Study Title

Solubility Study of Perfluorooctanesulfonate (PFOS), SD-018 and Perfluorooctanesulfonamide (FOSA), SE-027 in Male/Female Human Urine

Data Requirement

40 CFR 160.105(b)

Author

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Phase Completion Date

Date of signing

Performing Laboratory

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Project Identification

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3M LIMS#: 25160 (PFOS SD-018), and #25161 (FOSA SE-027)

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Experimental Dates

Experimental Start Date: 23 April 2001
Experimental Termination Date: 01 May 2001

Archives

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

SUMMARY

The approximate solubility of Perfluorooctanesulfonate (PFOS) and Perfluorooctanesulfonamide FOSA in human urine was determined by performing a solubility screen test, followed by a quantitative solubility determination via Shake Flask Method.

Results are presented in table 1a and 1b:

Table 1a. Summary Table of Solubility of PFOS SD-018 in Urine.

SAMPLING EVENT	PFOS SOLUBILITY IN URINE (CORRECTED FOR STD. PURITY)	Standard Deviation	% CV
Day 1	302µg/mL	12	4%
Day 2	325 µg/mL	40	12%
Day 3	287 µg/mL	30	10%
Average Value	**305 µg/mL**	14	5%

The solubility of PFOS SD-018 in urine was determined to be 305 µg/mL at 23-24 °C.

Table 1b. Summary Table of Solubility of FOSA SE-027 in Urine.

SAMPLING EVENT	FOSA SOLUBILITY IN URINE	Standard Deviation	% CV
Day 1	2.56 µg/mL	0.38	15%
Day 2	2.88 µg/mL	0.12	4%
Day 3	2.61 µg/mL	0.05	2%
Average Value	**2.68 µg/mL**	0.18	7%

The solubility of FOSA SE-027 in urine was determined to be 2.68 µg/mL at 23-24 °C.

PURPOSE

The purpose of this solubility study was to determine the solubility of PFOS and FOSA in urine.

TEST SUBSTANCE

PFOS, Test Control Reference #SD-018 and FOSA, Test Control Reference # SE-027

Table 2. Characterization of the Test Substance

TEST SUBSTANCE	PFOS	FOSA
Source	98-0211-0888-5	R. Buckanin, 3M
Expiration Date	08/31/01	Not Available
Storage Conditions	Frozen	Frozen
3M Laboratory Identification Number	SD-018	SE-027
Physical Description	White powder	White powder/chunks
Purity	86.9%	To Be Determined

REFERENCE SUBSTANCE

Table 3. Characterization of the Analytical Reference Substances

REFERENCE SUBSTANCE	PFOS	FOSA	THPFOS	PFBS
Source	Re-crystallized by George Moore, 3M Specialty Materials, Bldg. 236-1B-10	R. Buckanin, 3M	SynQuest Labs	Pat Savu, 3M
Expiration Date	01-01-2010	Not Available	Not Available	Not Available
Storage Conditions	Frozen	Frozen	Frozen	Frozen
3M Laboratory Identification Number	TCR-00017-46	SE-027	TCR-00017-047 Lot #Q75-30	Primary Standard, TCR-99030-23 (L-7038, F-8442); Lot 101
Physical Description	White Powder	White powder/chunks	White Powder	White Powder
Purity	97.9%	To Be Determined	*	*

** The purity of THPFOS and PFBS (currently being determined by Centre Analytical Laboratory) will not affect any analytical findings since it is used as a reference substance only.*

TEST SYSTEM

Table 4. Description of the test system used in this study

TEST SYSTEM/SOLVENT	HUMAN URINE
Source	Lampire
Expiration Date	03-16-05
Storage Conditions	Frozen
3M Laboratory Identification Number	TN-A 4822
Purity/Grade	Normal Male/Female Urine

METHOD SUMMARIES

The solubility determination of PFOS SD-018 and FOSA SE-027 in urine was performed according to 3M Environmental Laboratory methods ETS-8-170.1 "Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents;" and ETS-8-172.1 "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. 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 Solubility Screen Test Method (ETS-8-170.1) was used to determine the solubility range of PFOS SD-018 and FOSA SE-027 in urine. Incremental amounts of urine were added to PFOS and FOSA neat material and a qualitative determination of the solubility point was made. The screen test indicated a solubility of PFOS in urine of less than 200 μg/mL, and FOSA in urine of less than 200 μg/mL at 23 °C. Therefore the Shake Flask Method was used for the quantitative determination of PFOS and FOSA solubility in urine. Detailed descriptions of the Solubility Screen Test Method and Shake Flask methods used in this study are located in Attachment A.

PREPARATORY METHODS

- **ETS-8-170.1 "Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents."** This method is a prerequisite to the shake flask method and gives an estimate of the solubility point of the test substance in the solvent of choice. Approximately ten milligrams of PFOS/FOSA was weighed into glass vials, and varying amounts of urine were added in a step-wise fashion. After each urine addition, the vials were shaken, sonicated, and observed for particulate. When particulate was observed in a final volume of 2 mL of urine (approximately 5000 μg/mL) for both PFOS and FOSA, the final step of the screen test was initiated. Approximately 10 mg PFOS/FOSA was placed into a 100 mL volumetric flask and urine was added in a step-wise fashion up to 50 mL (with shaking, vortex-mixing, sonicating, and observations for particulate in between). Due to fine particulates present in the urine, it was difficult to tell the difference between urine that did and did not have the test substance in it. This indistinguishable difference between FOSA and PFOS in urine and a urine blank sample was determined to be the stopping point for the screen test. The approximate solubility of PFOS and FOSA in urine at 23 °C was 200 μg/mL. This concentration estimation was used as a starting point for the shake flask method, and as a guide to determine the amount of dilution necessary to bring the final sample concentration into the analytical range of method ETS-8-155.0 (approximately 2.5 – 1000 ng test substance/mL).

Table 5. Solubility Screen Test Sample Preparation

STEP	CENTRIFUGE TUBES PREPARATION
1	10 mg test substance weighed into tared 4.0 mL glass screw-capped vial
2	0.1 mL of urine added to glass screw-capped vial
3	Following the addition of urine, vial was capped, shaken vigorously and vortexed, and sonicated. Mixing and sonication times recorded on the worksheet.

Table 5. Solubility Screen Test Sample Preparation

STEP	CENTRIFUGE TUBES PREPARATION
4	Sample checked visually for undissolved particles of the test substance. (If all of the test substance is dissolved, the solubility is determined to be greater than 10% (mass/volume) and no additional solubility testing is required. If a portion of the test substance remains undissolved, additional dilutions are performed using the appropriate solvent.) Observation made: undissolved particles present... continue to step 5.
5	Step 2 repeated with total volume of urine of 0.5, 1, and 2 mL, respectively. Following urine addition, vial was shaken, sonicated, and observed for particulate.
6	Following the final urine addition to bring the volume of urine to 2 mL, there was still undissolved PFOS and FOSA. The screen test method would therefore be continued to step 7.
7	Approximately 10 mg PFOS/FOSA were weighed into 100 mL volumetric flasks and urine was added stepwise up to 50 mL. Following each addition of urine the flask was capped, vortex-mixed, sonicated, and allowed to settle. The appearance of urine without test substance looked about the same as urine with PFOS or FOSA at 50 mL.
8	The concentration of urine was approximately 200 µg/mL and there was still particulate. The shake flask method shall be used for the quantitative determination of PFOS/FOSA solubility in urine.

- **ETS-8-172.1 "Shake Flask Method: Solubility Determination of a Test Substance in Various Solvents."** Approximately 10 mg test substance was weighed into a tared 15 mL plastic centrifuge tube. Urine was added gravimetrically to approximately 4.7-5 g (approximately 5 mL if density of urine is assumed to be approximately 1 g/mL). For each test substance study, nine centrifuge tubes were prepared, along with three method blanks (tubes with 5 g ± 0.5 g urine only). The centrifuge tubes were placed in a temperature-controlled orbital shaker set at approximately 30 °C. See the following table documenting the shake flask method preparation:

Table 6. Shake Flask Method Preparation for Study

STEP	CENTRIFUGE TUBE PREPARATION	REPLICATES	DATE(S) PERFORMED
1	≈ 0.010 g PFOS/FOSA weighed into tared 15 mL centrifuge tube	9 PFOS/ 9 FOSA	04-23-01
2	Urine added to centrifuge tube such that total weight of PFOS/FOSA and urine is approximately 5 g.	9 PFOS/ 9 FOSA	04-23-01
3	Method Blank Preparation- Approximately 5 g urine added to a tared centrifuge tube gravimetrically.	3 PFOS/ 3 FOSA	04-23-01
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 PFOS /12 FOSA	04-23-01

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 (3 containing PFOS and 3 containing FOSA) and a method blank were removed for analysis. The centrifuge tubes with PFOS and FOSA in urine, and method blanks (urine only) were allowed to equilibrate at 23-24 °C for 24 hours, centrifuged, and aliquots of

supernatant were collected. For the PFOS samples, aliquots were first diluted 100X (990 µL methanol: 10 µL sample), and then diluted further to 1,000X (900 µL methanol: 100 µL sample). For the FOSA samples, aliquots were diluted 10X (900 µL methanol: 100 µL sample). Final dilutions were spiked with internal standard (17 µL of 15 µg THPFOS and PFBS/mL) and every fourth sample replicate was spiked with 4 µL test analyte (25.05 µg/mL PFOS, 24 µg/mL FOSA). Samples were analyzed via HPLC/ES/MS according to analytical method ETS-8-155.0. Four injections (three samples and one sample spike) of each sample were performed.

The following table describes the sample collection and preparation regimen:

Table 7. Sample Collection and Preparation for Solubility Study

STEP	PROCEDURE	REPLICATES		DATE(S) PERFORMED
1	At 24, 48, and 72 hours, 3 test substance + urine, and 1 urine (method blank) centrifuge tubes per PFOS and FOSA are removed from the incubator and equilibrated at 23-24 °C for approximately 24 hours.	4 centrifuge tubes per day for PFOS and FOSA	Day1	04-24-01
			Day2	04-25-01
			Day3	04-26-01
2	Aliquots of the supernatant (urine containing dissolved test substance or urine only from the method blanks) were collected and dispensed into autovials.	4 per centrifuge tube per FOSA and PFOS	Day1	04-24-01
			Day2	04-25-01
			Day3	04-26-01
3a PFOS	The PFOS aliquots were diluted 100X with methanol (using a dual syringe diluter: 990 µL methanol, 10 µLsample), followed by a second dilution of 10X (900 µL methanol, 100 µLsample) for a final dilution factor of 1,000X. 17 µL THPFOS and PFBS solution at 15 µg/mL in methanol were then added to the final dilutions, and every fourth sample was spiked with 4 µL 25.05 µg/mL PFOS	Day 1		04-26-01
		Day 2		05-01-01
		Day 3		04-26-01
3b FOSA	The FOSA aliquots were diluted 10X with methanol (using a dual syringe diluter: 900 µL methanol, 100 µLsample), 17 µL THPFOS and PFBS solution at 15 µg/mL in methanol were then added to the final dilutions, and every fourth sample was spiked with 4 µL 24 µg/mL FOSA.	Day 1		04-26-01
		Day 2		04-26-01
		Day 3		05-01-01
4	Samples were analyzed via HPLC/ES/MS on HP 1100 LC/MSD	Day 1		04-26-01
		Day 2		PFOS: 05-01-01/FOSA: 04-26-01
		Day 3		PFOS: 04-30-01/FOSA: 05-01-01

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 2.5-1000 ng PFOS/mL. FOSA levels were evaluated versus calibration standards ranging in concentration from 2.5-1000 ng FOSA/mL. Internal Standard quantification was used to normalize the data. PFBS was chosen as the internal reference standard to quantify the data, THPFOS was added as an optional internal standard during preparation for analysis, but was not used to quantify the data. Target ions were 499 m/z (PFOS anion), 498 m/z (FOSA anion), 427 m/z (THPFOS anion), and 299 m/z (PFBS anion).

