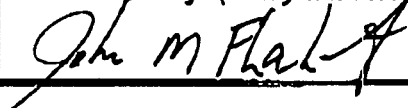
17 May 01

IV. Impact on Study / Project

The impact of this deviation on the study is minimal. The lack of a matrix spike has no bearing on the final results. The matrix is well defined and characterized. The matrix spike is actually not possible in this study since the matrix is saturated with the analyte of interest.

Authorized By: (Study Director / Project Lead)

Date:



5/17/01

3M Environmental Laboratory

Deviation No. _____

RECEIVED TIME
P. 5 NO. 533

MAY. 17.

2:28PM

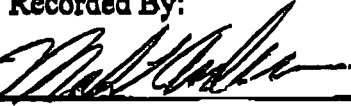
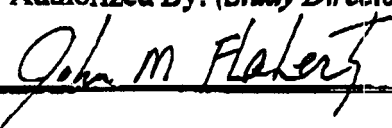
FACT-TCR002 (LIMS #E00-1716), Page 134 of 138

CENTRE ANALYTICAL LAB

4:16PM

MAY. 17. 2001

Record of Deviation

I. Identification	
Study / Project No. TCR-002	
Deviation Type: ☐ SOP ☒ Method ☐ Equipment Procedure (Check one) ☐ Protocol ☐ Other:	
Document Number: ETS-8-155.0	Date(s) of occurrence: 01 Jan 01 to 07 Feb 01
II. Description:	
Required Procedure/process: ETS-8-155.0 section 10.1.2 states, "Analyze a method blank and a matrix blank prior to each calibration curve."	
Actual Procedure/process: In this experiment, the method and matrix blanks are essentially the same. The method/matrix blanks were not ran before the calibration curves. The method blanks were ran after the first calibration curve.	
III. Actions Taken: (such as amendment issued, SOP revision, etc.) The ETS-8-155.0 method has been revised so that the method and matrix blanks are to be ran after the first calibration curve.	
Recorded By: 	Date: 17 May 01
IV. Impact on Study / Project	
The impact of this deviation on the study is minimal. The placement of the blanks have no bearing on the results. See Corrective Action.	
Authorized By: (Study Director / Project Lead) 	Date: 5/17/01

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. _____
(assigned by Study Director or Project Lead at the end of study or project)

RECEIVED TIME
P. 2 EES NO.

MAY. 17. 2:28PM

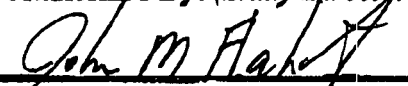
FACT-TCR002 (LIMS #E00-1716) Page 135 of 138

CENTRE ANALYTICAL LAB

4:14PM

MAY. 17. 2001

Record of Deviation

I. Identification	
Study / Project No. TCR-002	
Deviation Type: ☐ SOP ☒ Method ☐ Equipment Procedure (Check one) ☐ Protocol ☐ Other:	
Document Number: ETS-8-155.0	Date(s) of occurrence: 01 Jan 01 to 07 Feb 01
II. Description:	
Required Procedure/process: In dealing with the standard curve, the curve must have a coefficient of determination (r^2) value greater than or equal to 0.980 and individual curve points must have 'Percent Deviations' equal to or less than +/- 30%.	
Actual Procedure/process: In some cases, high and/or low curve points were eliminated so that the r^2 value would meet the criteria. In other cases, high and/or low curve points were eliminated, as they were not within the +/-30% accuracy requirements. When this was done, the curve point(s) affected and the action(s) taken were annotated on the 'Data Summary Sheet'.	
The following is a list of all curve points affected and the reasons why. (Day 1 not used) 3.5% NaCl Day 2, (H010205A), points HILL0025, 0026, 0066 and 0067. 3.5% NaCl Day 2, (H010205A), points HILL0025 and 0066. All of these points were excluded to provide a better fit over the range appropriate to the data.	
III. Actions Taken: (such as amendment issued, SOP revision, etc.)	
The 3M Environmental Method ETS-8-155.0 was amended to take into account the elimination of curve points. Curve points may be omitted for the following reasons: (Comments are bulleted as they would appear on the summary sheets.) (1) "High/low curve points were excluded to provide a better fit over the range appropriate to the data." (2) "Low level point(s) were not 2x higher than the extraction blank; these points were excluded from the curve to disqualify a data range that may have been significantly affected by background levels of the analyte." (3) "High/low curve point(s) were excluded as they were not within the +/- 30% accuracy requirements of the method when the curves were evaluated over a range appropriate to the data."	
Recorded By: 	Date: 17 May 01
IV. Impact on Study / Project	
The elimination of curve points had a positive impact on this study. If curve points were not eliminated, then the curves would not be as accurate.	
Authorized By: (Study Director / Project Lead) 	Date: 5/17/01