Analytical Equipment

Liquid Chromatograph: Hewlett-Packard® Series 1100 Liquid Chromatograph system
Analytical column: Keystone® Betasil™ C_{18} 2x50mm, 5µm particle size
Column temperature: Ambient
Cycle Time: 10 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:

Time	% C
0.00	15 %
0.50	15 %
0.80	60 %
3.50	100 %
6.50	100 %
7.00	15 %
10.00	Stop

Mass Spectrometer: Hewlett-Packard® Series 1100 API/Mass Spectrometer Detector
Software: HP ChemStation™ 6.0
Fragmentor Voltage: m/z 299=100 V; m/z 427=100 V; m/z 499= 140 V; m/z 498=80 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 0.980 or greater (R^2 *values ranged from 0.9993-0.9998*).
- **Calibration Standards.** Twelve standards ranging in concentration from approximately 2.5 to 1000 ng/mL PFOS/FOSA in methanol were used for the calibration curves. Calibration curves were run before and after every analytical sequence. Calibration standards were also evaluated for accuracy, and were consistently ≤ 30% difference from the expected value.
- **Lab Control Spikes.** 1 mL methanol was spiked with 4 µL 25.05 µg/mL PFOS spike solution, and another 1 mL was spiked with 4 µL 24 µg/mL FOSA spike solution, (the same 25 ppm spike solutions spiked into every fourth replicate for sample recoveries). The lab control sample was used to evaluate the recovery of the test substance in methanol only. Recoveries for PFOS ranged from 89-90%, and recoveries for FOSA ranged from 103-111% for Days 1,2, and 3.
- **Matrix Spikes.** Matrix spikes were prepared to demonstrate any possible matrix affects (i.e. signal suppression or enhancement) from the urine. Matrix spike recoveries for PFOS and FOSA samples were as follows: ± 30% with the following PFOS sample exceptions: 042301-PFOS-3.1.4MS (65%), 042301-PFOS-2.1.4 (52%), 042301-PFOS-2.2.4 (135%), 042301-PFOS-2.3.4 (53%). The MS samples from day 2 were done with a 5 uL syringe that seemed to have a loose plunger, therefore giving erratic results. Excluding the Day 2 PFOS spike recoveries may be disregarded. Overall, there appears to be no significant difference between Lab Control Spikes (test analyte spiked into methanol only) and Method Blank (test analyte spiked into urine only), or Sample Spikes (test analyte spiked into urine samples containing test analyte).
- **Sample Replicates.** Samples were within the 15% precision requirement.
- **Continuing Calibration Verification.** For quantitative determinations, a mid-level matrix calibration check was analyzed every ten samples to monitor instrumental drift, with a limit of ±30% deviation of the target concentrations.
- **Limit of Quantitation (LOQ).** The LOQ is equal to the lowest standard in the calibration curve. See tables 8a and 8b for the calibration curve ranges used in the study.

Table 8a. PFOS Calibration Standard Reprocessing Summary

Data Sub Batch, Analysis Date	Calibration Standards	Standards not used, reason for exclusion
Day 1 data:	PFOS: 5-501 ng/mL	2.5 & 1000 ng/mL since only 10 calibration levels may be reprocessed using Target data analysis software.
Day 2 data:	PFOS: 10-501 ng/mL	2.5 & 1000 ng/mL since only 10 calibration levels may be reprocessed in Target. 5 ng/mL std. was disabled because the 5 ng/mL CCVs did not meet the 30% accuracy requirement. (Data at the high end of the curve passed all criteria).
Day 3 data:	PFOS: 5-501 ng/mL	2.5 & 1000 ng/mL since only 10 calibration levels may be reprocessed in Target.

Table 8b. FOSA Calibration Standard Reprocessing Summary

DATA SUB BATCH, ANALYSIS DATE	CALIBRATION STANDARDS	STANDARDS NOT USED, REASON FOR EXCLUSION
Day 1 and 2 data:	FOSA: 10-499	2.5, & 1000 ng/mL were not used as calibrants since Target allows 10 levels only. The 5 ng/mL standard was disabled due to an elevated level of FOSA in some blank samples.
Day 3 data:	FOSA: 10-499	2.5, 5, & 1000 ng/mL standards were excluded/disabled to remain consistent with Day 1 and 2 data.

- **Blanks.**
  - Method blanks (urine 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). Method blank samples for Day 3 FOSA had a level of FOSA (20, 18, and 15 ng FOSA/mL on column) that was most likely due to FOSA dust in the analytical balance on the day of initial prep of the centrifuge tubes. Method blank samples from Days 1 and 2, a diluter blank, and internal standard blank ruled out all other possible forms of contamination. Therefore since it was determined that the FOSA was not in the urine prior to the sample preparation, this finding (i.e. FOSA in the day 3 method blank samples) is inconsequential.
  - 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 be applied to:
  - Pure substance. Purity of PFOS SD-018 has been determined to be 86.9%. The purity of FOSA SE-027 has yet to be determined. Since it is possible that impurities may exist in the FOSA, and the PFOS SD-018 is 86.9% pure, the reported solubility may be greater than that of a pure substance.
  - Substances that are stable in water. Hydrolytic studies have been conducted for PFOS TCR-00017-46 (primary standard material, Report # W1878) and FOSA (Project #EL-1132) at the 3M Environmental Laboratory showing stability.

DATA SUMMARY

Tables 9a and 9b summarize individual sample data. Representative chromatograms are presented in Attachment C. The table displays PFOS and FOSA concentrations for individual injections. Also included are the average concentrations for each Centrifuge Tube replicate, standard deviation, and % coefficient of variation.

Table 9a. PFOS SD-018 Solubility in Urine Data Summary

	Sample ID	SD-018 PFOS ug/mL Corrected for Purity	Average ug/mL Std Dev %Coefficient of Variation
Day 1 Centrifuge Tube #1	042301-PFOS-1.1.1 Smp	286	311
	042301-PFOS-1.1.2 Dup	325	22.0
	042301-PFOS-1.1.3 Trip	323	7%
Day 1 Centrifuge Tube #2	042301-PFOS-1.2.1 Smp	292	288
	042301-PFOS-1.2.2 Dup	289	4.0
	042301-PFOS-1.2.3 Trip	284	1%
Day 1 Centrifuge Tube #3	042301-PFOS-1.3.1 Smp	314	306
	042301-PFOS-1.3.2 Dup	301	7.2
	042301-PFOS-1.3.3 Trip	302	2%
Day 2 Centrifuge Tube #1	042301-PFOS-2.1.1 Smp	305	358
	042301-PFOS-2.1.2 Dup	412	54
	042301-PFOS-2.1.3 Trip	356	15%
Day 2 Centrifuge Tube #2	042301-PFOS-2.2.1 Smp	289	280
	042301-PFOS-2.2.2 Dup	288	15
	042301-PFOS-2.2.3 Trip	263	5%
Day 2 Centrifuge Tube #3	042301-PFOS-2.3.1 Smp	332	338
	042301-PFOS-2.3.2 Dup	338	7
	042301-PFOS-2.3.3 Trip	345	2%
Day 3 Centrifuge Tube #1	042301-PFOS-3.1.1 Smp	262	269
	042301-PFOS-3.1.2 Dup	287	15.4
	042301-PFOS-3.1.3 Trip	259	6%
Day 3 Centrifuge Tube #2	042301-PFOS-3.2.1 Smp	302	322
	042301-PFOS-3.2.2 Dup	352	26.5
	042301-PFOS-3.2.3 Trip	312	8%
Day 3 Centrifuge Tube #3	042301-PFOS-3.3.1 Smp	286	271
	042301-PFOS-3.3.2 Dup	257	14.5
	042301-PFOS-3.3.3 Trip	270	5%

Table 9b. FOSA SE-027 Solubility in Urine Data Summary

	Sample ID	SE-027 FOSA ug/mL Corrected for Purity	Average ug/mL Std Dev %Coefficient of Variation
Day 1 Centrifuge Tube #1	042301-FOSA-1.1.1 Smp	~~3.43~~	2.18
	042301-FOSA-1.1.2 Dup	2.23	4.6 % RPD
	042301-FOSA-1.1.3 Trip	2.13	(Rep 1 excluded from calc.)
Day 1 Centrifuge Tube #2	042301-FOSA-1.2.1 Smp	3.09	3.11
	042301-FOSA-1.2.2 Dup	3.13	0.0
	042301-FOSA-1.2.3 Trip	3.12	1%
Day 1 Centrifuge Tube #3	042301-FOSA-1.3.1 Smp	2.48	2.37
	042301-FOSA-1.3.2 Dup	2.33	0.1
	042301-FOSA-1.3.3 Trip	2.30	4%
Day 2 Centrifuge Tube #1	042301-FOSA-2.1.1 Smp	3.17	3.02
	042301-FOSA-2.1.2 Dup	2.96	0.1
	042301-FOSA-2.1.3 Trip	2.91	5%
Day 2 Centrifuge Tube #2	042301-FOSA-2.2.1 Smp	2.98	2.82
	042301-FOSA-2.2.2 Dup	2.80	0.1
	042301-FOSA-2.2.3 Trip	2.69	5%
Day 2 Centrifuge Tube #3	042301-FOSA-2.3.1 Smp	2.89	2.81
	042301-FOSA-2.3.2 Dup	2.79	0.1
	042301-FOSA-2.3.3 Trip	2.75	3%

Day 3 Centrifuge Tube #1	042301-FOSA-3.1.1, Smp	2.69	2.60
	042301-FOSA-3.1.2, Dup	2.51	0.1
	042301-FOSA-3.1.3, Trip	2.60	3%
Day 3 Centrifuge Tube #2	042301-FOSA-3.2.1, Smp	2.57	2.56
	042301-FOSA-3.2.2, Dup	2.61	0.1
	042301-FOSA-3.2.3, Trip	2.51	2%
Day 3 Centrifuge Tube #3	042301-FOSA-3.3.1, Smp	2.63	2.66
	042301-FOSA-3.3.2, Dup	2.74	0.1
	042301-FOSA-3.3.3, Trip	2.60	3%

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

Table 10a. Summary Table of Solubility of PFOS SD-018 in Urine.

Sampling Event	PFOS Solubility in Urine (Corrected for Purity)	Standard Deviation	% CV
Day 1	302 µg/mL	12	4%
Day 2	325 µg/mL	40	12%
Day 3	287 µg/mL	30	10%
Average Value	**305 µg/mL**	14	5%

The solubility of PFOS SD-018 in urine is 305 µg/mL at 23-24 °C.

Table 10b. Summary Table of Solubility of FOSA SE-027 in Urine.

Sampling Event	FOSA Solubility in Urine (Corrected for Purity)	Standard Deviation	% CV
Day 1	2.56 µg/mL	0.38	15%
Day 2	2.88 µg/mL	0.12	4%
Day 3	2.61 µg/mL	0.047	2%
Average Value	**2.68 µg/mL**	0.2	7%

The solubility of FOSA SE-027 at 23-24 °C is 2.68 µg/mL.

Attachment B contains data summary tables.

Statistical Methods

Statistical methods included calculating means and standard deviations. Outlier data points were excluded using Dixon's Q-Test. Refer to Attachment E for formulas and example calculations.

Statement of Conclusion

Under the conditions of the present study, the solubility of PFOS SD-018 in urine is 305 µg/mL at 23-24 °C. The solubility of FOSA SE-027 at 23-24 °C is 2.68 µg/mL.

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.

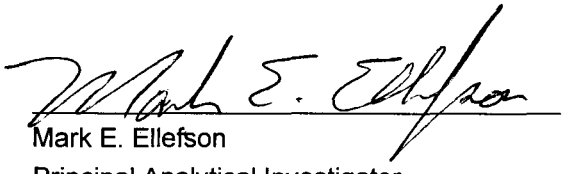
2. OECD 105: Water Solubility. Adopted 27July, 1995.

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

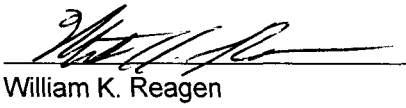
SIGNATURE PAGE

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

06/04/01
Date



William K. Reagen
Laboratory Manager

06/04/01
Date

ATTACHMENT A: EXTRACTION AND ANALYTICAL METHODS

3M ENVIRONMENTAL LABORATORY

SOLUBILITY SCREEN TEST: APPROXIMATE SOLUBILITY DETERMINATION OF A TEST SUBSTANCE IN VARIOUS SOLVENTS

Method Number: ETS-8-170.1 **Adoption Date: 09/08/00**

Effective Date: 03/14/01

Approved By:

William K. Reagen, Laboratory Manager — Date: 03/14/01

Mark E. Ellefson, Project Leader — Date: 03/14/01

1.0 SCOPE AND APPLICATION

1.1 Purpose. According to methods set forth by the United States Environmental Protection Agency (US EPA) and the Organization for the Economic Cooperation and Development (OECD) a preliminary study, which will be outlined in this method, serves as a prerequisite to performing solubility testing via the Column Elution Method and Shake Flask Methods. The EPA and OECD solubility testing guidelines state that if the preliminary solubility determination test indicates a solubility of $> 10^{-2}$ g/L (10 ppm), the Shake Flask Method (ETS-8-172.0) is to be used. If preliminary testing indicates a solubility of $< 10^{-2}$ g/L (10 ppm) the Column Elution Method (ETS-8-171.0) will be used to determine the solubility in the solvent of interest. Additionally, due to the tendency of certain compounds to be highly soluble in particular solvents, no further solubility determination will be necessary for compounds having solubility greater than 10% (mass/volume).

1.2 Compatible Analytes. Test substance and degradation products for solubility testing.

1.3 **Acceptable Matrices**. Aqueous (e.g. Milli-Q water, 0.01M $CaCl_2$), Acetone, Methanol, or other solvent(s) of interest.

2.0 SUMMARY OF METHOD

2.1 **Preliminary Solubility Determination in solvent.** Weigh approximately 10 mg of test substance into a 2.5 mL-4 mL glass screw-capped vial. Add test solvent in varying increments to the vial. After each solvent addition, vortex mix for approximately 15 sec., and sonicate 2-5 minutes. Make observations of the solution and visually confirm whether or not particles are present; it may be necessary to allow the solution to sit for up to 5 minutes. Add test solvent in a stepwise fashion up to 2 mL. If the substance fully dissolves after the first solvent addition of 100 µL, the test substance shall be considered highly soluble, and no further testing is required. If there are still undissolved particles in the vial, weigh 10 mg of test substance into a 100 mL glass stoppered volumetric flask or graduated cylinder. Add test solvent until visual confirmation of the substance dissolution has been achieved. Record all observations on a standardized preparation sheet or logbook. Once 100 mL solvent has been reached, the approximate concentration is at 100 µg/mL. If the solubility limit has not yet been achieved, weigh approximately 10 mg of test substance and transfer to a 1 L graduated cylinder/volumetric flask (concentration is approximately 10 ppm). The container should be sonicated and allowed to sit overnight to allow for maximal dissolution. If the undissolved particles are still observed after sitting > 12 hours, the column elution method will be utilized (ETS-8-171.0). If the test substance dissolves in 1 L or less of solvent, the shake flask method (ETS-8-172.0) will be used to determine the solubility.

3.0 DEFINITIONS

3.1 **Test substance.** A liquid or solid material for which the relative solubility in a specified solvent will be determined.

3.2 **Test solvent.** The matrix to which the test substance of interest is introduced. The test solvent may include but is not limited to aqueous matrices, including ASTM Type I Water and various salt matrices (e.g. 0.01M $CaCl_{2\,(aq)}$); and organic solvent matrices, including methanol, and acetone.

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 should be triple rinsed with acetone and methanol to reduce the possibility of accidental contamination.

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 screening procedure.

6.0 EQUIPMENT

6.1 Analytical balance sensitive to 0.1 mg.

6.2 Vortex mixer.

6.3 Sonicating device.

7.0 SUPPLIES AND MATERIALS

7.1 Disposable glass graduated pipettes, 1 mL to 100 mL.

7.2 Disposable glass Pasteur pipettes and rubber bulbs.

7.3 Glass beakers, various sizes.

7.4 2.5 mL-4 mL glass screw-top vial.

7.5 Glass volumetric flasks, 10 mL to 1000 mL.

7.6 10 µL-1000 µL Pipetteman™ manual pipettor and plastic pipette tips, or equivalent.

8.0 REAGENTS AND STANDARDS

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 I, or equivalent.

8.4 **Calcium Chloride Dihydrate,** Approximately 99% or better, from Sigma or equivalent.

8.5 **0.01 M $CaCl_2$,** A 0.01 M $CaCl_2$ stock solution is prepared by weighing 1.5 g $CaCl_2$ Dihydrate in a weigh boat and transferring to a 1 L volumetric flask and diluting to the mark with Milli-Q™ water.

8.6 **Test substance of known purity.**

9.0 SAMPLE HANDLING

9.1 Record times of initial preparation and dilution on a sample preparation sheet or logbook.

9.2 Once the preliminary testing has been completed and the appropriate information is extracted, the screen test samples should be disposed into the proper waste stream.

10.0 QUALITY CONTROL

10.1 Not applicable.

11.0 CALIBRATION AND STANDARDIZATION

11.1 The compounds of interest must be of known characterization according to laboratory specifications.

11.2 All equipment used, such as the analytical balance, should be calibrated daily prior to use.

12.0 PROCEDURES

12.1 Solubility screen.

12.1.1 Weigh 10 mg ± 1 mg of test substance into a 4 mL glass screw-top vial or equivalent. Record the weight (refer to attachment A for an example of a standardized prep sheet).

12.1.2 Add solvent (e.g. water, methanol, acetone) according to the table below to the test substance.

4 mL vial or equivalent	Solvent addition 1	Solvent addition 2	Solvent addition 3	Solvent addition 4
Total volume solvent added (mL)…	0.1	0.5	1	2
Approximate Solubility (µg/mL)…	100000	20000	10000	5000

12.1.3 Following addition of solvent, vortex mix approximately 15 seconds, sonicate 2-5 minutes. Visually check for undissolved particles. If undissolved test substance remains, continue adding solvent according to the above chart. It may be necessary to allow the solution to settle for up to 5 minutes before making observations. Record observations on the standardized prep sheet.

12.1.4 If all particles dissolve, then estimate the approximate solubility and document the concentration. Because the concentration is >10 µg/mL, the shake flask method (ETS-8-172.0) will be used to determine the accurate solubility point. However, if the test substance dissolves after the *first* solvent addition of 100 µL the solubility is determined to be >10% (mass/volume). The test substance shall be regarded as "highly soluble," and no further testing is required.

12.1.5 If the test substance did not dissolve in the 2 mL of solvent, weigh 10 mg ± 1 mg of test substance into a 100 mL glass volumetric flask or equivalent. Record the weight (refer to attachment A for an example of a standardized prep sheet).

12.1.6 Add solvent according to the table below to the test substance to deliver the appropriate amount of solvent. As described in 12.1.3, vortex mix, sonicate, and allow the test solution to settle. Record all observations and procedures on the preparation sheet.

100 mL volumetric flask or equivalent	Solvent addition 1	Solvent addition 2	Solvent addition 3
Total volume solvent added (mL)...	10	50	100
Approximate Solubility (µg/mL)...	1000	200	100

12.1.7 If the solubility point is reached at or prior to the complete addition of 100 mL, the concentration estimation at which the substance is soluble should be documented and shall be used as a starting point for the shake flask method (ETS-8-172.0).

12.1.8 If the 100 mL solvent level has been reached without complete dissolution of test substance, weigh out 10 mg ± 1 mg test substance and transfer to a 1 L volumetric flask, graduated cylinder, or equivalent. Dilute to 1 L with test solvent. The approximate concentration of test substance is at 10 µg/mL. The flask should be allowed to sit 12-24 hours to allow for maximal dissolution. If the undissolved particles are still observed, the column elution method will be utilized (ETS-8-171.0). If no visible particulates are observed, the shake flask method (ETS-8-172.0) shall be used to determine the solubility.

13.0 Data Analysis and Calculations

13.1 The solubility determination in this method is qualitative/semi-quantitative.

13.2 The point to which the substance dissolves in solvent is confirmed visually. The solubility point is therefore a qualitative determination.

13.3 The concentration estimation is semi-quantitative in that the approximate concentration is calculated by the following equation:

$c = a/b$

Where:

c= the semi-quantitative concentration µg/mL,

a= amount of substance weighed out (µg), and

b= the approximate amount of solvent added (mL).

14.0 Method Performance

14.1 Limitation of data. The accuracy to which the solubility is determined is subject to a large margin of error due to the way in which the solvent is added to the solute. Since large and varying increments of solvent are being added to the solute, this error margin must be considered when reporting the concentration estimation.

14.2 The data obtained through this study is an estimation only and should be treated as a qualitative estimation of the solubility of test substance in a given solvent.