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. _____

(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification	
Study / Project No. TCR-002	
Deviation Type: (Check one)	☐ SOP ☒ Method ☐ Equipment Procedure ☐ Protocol ☐ Other:
Document Number: ETS-8-172.1	Date(s) of occurrence: 22 Jan 01 to 07 Feb 01
II. Description:	
Required Procedure/process: ETS-8-172.1 indicates that a screen test must be performed prior to the shake flask method.	
Actual Procedure/process: In the seawater and 3.5% NaCl portions of this study, the screen test was not done.	
III. Actions Taken:	
Notes to File were written concerning this issue.	
Recorded By: <i>Mark L. Anderson</i>	Date: <i>17 May 01</i>
IV. Impact on Study / Project	
There is minimal impact on this study by not doing screening tests in these matrices. The screen tests were not done because of two main reasons: 1- It was not done to conserve material, and 2- The solubility of PFOS was already known for water and it was also known that the solubility would decrease in seawater and 3.5% NaCl.	
Authorized By: (Study Director / Project Lead) <i>John M. Flaherty</i>	Date: <i>5/17/01</i>

ATTACHMENT E: SAMPLE CALCULATIONS

Mean and standard deviation were calculated using functions provided in Microsoft Excel® software.

Standard deviation was used to measure the scatter about the mean of a data set; thus, it can be used to estimate the precision of a method. Relative standard deviation presents a measure of the magnitude of the standard deviation and is calculated by dividing the standard deviation by the mean. Means are calculated by adding individual entities and dividing the resultant sum by the number of individual entities. Standard deviation was calculated using the following equation:

$$\sqrt{\frac{n\sum x^2 - (\sum x)^2}{n(n-1)}}$$

-Where n is the number of observations, and x is sample concentration.

Average/Standard Deviation /%RSD example calculations:

Sample ID	Concentration $\mu\text{g/mL}$	Calculations
Rep1	647.9	Average $(647.9+723.4+723.0+717.3)/4= 702.9$ Std Dev (see above equation)= 36.8 %RSD $(36.8/702.9)= 5.2\%$
Rep 2	723.4	
Rep 3	723.0	
Rep 4	717.3	

Data that did not meet acceptance criteria as described in the OPPTS and OECD guidelines was excluded using statistical justification provided by Dixon's Q-test. A data point may be excluded if " Q_{observed} " is greater than " $Q_{\text{tabulated}}$ " with 90% confidence. $Q_{\text{observed}} = \text{gap}/\text{range}$ where "gap" is the difference between the questionable data point and the closest value of the data set, and "range" is the difference between the highest and lowest value of the data set. $Q_{\text{tabulated}}$ (90% confidence) for 4 observations (or data points) is 0.679 and for 3 observations is 0.886.

Q-Test Example (Shaded number represents the suspected outlier):

Description	$\mu\text{g/mL}$
Sample Conc., Rep1	607.1
Sample Conc., Rep2	591.7
Sample Conc., Rep3	626.1
Sample Conc., Rep4	2648.8
Range (Rep4-Rep2):	2057.14
Gap (Rep4-Rep3):	2022.66
Q_{observed} (Range/Gap):	0.98
No. observations:	4
Retain?	no

Q-Test example conclusion: Since Q_{observed} , 0.98, was greater than $Q_{\text{tabulated}}$, 0.679, rep 4 may be excluded.