14.3 For an accurate measurement of the substance solubility concentration, the shake flask method, or column elution method will be used.

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.

17.0 ATTACHMENTS

17.1 **Attachment A**-Example of a Standardized Sample Preparation Sheet: Solubility Screen Prep Sheet.

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 3M Environmental Laboratory Method ETS-8-171.0, "Column Elution Method: Solubility Determination of Test Substance in Various Solvents."

18.5 3M Environmental Laboratory Method ETS-8-172.0, "Shake Flask Method: Solubility Determination of Test Substance in Various Solvents."

19.0 AFFECTED DOCUMENTS

19.1 ETS-8-171.0, "Column Elution Method: Solubility Determination of Test Substance in Various Solvents."

19.2 ETS-8-172.0, "Shake Flask Method: Solubility Determination of Test Substance in Various Solvents."

20.0 REVISIONS

Revision Number	Reason For Revision	Revision Date
1	It was desirable to make the method more universally applicable by removing references to specific test substances.	03/13/01

Attachment A: Example Preparation Worksheet, page 1 of 2

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: _____ ***Sample preparation worksheet***

Test Substance: _____ **ID#:** _____ **Date:** _____

Source: _____

Solvent: _____ **ID#:** _____

Source: _____

Step

1 *Weigh approximately 10 mg ± 1 mg of test substance into a 2.5 mL-4 mL glass screw-top vial.*

Weight of test substance: _____ mg Balance ID: _____ Date/Initials: _____

2nd replicate optional: _____ mg Thermometer ID: _____

2 *Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.*

Solubility data	volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst _____ Date/Time ____/____ Total volume solvent added (mL) Approximate Solubility (ug/mL)	**Step 2-1** 0.1 100,000	Flask vortex-mixed? Yes/No Room Temp ____°C Flask sonicated? Yes/No Time ____min Flask allowed to settle? Yes/No Time ____min	Is there solute still present? ~~Yes~~ continue to step 2-2 **NO-** The solution is at 10%, and is considered "infinitely soluble." No further solubility testing is required.
Analyst _____ Date/Time ____/____ Total volume solvent added (mL) Approximate Solubility (ug/mL)	**Step 2-2** 0.5 20,000	Flask vortex-mixed? Yes/No Room Temp ____°C Flask sonicated? Yes/No Time ____min Flask allowed to settle? Yes/No Time ____min	Is there solute still present? Yes/No
Analyst _____ Date/Time ____/____ Total volume solvent added (mL) Approximate Solubility (ug/mL)	**Step 2-3** 1. 10,000	Flask vortex-mixed? Yes/No Room Temp ____°C Flask sonicated? Yes/No Time ____min Flask allowed to settle? Yes/No Time ____min	Is there solute still present? Yes/No
Analyst _____ Date/Time ____/____ Total volume solvent added (mL) Approximate Solubility (ug/mL)	**Step 2-4** 2 5,000	Flask vortex-mixed? Yes/No Room Temp ____°C Flask sonicated? Yes/No Time ____min Flask allowed to settle? Yes/No Time ____min	Is there solute still present? Yes/No

Conclusions:

Did the substance dissolve in 2 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: _____ ug/ml

The solubility of the test substance is to be determined via the shake flask method.

or **No** Continue to page two for further testing.

Notes/ additional comments:

Attachment A Cont.: Example Preparation Worksheet, page 2 of 2

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: _____ ***Sample preparation worksheet***

Test Substance: _____ **ID#:** _____ **Date:** _____

Source: _____

Solvent: _____ **ID#:** _____

Source: _____

Step (continued from page 1)

*****Did the test substance dissolve in 2 mL? Yes/No If YES, the test is complete, and there is no need to continue with the screen test. If NO, continue to step 3 to determine the approximate solubility of the substance.**

3 *Weigh approximately 10 mg ± 1 mg of test substance into a 100 mL graduated stoppered cylinder/volumetric flask.*

Weight of test substance: _____mg Balance ID: _____. Date/Initials: _____

2nd replicate optional: _____mg Thermometer ID: _____.

4 *Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.*

Solubility data	volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst Date/Time____/____	**Step 4-1**	Flask vortex-mixed? Yes/No Room Temp____°C	Is there solute still present? Yes/No
____ Total volume solvent added (mL)...	10	Flask sonicated? Yes/No Time____min	
Approximate Solubility (ug/mL)...	1,000	Flask allowed to settle? Yes/No Time____min	
Analyst Date/Time____/____	**Step 4-2**	Flask vortex-mixed? Yes/No Room Temp____°C	Is there solute still present? Yes/No
____ Total volume solvent added (mL)...	50	Flask sonicated? Yes/No Time____min	
Approximate Solubility (ug/mL)...	200	Flask allowed to settle? Yes/No Time____min	
Analyst Date/Time____/____	**Step 4-3**	Flask vortex-mixed? Yes/No Room Temp____°C	Is there solute still present? Yes/No
____ Total volume solvent added (mL)...	100	Flask sonicated? Yes/No Time____min	
Approximate Solubility (ug/mL)...	100	Flask allowed to settle? Yes/No Time____min	

5 *Weigh approximately 10 mg ± 0.5 mg of test substance into a 1 L graduated stoppered cylinder/volumetric flask/or equivalent.*

Weight of test substance: _____mg Balance ID: _____. Date/Initials: _____

Analyst Date/Time____/____	**Step 5-1**	Flask vortex-mixed? Yes/No Room Temp____°C	Is there solute still present? Yes/No
____ Total volume solvent added (mL)...	1000	Flask sonicated? Yes/No Time____min	
Approximate Solubility (ug/mL)...	10	Flask allowed to settle? Yes/No Time____min	

Conclusions:

Did the substance dissolve in 1000 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: _____ug/ml

The solubility of the test substance is to be determined via the shake flask method.

or **No** -The solubility of the test substance is to be determined via the column elution method.

Notes/ additional comments:

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 — Date: 03/27/01

Mark E. Ellefson, Project Lead — Date: 03/27/01

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.

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 °C ± 2 °C, shaking for approximately 24 hours. At approximately 24 hours, the first time point will be pulled and equilibrated at room temperature (about 20 °C-26 °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 ± 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 $CaCl_2$ solution,** Example: A 0.01 M $CaCl_2$ stock solution is prepared by weighing 1.4 g $CaCl_2$ in a weigh boat and transferring to a 1 L volumetric flask and diluting to the mark with Milli-Q™ 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 °C ± 2 °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 20-26 °C (record the actual temperature) for about a 24 hour period.

12.2.5 After about 24 hours of equilibration at 20 °C-26 °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:

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

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

A/B X 100 = Sample %RSD

where: A= standard deviation of averaged samples
B= average of samples

Sample RPD will be calculated using the following equation:

$|A-B|/((A+B)/2)$= 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).

$Q_{observed}$= gap/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

Revision Number	Reason For Revision	Revision Date
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 #_____

Temperature of Study:_____°C ***Sample preparation worksheet*** **Analyst(s):** _____

Test Substance: _____ **ID#:** _____ **Date:** _____

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#: "sampleID" = _____	Sample Description	weight test substance added:	Total mass (solute + solvent) added:	[1]Internal Standard addition _____uL/_____mls soln IS added to dilution factor:	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

Test vessels placed on orbital incubator? Y/N Incubator ID:_____ Speed:_____ rpm Initial Temperature: _____°C

[1]Internal Standard:_____ ID#: _____

Int. Std. Conc.: _____ ug/ml

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 — Date: 11/10/00

Group Leader — Date: 11/10/00

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 µm 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 µL injection

12.1.1.2 Flow rate = 300 µL/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:	Time (min.)	% B
	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 µL/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:

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

13.1.2 Calculate percent difference using the following equation:

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

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

On-Column Concentration (µg/mL) x Dilution Factors = 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 Sprectrometer".

19.0 AFFECTED DOCUMENTS

19.1 None

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

ATTACHMENT B: DATA SUMMARY TABLES

ATTACHMENT B: DATA SUMMARY TABLES

Solubility Study

Test Substance: PFOS, SD-018 (purity = 86.9%); and FOSA, SE-027 (purity TBD)
Test Matrix: Male/Female Human Urine
Test Temperature: 23-24 °C

Method(s) used: The solubility determination was performed according to United States Environmental Protection Agency OPPTS 830.7840, "Water Solubility: Column Elution Method; Shake Flask Method" and OECD 105, "Water Solubility" Guidelines, as implemented by 3M Laboratory in methods ETS-8-170.1 "Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents;" and ETS-8-172.1 "Shake Flask Method; Solubility Determination of a Test Substance in Various Solvents". All analyses were performed via LC/MS according to the method ETS-8-155.0 "Analysis of Perfluorooctane Sulfonate or Other Fluorochemicals in Waste Stream or Water Extracts Using HPLC-Electrospray/Mass Spectrometry".

Results:

PFOS	Average µg/mL	Std Dev	Precision (%RSD)
Day 1	302	12	4%
Day 2	325	40	12%
Day 3	287	30	10%
Total	**305**	**14.2**	**5%**

FOSA	Average µg/mL	Std Dev	Precision (%RSD)
Day 1	2.56	0.38	15%
Day 2	2.88	0.12	4%
Day 3	2.61	0.047	2%
Total	**2.68**	**0.2**	**7%**

Results for PFOS were corrected for standard purity.
Purity for FOSA is unknown, therefore no corrections were made.

KLT 05/14/01

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Exact Copy of Original

KLT Initial

05-15-01 Date

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Project # E01-0768

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FOSA SE-027 Urine Solubility Results

SampType	File	Sample Name	Sample Desc.	Compound Name	RT	Area	Amount (ng/mL)	Corrected for 1:10 dilution µg/mL FOSA	Caluculations Average (µg/mL) Std Dev % RSD Spike % Recov.		
SAMPLE	HILL0126.D	~~FOSA-1.1.1~~	042301-FOSA-1.1.1 Smp	~~FOSA~~	~~5.89~~	~~9090670~~	~~343~~	~~3.43~~	2.60	2.18	
SAMPLE	HILL0127.D	FOSA-1.1.2	042301-FOSA-1.1.2 Dup	FOSA	5.89	6180725	223	2.23	0.7	4.4%	
SAMPLE	HILL0128.D	FOSA-1.1.3	042301-FOSA-1.1.3 Trip	FOSA	5.886	5838914	213	2.13	28%	Rep2,3 ave&RPD	
SAMPLE	HILL0130.D	FOSA-1.2.1	042301-FOSA-1.2.1 Smp	FOSA	5.889	8385467	309	3.09	3.11		Day 1 results, µg/mL
SAMPLE	HILL0135.D	FOSA-1.2.2	042301-FOSA-1.2.2 Dup	FOSA	5.885	8541720	313	3.13	0.0		average 2.56
SAMPLE	HILL0136.D	FOSA-1.2.3	042301-FOSA-1.2.3 Trip	FOSA	5.888	8450504	312	3.12	1%		std dev 0.38
SAMPLE	HILL0138.D	FOSA-1.3.1	042301-FOSA-1.3.1 Smp	FOSA	5.882	6883587	248	2.48	2.37		%RSD: 15%
SAMPLE	HILL0139.D	FOSA-1.3.2	042301-FOSA-1.3.2 Dup	FOSA	5.886	6433005	233	2.33	0.1		
SAMPLE	HILL0141.D	FOSA-1.3.3	042301-FOSA-1.3.3 Trip	FOSA	5.886	6255010	230	2.30	4%		
	from HPLC/MSD "Hillary" analytical sequence H010426.s, reprocessed with Target Software by KLT										
SAMPLE	HILL0152.D	FOSA-2.1.1	042301-FOSA-2.1.1 Smp	FOSA	5.879	8103858	317	3.17	3.02		
SAMPLE	HILL0153.D	FOSA-2.1.2	042301-FOSA-2.1.2 Dup	FOSA	5.879	8151376	296	2.96	0.1		
SAMPLE	HILL0154.D	FOSA-2.1.3	042301-FOSA-2.1.3 Trip	FOSA	5.88	7853370	291	2.91	5%		
SAMPLE	HILL0157.D	FOSA-2.2.1	042301-FOSA-2.2.1 Smp	FOSA	5.887	7993752	298	2.98	2.82		Day 2 results, µg/mL
SAMPLE	HILL0158.D	FOSA-2.2.2	042301-FOSA-2.2.2 Dup	FOSA	5.886	7697039	280	2.80	0.1		average 2.88
SAMPLE	HILL0159.D	FOSA-2.2.3	042301-FOSA-2.2.3 Trip	FOSA	5.887	7443513	269	2.69	5%		std dev 0.12
SAMPLE	HILL0165.D	FOSA-2.3.1	042301-FOSA-2.3.1 Smp	FOSA	5.879	7850050	289	2.89	2.81		%RSD: 4%
SAMPLE	HILL0166.D	FOSA-2.3.2	042301-FOSA-2.3.2 Dup	FOSA	5.882	7536620	279	2.79	0.1		
SAMPLE	HILL0167.D	FOSA-2.3.3	042301-FOSA-2.3.3 Trip	FOSA	5.88	7535404	275	2.75	3%		
	from HPLC/MSD "Hillary" analytical sequence H010426.s, reprocessed with Target Software by KLT										
SAMPLE	HILL0097.D	FOSA-3.1.1	042301-FOSA-3.1.1, Smp	FOSA	5.909	7201703	269	2.69	2.60		
SAMPLE	HILL0099.D	FOSA-3.1.2	042301-FOSA-3.1.2, Dup	FOSA	5.901	6731194	251	2.51	0.1		
SAMPLE	HILL0100.D	FOSA-3.1.3	042301-FOSA-3.1.3, Trip	FOSA	5.908	6998562	260	2.60	3%		
SAMPLE	HILL0101.D	FOSA-3.2.1	042301-FOSA-3.2.1, Smp	FOSA	5.908	6990791	257	2.57	2.56		Day 3 results, µg/mL
SAMPLE	HILL0102.D	FOSA-3.2.2	042301-FOSA-3.2.2, Dup	FOSA	5.922	7009317	261	2.61	0.1		average 2.61
SAMPLE	HILL0103.D	FOSA-3.2.3	042301-FOSA-3.2.3, Trip	FOSA	5.895	6836606	251	2.51	2%		std dev 0.05
SAMPLE	HILL0108.D	FOSA-3.3.1	042301-FOSA-3.3.1, Smp	FOSA	5.916	7065688	263	2.63	2.66		%RSD: 2%
SAMPLE	HILL0109.D	FOSA-3.3.2	042301-FOSA-3.3.2, Dup	FOSA	5.906	7377514	274	2.74	0.1		
SAMPLE	HILL0110.D	FOSA-3.3.3	042301-FOSA-3.3.3, Trip	FOSA	5.9	7030743	260	2.60	3%		
	from HPLC/MSD "Hillary" analytical sequence H010501.s, reprocessed with Target Software by KLT										

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3M Environmental Laboratory
Project # E01-0768

5-29-01

KLT 05-14-01

BACK TO MAIN

PFOS SD-018 Urine Solubility Results

Corrected for 1:1,000 dilution

Sample Name	Misc Info	Compound Name			RT	Area	Amt (ng/mL)	Corrected for std purity ng/mL	Average µg/mL Std Dev %RSD
SAMPLE	HILL0037.D	PFOS-1.1.1	042301-PFOS-1.1.1 Smp	PFOS	5.271	7570273	292.6	286	311
SAMPLE	HILL0038.D	PFOS-1.1.2	042301-PFOS-1.1.2 Dup	PFOS	5.277	8619490	331.8	325	22.0
SAMPLE	HILL0039.D	PFOS-1.1.3	042301-PFOS-1.1.3 Trip	PFOS	5.255	8589480	329.8	323	7%
SAMPLE	HILL0041.D	PFOS-1.2.1	042301-PFOS-1.2.1 Smp	PFOS	5.251	7600633	298.7	292	288
SAMPLE	HILL0046.D	PFOS-1.2.2	042301-PFOS-1.2.2 Dup	PFOS	5.263	7571396	294.9	289	4.0
SAMPLE	HILL0047.D	PFOS-1.2.3	042301-PFOS-1.2.3 Trip	PFOS	5.264	7594271	290.2	284	1%
SAMPLE	HILL0049.D	PFOS-1.3.1	042301-PFOS-1.3.1 Smp	PFOS	5.27	8267658	320.6	314	306
SAMPLE	HILL0051.D	PFOS-1.3.2	042301-PFOS-1.3.2 Dup	PFOS	5.263	8045600	307.6	301	7.2
SAMPLE	HILL0052.D	PFOS-1.3.3	042301-PFOS-1.3.3 Trip	PFOS	5.272	8019673	308.5	302	2%
	from HPLC/MSD "Hillary" analytical sequence H010426.s, reprocessed with Target Software by KLT								
SAMPLE	HILL0037.D	PFOS-2.1.1	042301-PFOS-2.1.1 Smp	PFOS	5.248	7420988	312	305	358
SAMPLE	HILL0038.D	PFOS-2.1.2	042301-PFOS-2.1.2 Dup	PFOS	5.248	9885661	421	412	54
SAMPLE	HILL0039.D	PFOS-2.1.3	042301-PFOS-2.1.3 Trip	PFOS	5.256	8832844	363	356	15%
SAMPLE	HILL0045.D	PFOS-2.2.1	042301-PFOS-2.2.1 Smp	PFOS	5.263	7021826	295	289	280
SAMPLE	HILL0046.D	PFOS-2.2.2	042301-PFOS-2.2.2 Dup	PFOS	5.255	7094280	294	288	15
SAMPLE	HILL0047.D	PFOS-2.2.3	042301-PFOS-2.2.3 Trip	PFOS	5.25	6410858	269	263	5%
SAMPLE	HILL0050.D	PFOS-2.3.1	042301-PFOS-2.3.1 Smp	PFOS	5.257	8177762	339	332	338
SAMPLE	HILL0051.D	PFOS-2.3.2	042301-PFOS-2.3.2 Dup	PFOS	5.249	8319423	345	338	7
SAMPLE	HILL0052.D	PFOS-2.3.3	042301-PFOS-2.3.3 Trip	PFOS	5.249	8547947	352	345	2%
	from HPLC/MSD "Hillary" analytical sequence H010501.s, reprocessed with Target Software by KLT								
SAMPLE	HILL0064.D	PFOS-3.1.1	042301-PFOS-3.1.1 Smp	PFOS	5.237	6815087	267.6	262	269
SAMPLE	HILL0065.D	PFOS-3.1.2	042301-PFOS-3.1.2 Dup	PFOS	5.242	7544482	292.7	287	15.4
SAMPLE	HILL0066.D	PFOS-3.1.3	042301-PFOS-3.1.3 Trip	PFOS	5.242	6794540	265.0	259	6%
SAMPLE	HILL0072.D	PFOS-3.2.1	042301-PFOS-3.2.1 Smp	PFOS	5.245	8117378	308.4	302	322
SAMPLE	HILL0073.D	PFOS-3.2.2	042301-PFOS-3.2.2 Dup	PFOS	5.249	8786445	359.3	352	26.5
SAMPLE	HILL0074.D	PFOS-3.2.3	042301-PFOS-3.2.3 Trip	PFOS	5.251	9095599	319.1	312	8%
SAMPLE	HILL0077.D	PFOS-3.3.1	042301-PFOS-3.3.1 Smp	PFOS	5.238	7558687	292.4	286	271
SAMPLE	HILL0078.D	PFOS-3.3.2	042301-PFOS-3.3.2 Dup	PFOS	5.243	6689145	262.7	257	14.5
SAMPLE	HILL0079.D	PFOS-3.3.3	042301-PFOS-3.3.3 Trip	PFOS	5.234	7103605	275.7	270	5%
	from HPLC/MSD "Hillary" analytical sequence H010430.s, reprocessed with Target Software by KLT								

Day 1 results, µg/mL
average 302
std dev 12
%RSD: 4.0%

Day 2 results, µg/mL
average 325
std dev 40
%RSD: 12%

Day 3 results, µg/mL
average 287
std dev 30
%RSD: 10%

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KLT 05/15/01
Initial Date

KLT 05/14/01

BACK TO MAIN

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Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: *H010426.s/PFOS0425.m (ChemStation); Reprocessing Method: H010426t.m (Target)*

Inj Date	Batch	SampType	File	Sample Name	Sample Desc.	Compound Name	RT	Area	Amount	Compound Name	RT	Area	Amount	
4/26/01 19:37	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0016.D	01003-07-01	0 ng/mL PFOS	THPFOS	5.115	1238088	254	PFBS	4.373	1933641	254	
4/26/01 19:48	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0017.D	01003-07-02	2.5 ng/mL PFOS	THPFOS	5.11	1251680	254	PFBS	4.382	1933710	254	
4/26/01 19:59	D:\chem\Hillary.i\H010426.b	CALIB_1	HILL0018.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.125	1273626	254	PFBS	4.383	1932682	254	
4/26/01 20:10	D:\chem\Hillary.i\H010426.b	CALIB_2	HILL0019.D	01003-07-04	10 ng/mL PFOS	THPFOS	5.112	1280488	254	PFBS	4.39	1943358	254	
4/26/01 20:21	D:\chem\Hillary.i\H010426.b	CALIB_3	HILL0020.D	01003-07-05	25 ng/mL PFOS	THPFOS	5.097	1289080	254	PFBS	4.383	1949841	254	
4/26/01 20:33	D:\chem\Hillary.i\H010426.b	CALIB_4	HILL0021.D	01003-07-06	40 ng/mL PFOS	THPFOS	5.115	1294694	254	PFBS	4.38	1941643	254	
4/26/01 20:44	D:\chem\Hillary.i\H010426.b	CALIB_5	HILL0022.D	01003-07-07	50.1 ng/mL PFOS	THPFOS	5.094	1319187	254	PFBS	4.38	1930302	254	
4/26/01 20:55	D:\chem\Hillary.i\H010426.b	CALIB_6	HILL0023.D	01003-07-08	75.1 ng/mL PFOS	THPFOS	5.131	1320686	254	PFBS	4.367	1953486	254	
4/26/01 21:06	D:\chem\Hillary.i\H010426.b	CALIB_7	HILL0024.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.097	1316830	254	PFBS	4.376	1971932	254	
4/26/01 21:17	D:\chem\Hillary.i\H010426.b	CALIB_8	HILL0025.D	01003-07-10	250.5 ng/mL PFOS	THPFOS	5.117	1333532	254	PFBS	4.375	1956172	254	
4/26/01 21:28	D:\chem\Hillary.i\H010426.b	CALIB_9	HILL0026.D	01003-07-11	400.8 ng/mL PFOS	THPFOS	5.11	1326197	254	PFBS	4.382	1987259	254	
4/26/01 21:40	D:\chem\Hillary.i\H010426.b	CALIB_10	HILL0027.D	01003-07-12	501 ng/mL PFOS	THPFOS	5.11	1324859	254	PFBS	4.389	1977603	254	
4/26/01 21:51	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0028.D	01003-07-13	1002 ng/mL PFOS	THPFOS	5.109	1323984	254	PFBS	4.374	1983865	254	
4/27/01 0:28	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0042.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.111	1428715	254	PFBS	4.397	2021892	254	
4/27/01 0:39	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0043.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.118	1441029	254	PFBS	4.401	2000887	254	
4/27/01 3:05	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0056.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.141	1487926	254	PFBS	4.405	2033053	254	
4/27/01 3:17	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0057.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.125	1456794	254	PFBS	4.397	2045778	254	
4/27/01 5:53	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0071.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.122	1499176	254	PFBS	4.407	2055427	254	
4/27/01 6:04	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0072.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.133	1477095	254	PFBS	4.411	2044996	254	
4/27/01 7:34	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0080.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.133	1504963	254	PFBS	4.411	2044088	254	
4/27/01 7:45	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0081.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.13	1469125	254	PFBS	4.408	2044316	254	
4/27/01 8:19	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0084.D	01003-07-01	0 ng/mL PFOS	THPFOS	5.127	1491338	254	PFBS	4.412	2052700	254	
4/27/01 8:30	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0085.D	01003-07-02	2.5 ng/mL PFOS	THPFOS	5.13	1484350	254	PFBS	4.409	2038788	254	
4/27/01 8:41	D:\chem\Hillary.i\H010426.b	CALIB_11	HILL0086.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.123	1517040	254	PFBS	4.408	2035215	254	
4/27/01 8:52	D:\chem\Hillary.i\H010426.b	CALIB_12	HILL0087.D	01003-07-04	10 ng/mL PFOS	THPFOS	5.13	1493288	254	PFBS	4.409	2007995	254	
4/27/01 9:03	D:\chem\Hillary.i\H010426.b	CALIB_13	HILL0088.D	01003-07-05	25 ng/mL PFOS	THPFOS	5.123	1476860	254	PFBS	4.409	2020150	254	
4/27/01 9:15	D:\chem\Hillary.i\H010426.b	CALIB_14	HILL0089.D	01003-07-06	40 ng/mL PFOS	THPFOS	5.124	1492539	254	PFBS	4.41	2027822	254	
4/27/01 9:26	D:\chem\Hillary.i\H010426.b	CALIB_15	HILL0090.D	01003-07-07	50.1 ng/mL PFOS	THPFOS	5.13	1489446	254	PFBS	4.416	2029826	254	
4/27/01 9:37	D:\chem\Hillary.i\H010426.b	CALIB_16	HILL0091.D	01003-07-08	75.1 ng/mL PFOS	THPFOS	5.124	1438972	254	PFBS	4.409	1952364	254	
4/27/01 9:48	D:\chem\Hillary.i\H010426.b	CALIB_17	HILL0092.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.122	1481229	254	PFBS	4.407	2023371	254	
4/27/01 9:59	D:\chem\Hillary.i\H010426.b	CALIB_18	HILL0093.D	01003-07-10	250.5 ng/mL PFOS	THPFOS	5.123	1455720	254	PFBS	4.409	1982961	254	
4/27/01 10:10	D:\chem\Hillary.i\H010426.b	CALIB_19	HILL0094.D	01003-07-11	400.8 ng/mL PFOS	THPFOS	5.123	1457008	254	PFBS	4.402	2031354	254	
4/27/01 10:22	D:\chem\Hillary.i\H010426.b	CALIB_20	HILL0095.D	01003-07-12	501 ng/mL PFOS	THPFOS	5.132	1455144	254	PFBS	4.41	2025464	254	
4/27/01 10:33	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0096.D	01003-07-13	1002 ng/mL PFOS	THPFOS	5.123	1426062	254	PFBS	4.402	1994320	254	
Two curves were averaged using Target software to quantify data. A standard curve range of only 10 levels is allowed. 5-500 ng/mL was the range chosen.								1406375				1997302	2596492	+30% response
								88296				41289	1398111	-30% response
								6.3%				2.1%		

Inj Date	Batch	SampType	File	Sample Name	Sample Desc.	Compound Name	RT	Area	Amount	Compound Name	RT	Area	Amount
4/26/01 22:24	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0031.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	THPFOS	5.105	1356608	254	PFBS	4.377	1932182	254
4/26/01 22:36	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0032.D	PFOS-1.4.1blk	042301-PFOS-1.4.1 Mtd Blk	THPFOS	5.112	1357667	254	PFBS	4.37	1900574	254
4/26/01 22:47	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0033.D	PFOS-1.4.2blk	042301-PFOS-1.4.2 Mtd Blk	THPFOS	5.111	1371641	254	PFBS	4.369	1910506	254
4/26/01 22:58	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0034.D	PFOS-1.4.3blk	042301-PFOS-1.4.3 Mtd Blk	THPFOS	5.117	1347413	254	PFBS	4.374	1921866	254
4/26/01 23:09	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0035.D	PFOS-1.4.4blkMS	042301-PFOS-1.4.4 Mtd Blk Spike	THPFOS	5.137	1362229	254	PFBS	4.388	1925821	254
4/26/01 23:32	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0037.D	PFOS-1.1.1	042301-PFOS-1.1.1 Smp	THPFOS	5.123	1239933	254	PFBS	4.388	2179012	254
4/26/01 23:43	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0038.D	PFOS-1.1.2	042301-PFOS-1.1.2 Dup	THPFOS	5.123	1242749	254	PFBS	4.374	2222026	254
4/26/01 23:54	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0039.D	PFOS-1.1.3	042301-PFOS-1.1.3 Trip	THPFOS	5.115	1244758	254	PFBS	4.393	2225836	254
4/27/01 0:05	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0040.D	PFOS-1.1.4MS	042301-PFOS-1.1.4 Spike	THPFOS	5.117	1243279	254	PFBS	4.395	2165910	254
4/27/01 0:16	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0041.D	PFOS-1.2.1	042301-PFOS-1.2.1 Smp	THPFOS	5.111	1262274	254	PFBS	4.397	2148762	254
4/27/01 1:12	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0046.D	PFOS-1.2.2	042301-PFOS-1.2.2 Dup	THPFOS	5.123	1279599	254	PFBS	4.388	2164574	254
4/27/01 1:24	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0047.D	PFOS-1.2.3	042301-PFOS-1.2.3 Trip	THPFOS	5.124	1288849	254	PFBS	4.395	2202302	254
4/27/01 1:35	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0048.D	PFOS-1.2.4MS	042301-PFOS-1.2.4 Spike	THPFOS	5.115	1283560	254	PFBS	4.394	2190236	254

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05-16-01 p.1065
Date

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Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/PFOS0425.m (ChemStation); Reprocessing Method: H010426t.m (Target)

4/27/01 1:47	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0049.D	PFOS-1.3.1	042301-PFOS-1.3.1 Smp	THPFOS	5.13	1269547	254	PFBS	4.394	2195975	254
4/27/01 2:09	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0051.D	PFOS-1.3.2	042301-PFOS-1.3.2 Dup	THPFOS	5.116	1282405	254	PFBS	4.394	2216090	254
4/27/01 2:20	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0052.D	PFOS-1.3.3	042301-PFOS-1.3.3 Trip	THPFOS	5.132	1283479	254	PFBS	4.396	2203291	254
4/27/01 2:31	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0053.D	PFOS-1.3.4MS	042301-PFOS-1.3.4 Spike	THPFOS	5.116	1277667	254	PFBS	4.394	2220887	254
4/27/01 2:43	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0054.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	THPFOS	5.123	1423859	254	PFBS	4.394	1977507	254
4/27/01 2:54	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0055.D	PFOS-2.4.1blk	042301-PFOS-2.4.1 Mtd Blk	THPFOS	5.13	1433506	254	PFBS	4.395	1931697	254
4/27/01 3:50	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0060.D	PFOS-2.4.2blk	042301-PFOS-2.4.2 Mtd Blk	THPFOS	5.131	1413614	254	PFBS	4.409	1944188	254
4/27/01 4:01	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0061.D	PFOS-2.4.3blk	042301-PFOS-2.4.3 Mtd Blk	THPFOS	5.119	1421004	254	PFBS	4.398	1913426	254
4/27/01 4:13	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0062.D	PFOS-2.4.4blkMS	042301-PFOS-2.4.4 Mtd Blk Spike	THPFOS	5.13	1403989	254	PFBS	4.408	1898991	254
4/27/01 4:24	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0063.D	PFOS-2.1.1	042301-PFOS-2.1.1 Smp	THPFOS	5.122	1287849	254	PFBS	4.4	2201997	254
4/27/01 4:35	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0064.D	PFOS-2.1.2	042301-PFOS-2.1.2 Dup	THPFOS	5.136	1278367	254	PFBS	4.415	2231938	254
4/27/01 4:57	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0066.D	PFOS-2.1.3	042301-PFOS-2.1.3 Trip	THPFOS	5.123	1294319	254	PFBS	4.409	2202521	254
4/27/01 5:08	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0067.D	PFOS-2.1.4MS	042301-PFOS-2.1.4 Spike	THPFOS	5.13	1272974	254	PFBS	4.409	2203090	254
4/27/01 5:20	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0068.D	PFOS-2.2.1	042301-PFOS-2.2.1 Smp	THPFOS	5.136	1434509	254	PFBS	4.401	1973173	254
4/27/01 5:31	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0069.D	PFOS-2.2.2	042301-PFOS-2.2.2 Dup	THPFOS	5.137	1421082	254	PFBS	4.409	1956138	254
4/27/01 5:42	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0070.D	PFOS-2.2.3	042301-PFOS-2.2.3 Trip	THPFOS	5.131	1441500	254	PFBS	4.41	1940430	254
4/27/01 6:38	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0075.D	PFOS-2.2.4MS	042301-PFOS-2.2.4 Spike	THPFOS	5.137	1430472	254	PFBS	4.423	1930607	254
4/27/01 6:49	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0076.D	PFOS-2.3.1	042301-PFOS-2.3.1 Smp	THPFOS	5.129	1423581	254	PFBS	4.415	1936770	254
4/27/01 7:00	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0077.D	PFOS-2.3.2	042301-PFOS-2.3.2 Dup	THPFOS	5.123	1417186	254	PFBS	4.416	1899523	254
4/27/01 7:11	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0078.D	PFOS-2.3.3	042301-PFOS-2.3.3 Trip	THPFOS	5.133	1411658	254	PFBS	4.419	1917144	254
4/27/01 7:23	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0079.D	PFOS-2.3.4MS	042301-PFOS-2.3.4 Spike	THPFOS	5.137	1451012	254	PFBS	4.408	1951546	254
								1342769				2056957	2674044
								73849				137331	1439870
								5.5%				6.7%	

Inj Date	Batch	SampType	File	Sample Name	Sample Desc.	Compound Name	RT	Area	Amount	Compound Name	RT	Area	Amount
4/26/01 19:14	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0014.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.387	8824	0
4/26/01 19:26	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0015.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
4/26/01 22:02	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0029.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.39	9125	0
4/26/01 22:13	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0030.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
4/26/01 23:20	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0036.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.402	8664	0
4/27/01 0:50	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0044.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	5.255	7825	0	PFBS	4.407	8480	0
4/27/01 1:01	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0045.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
4/27/01 1:58	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0050.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.408	8742	0
4/27/01 3:28	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0056.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.423	8244	0
4/27/01 3:39	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0059.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
4/27/01 4:46	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0065.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	5.256	7710	0	PFBS	4.423	9199	0
4/27/01 6:15	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0073.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.429	8487	0
4/27/01 6:27	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0074.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
4/27/01 7:56	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0082.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.436	8442	0
4/27/01 8:08	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0083.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
4/27/01 10:44	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0097.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.431	9918	0
4/27/01 10:55	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0098.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/PFOS0425.m (ChemStation); Reprocessing Method: H010426t.m (Target)

Inj Date	Batch	SampType	File	Sample Name	Sample Desc.	Compound Name	RT	Area	Amount	%Theoretical
4/26/01 19:37	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0016.D	01003-07-01	0 ng/mL PFOS	PFOS	0	0	0	-
4/26/01 19:48	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0017.D	01003-07-02	2.5 ng/mL PFOS	PFOS	5.32	81289	2.8	-
4/26/01 19:59	D:\chem\Hillary.i\H010426.b	CALIB_1	HILL0018.D	01003-07-03	5 ng/mL PFOS	PFOS	5.335	138659	5.0	99.5%
4/26/01 20:10	D:\chem\Hillary.i\H010426.b	CALIB_2	HILL0019.D	01003-07-04	10 ng/mL PFOS	PFOS	5.329	254205	9.3	93.4%
4/26/01 20:21	D:\chem\Hillary.i\H010426.b	CALIB_3	HILL0020.D	01003-07-05	25 ng/mL PFOS	PFOS	5.315	660047	24.9	99.5%
4/26/01 20:33	D:\chem\Hillary.i\H010426.b	CALIB_4	HILL0021.D	01003-07-06	40 ng/mL PFOS	PFOS	5.332	1055533	40.5	101.2%
4/26/01 20:44	D:\chem\Hillary.i\H010426.b	CALIB_5	HILL0022.D	01003-07-07	50.1 ng/mL PFOS	PFOS	5.318	1291097	50.1	100.1%
4/26/01 20:55	D:\chem\Hillary.i\H010426.b	CALIB_6	HILL0023.D	01003-07-08	75.1 ng/mL PFOS	PFOS	5.348	1939424	75.6	100.7%
4/26/01 21:06	D:\chem\Hillary.i\H010426.b	CALIB_7	HILL0024.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.322	2537734	99.3	99.1%
4/26/01 21:17	D:\chem\Hillary.i\H010426.b	CALIB_8	HILL0025.D	01003-07-10	250.5 ng/mL PFOS	PFOS	5.335	6004211	254.6	101.6%
4/26/01 21:28	D:\chem\Hillary.i\H010426.b	CALIB_9	HILL0026.D	01003-07-11	400.8 ng/mL PFOS	PFOS	5.327	8957654	394.6	98.4%
4/26/01 21:40	D:\chem\Hillary.i\H010426.b	CALIB_10	HILL0027.D	01003-07-12	501 ng/mL PFOS	PFOS	5.328	10985845	504.8	100.8%
4/26/01 21:51	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0028.D	01003-07-13	1002 ng/mL PFOS	PFOS	5.326	18864507	983.7	-
4/27/01 0:28	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0042.D	01003-07-03	5 ng/mL PFOS	PFOS	5.329	176475	6.1	122.5%
4/27/01 0:39	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0043.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.333	2586427	99.7	99.5%
4/27/01 3:05	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0056.D	01003-07-03	5 ng/mL PFOS	PFOS	5.351	150106	5.1	102.6%
4/27/01 3:17	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0057.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.335	2627085	99.0	98.8%
4/27/01 5:53	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0071.D	01003-07-03	5 ng/mL PFOS	PFOS	5.325	151691	5.1	99.5%
4/27/01 6:04	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0072.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.336	2666281	100.6	100.4%
4/27/01 7:34	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0080.D	01003-07-03	5 ng/mL PFOS	PFOS	5.336	148867	5.1	101.1%
4/27/01 7:45	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0081.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.333	2633934	99.4	99.2%
4/27/01 8:19	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0084.D	01003-07-01	0 ng/mL PFOS	PFOS	0	0	0.0	-
4/27/01 8:30	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0085.D	01003-07-02	2.5 ng/mL PFOS	PFOS	5.333	83665	2.7	-
4/27/01 8:41	D:\chem\Hillary.i\H010426.b	CALIB_11	HILL0086.D	01003-07-03	5 ng/mL PFOS	PFOS	5.333	142999	4.9	97.4%
4/27/01 8:52	D:\chem\Hillary.i\H010426.b	CALIB_12	HILL0087.D	01003-07-04	10 ng/mL PFOS	PFOS	5.333	254810	9.1	90.5%
4/27/01 9:03	D:\chem\Hillary.i\H010426.b	CALIB_13	HILL0088.D	01003-07-05	25 ng/mL PFOS	PFOS	5.334	687053	25.0	100.0%
4/27/01 9:15	D:\chem\Hillary.i\H010426.b	CALIB_14	HILL0089.D	01003-07-06	40 ng/mL PFOS	PFOS	5.327	1065908	39.1	97.8%
4/27/01 9:26	D:\chem\Hillary.i\H010426.b	CALIB_15	HILL0090.D	01003-07-07	50.1 ng/mL PFOS	PFOS	5.34	1360299	50.2	100.3%
4/27/01 9:37	D:\chem\Hillary.i\H010426.b	CALIB_16	HILL0091.D	01003-07-08	75.1 ng/mL PFOS	PFOS	5.327	1965188	76.7	102.1%
4/27/01 9:48	D:\chem\Hillary.i\H010426.b	CALIB_17	HILL0092.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.332	2630530	100.3	100.1%
4/27/01 9:59	D:\chem\Hillary.i\H010426.b	CALIB_18	HILL0093.D	01003-07-10	250.5 ng/mL PFOS	PFOS	5.334	6077465	254.1	101.5%
4/27/01 10:10	D:\chem\Hillary.i\H010426.b	CALIB_19	HILL0094.D	01003-07-11	400.8 ng/mL PFOS	PFOS	5.326	9143568	393.9	98.3%
4/27/01 10:22	D:\chem\Hillary.i\H010426.b	CALIB_20	HILL0095.D	01003-07-12	501 ng/mL PFOS	PFOS	5.335	11226796	503.5	100.5%
4/27/01 10:33	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0096.D	01003-07-13	1002 ng/mL PFOS	PFOS	5.328	19703670	1033.7	-

Two curves were averaged using Target software to quantify data. A standard curve range of only 10 levels is allowed. 5-500 ng/mL was the range chosen.

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/PFOS0425.m (ChemStation); Reprocessing Method: H010426t.m (Target)

Inj Date	Batch	SampType	File	Sample Name	Sample Desc.	Compound Name	RT	Area	Amount	Sample Purity corrected ng/mL	Average µg/mL Std Dev %RSD Spike Recovery
4/26/01 22:24	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0031.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	PFOS	5.322	2260230	89.8	88	*90%*
4/26/01 22:36	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0032.D	PFOS-1.4.1blk	042301-PFOS-1.4.1 Mtd Blk	PFOS	0	0	0.0		
4/26/01 22:47	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0033.D	PFOS-1.4.2blk	042301-PFOS-1.4.2 Mtd Blk	PFOS	0	0	0.0		
4/26/01 22:58	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0034.D	PFOS-1.4.3blk	042301-PFOS-1.4.3 Mtd Blk	PFOS	0	0	0.0		
4/26/01 23:09	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0035.D	PFOS-1.4.4blkMS	042301-PFOS-1.4.4 Mtd Blk Spike	PFOS	5.354	2520637	101.1	99	*101%*
4/26/01 23:32	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0037.D	PFOS-1.1.1	042301-PFOS-1.1.1 Smp	PFOS	5.271	7570273	292.6	286	311
4/26/01 23:43	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0038.D	PFOS-1.1.2	042301-PFOS-1.1.2 Dup	PFOS	5.277	8619490	331.8	325	22.0
4/26/01 23:54	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0039.D	PFOS-1.1.3	042301-PFOS-1.1.3 Trip	PFOS	5.255	8589480	329.8	323	7%
4/27/01 0:05	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0040.D	PFOS-1.1.4MS	042301-PFOS-1.1.4 Spike	PFOS	5.264	9810483	396.8	388	*78%*
4/27/01 0:16	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0041.D	PFOS-1.2.1	042301-PFOS-1.2.1 Smp	PFOS	5.251	7600633	298.7	292	288
4/27/01 1:12	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0046.D	PFOS-1.2.2	042301-PFOS-1.2.2 Dup	PFOS	5.263	7571396	294.9	289	4.0
4/27/01 1:24	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0047.D	PFOS-1.2.3	042301-PFOS-1.2.3 Trip	PFOS	5.264	7594271	290.2	284	1%
4/27/01 1:35	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0048.D	PFOS-1.2.4MS	042301-PFOS-1.2.4 Spike	PFOS	5.256	9667899	385.1	377	*90%*
4/27/01 1:47	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0049.D	PFOS-1.3.1	042301-PFOS-1.3.1 Smp	PFOS	5.27	8267658	320.6	314	306
4/27/01 2:09	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0051.D	PFOS-1.3.2	042301-PFOS-1.3.2 Dup	PFOS	5.263	8045600	307.6	301	7.2
4/27/01 2:20	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0052.D	PFOS-1.3.3	042301-PFOS-1.3.3 Trip	PFOS	5.272	8019673	308.5	302	2%
4/27/01 2:31	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0053.D	PFOS-1.3.4MS	042301-PFOS-1.3.4 Spike	PFOS	5.263	10242365	405.2	397	*93%*
4/27/01 2:43	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0054.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	PFOS	5.333	2342701	91.0	89	*91%*
4/27/01 2:54	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0055.D	PFOS-2.4.1blk	042301-PFOS-2.4.1 Mtd Blk	PFOS	0	0	0.0		
4/27/01 3:50	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0060.D	PFOS-2.4.2blk	042301-PFOS-2.4.2 Mtd Blk	PFOS	0	0	0.0		
4/27/01 4:01	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0061.D	PFOS-2.4.3blk	042301-PFOS-2.4.3 Mtd Blk	PFOS	0	0	0.0		
4/27/01 4:13	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0062.D	PFOS-2.4.4blkMS	042301-PFOS-2.4.4 Mtd Blk Spike	PFOS	5.333	2571930	104.8	103	*105%*
4/27/01 4:24	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0063.D	PFOS-2.1.1	042301-PFOS-2.1.1 Smp	PFOS	5.262	7822147	300.1	294	308
4/27/01 4:35	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0064.D	PFOS-2.1.2	042301-PFOS-2.1.2 Dup	PFOS	5.276	8758474	336.2	329	18.3
4/27/01 4:57	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0066.D	PFOS-2.1.3	042301-PFOS-2.1.3 Trip	PFOS	5.257	8011616	308.3	302	6%
4/27/01 5:08	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0067.D	PFOS-2.1.4MS	042301-PFOS-2.1.4 Spike	PFOS	5.27	10379987	415.5	407	*101%*
4/27/01 5:20	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0068.D	PFOS-2.2.1	042301-PFOS-2.2.1 Smp	PFOS	5.36	46170	1.4		
4/27/01 5:31	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0069.D	PFOS-2.2.2	042301-PFOS-2.2.2 Dup	PFOS	0	0	0.0		
4/27/01 5:42	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0070.D	PFOS-2.2.3	042301-PFOS-2.2.3 Trip	PFOS	0	0	0.0		
4/27/01 6:38	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0075.D	PFOS-2.2.4MS	042301-PFOS-2.2.4 Spike	PFOS	5.34	2618661	104.9	103	*105%*
4/27/01 6:49	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0076.D	PFOS-2.3.1	042301-PFOS-2.3.1 Smp	PFOS	0	0	0.0		
4/27/01 7:00	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0077.D	PFOS-2.3.2	042301-PFOS-2.3.2 Dup	PFOS	0	0	0.0		
4/27/01 7:11	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0078.D	PFOS-2.3.3	042301-PFOS-2.3.3 Trip	PFOS	0	0	0.0		
4/27/01 7:23	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0079.D	PFOS-2.3.4MS	042301-PFOS-2.3.4 Spike	PFOS	5.34	2483685	98.1	96	*98%*

Day 1 results	
average	302
std dev	12
%RSD:	4.0%

3M Environmental Laboratory
Project # E01-0768

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/PFOS0425.m (ChemStation); Reprocessing Method: H010426t.m (Target)

Inj Date	Batch	SampType	File	Sample Name	Sample Desc.	Compound Name	RT	Area	Amount
4/26/01 19:14	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0014.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.424	36585	Below LOQ
4/26/01 19:26	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0015.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/26/01 22:02	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0029.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.419	65012	Below LOQ
4/26/01 22:13	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0030.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.431	22312	Below LOQ
4/26/01 23:20	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0036.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 0:50	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0044.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 1:01	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0045.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 1:58	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0050.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.367	31709	Below LOQ
4/27/01 3:28	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0058.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 3:39	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0059.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 4:46	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0065.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.34	33691	Below LOQ
4/27/01 6:15	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0073.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 6:27	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0074.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 7:56	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0082.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 8:08	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0083.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
4/27/01 10:44	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0097.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.433	50495	Below LOQ
4/27/01 10:55	D:\chem\Hillary.i\H010426.b	SAMPLE	HILL0098.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.431	14767	0

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Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/PFOS0425.m (ChemStation);
Reprocessing Method: H010501t.m (Target) Quant range: 10-501 ng/mL

Day 2 samples were re-aliquotted from the initial centrifuge tube on 05-01-01 into 1.5 mL centrifuge tubes, centrifuged, and then diluted 1:100 and 1:10 for a final dilution factor of 1:1,000 prior to analysis.

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amount	Compound Name	RT	Area	Amount
SAMPLE	HILL0016.D	01003-07-01	0 ng/mL PFOS	THPFOS	5.119	1282312	254	PFBS	4.39	1737872	254
SAMPLE	HILL0017.D	01003-07-02	2.5 ng/mL PFOS	THPFOS	5.116	1300843	254	PFBS	4.388	1742704	254
CALIB_1Disabled	HILL0018.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.123	1310415	254	PFBS	4.409	1726536	254
CALIB_2	HILL0019.D	01003-07-04	10 ng/mL PFOS	THPFOS	5.122	1318634	254	PFBS	4.408	1720895	254
CALIB_3	HILL0020.D	01003-07-05	25 ng/mL PFOS	THPFOS	5.117	1324328	254	PFBS	4.41	1770020	254
CALIB_4	HILL0021.D	01003-07-06	40 ng/mL PFOS	THPFOS	5.117	1329731	254	PFBS	4.409	1787802	254
CALIB_5	HILL0022.D	01003-07-07	50.1 ng/mL PFOS	THPFOS	5.124	1346360	254	PFBS	4.416	1786195	254
CALIB_6	HILL0023.D	01003-07-08	75.1 ng/mL PFOS	THPFOS	5.118	1335866	254	PFBS	4.411	1789034	254
CALIB_7	HILL0024.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.125	1352724	254	PFBS	4.404	1791645	254
CALIB_8	HILL0025.D	01003-07-10	250.5 ng/mL PFOS	THPFOS	5.124	1343468	254	PFBS	4.417	1774591	254
CALIB_9	HILL0026.D	01003-07-11	400.8 ng/mL PFOS	THPFOS	5.119	1338624	254	PFBS	4.411	1789155	254
CALIB_10	HILL0027.D	01003-07-12	501 ng/mL PFOS	THPFOS	5.115	1349352	254	PFBS	4.408	1796642	254
SAMPLE	HILL0028.D	01003-07-13	1002 ng/mL PFOS	THPFOS	5.116	1311155	254	PFBS	4.408	1764193	254
SAMPLE	HILL0041.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.129	1385797	254	PFBS	4.408	1769946	254
SAMPLE	HILL0042.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.117	1376301	254	PFBS	4.41	1764551	254
SAMPLE	HILL0054.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.122	1375001	254	PFBS	4.401	1783815	254
SAMPLE	HILL0055.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.117	1372102	254	PFBS	4.402	1769874	254
SAMPLE	HILL0058.D	01003-07-01	0 ng/mL PFOS	THPFOS	5.131	1373368	254	PFBS	4.409	1780686	254
SAMPLE	HILL0059.D	01003-07-02	2.5 ng/mL PFOS	THPFOS	5.108	1387395	254	PFBS	4.407	1788657	254
CALIB_11 Disabled	HILL0060.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.115	1396556	254	PFBS	4.401	1769985	254
CALIB_12	HILL0061.D	01003-07-04	10 ng/mL PFOS	THPFOS	5.117	1407101	254	PFBS	4.417	1750426	254
CALIB_13	HILL0062.D	01003-07-05	25 ng/mL PFOS	THPFOS	5.117	1388273	254	PFBS	4.416	1770162	254
CALIB_14	HILL0063.D	01003-07-06	40 ng/mL PFOS	THPFOS	5.131	1399058	254	PFBS	4.416	1793510	254
CALIB_15	HILL0064.D	01003-07-07	50.1 ng/mL PFOS	THPFOS	5.111	1399532	254	PFBS	4.411	1763775	254
CALIB_16	HILL0065.D	01003-07-08	75.1 ng/mL PFOS	THPFOS	5.115	1382061	254	PFBS	4.401	1748667	254
CALIB_17	HILL0066.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.122	1384218	254	PFBS	4.415	1777945	254
CALIB_18	HILL0067.D	01003-07-10	250.5 ng/mL PFOS	THPFOS	5.117	1375930	254	PFBS	4.402	1745338	254
CALIB_19	HILL0068.D	01003-07-11	400.8 ng/mL PFOS	THPFOS	5.103	1364296	254	PFBS	4.403	1751400	254
CALIB_20	HILL0069.D	01003-07-12	501 ng/mL PFOS	THPFOS	5.11	1378457	254	PFBS	4.402	1799585	254
SAMPLE	HILL0070.D	01003-07-13	1002 ng/mL PFOS	THPFOS	5.115	1357091	254	PFBS	4.407	1769921	254

3M Environmental Laboratory
Project # E01-0768

Exact Copy of Original

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Initial Date

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/PFOS0425.m (ChemStation);
Reprocessing Method: H010501t.m (Target) Quant range: 10-501 ng/mL

Day 2 samples were re-aliquotted from the initial centrifuge tube on 05-01-01 into 1.5 mL centrifuge tubes, centrifuged, and then diluted 1:100 and 1:10 for a final dilution factor of 1:1,000 prior to analysis.

SAMPLE	HILL0031.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	THPFOS	5.122	1322947	254	PFBS	4.414	1715710	254
SAMPLE	HILL0032.D	PFOS-2.4.1blk	042301-PFOS-2.4.1 Mtd Blk	THPFOS	5.115	1317272	254	PFBS	4.407	1718184	254
SAMPLE	HILL0033.D	PFOS-2.4.2blk	042301-PFOS-2.4.2 Mtd Blk	THPFOS	5.109	1319308	254	PFBS	4.408	1695951	254
SAMPLE	HILL0034.D	PFOS-2.4.3blk	042301-PFOS-2.4.3 Mtd Blk	THPFOS	5.122	1316462	254	PFBS	4.394	1698236	254
SAMPLE	HILL0035.D	PFOS-2.4.4blkMS	042301-PFOS-2.4.4 Mtd Blk Spike	THPFOS	5.111	1318338	254	PFBS	4.403	1686696	254
SAMPLE	HILL0037.D	PFOS-2.1.1	042301-PFOS-2.1.1 Smp	THPFOS	5.115	1215865	254	PFBS	4.408	1912250	254
SAMPLE	HILL0038.D	PFOS-2.1.2	042301-PFOS-2.1.2 Dup	THPFOS	5.108	1161890	254	PFBS	4.401	1969789	254
SAMPLE	HILL0039.D	PFOS-2.1.3	042301-PFOS-2.1.3 Trip	THPFOS	5.116	1218774	254	PFBS	4.409	1995209	254
SAMPLE	HILL0040.D	PFOS-2.1.4MS	042301-PFOS-2.1.4 Spike	THPFOS	5.116	1224336	254	PFBS	4.402	1951822	254
SAMPLE	HILL0045.D	PFOS-2.2.1	042301-PFOS-2.2.1 Smp	THPFOS	5.123	1256724	254	PFBS	4.408	1900616	254
SAMPLE	HILL0046.D	PFOS-2.2.2	042301-PFOS-2.2.2 Dup	THPFOS	5.122	1246843	254	PFBS	4.401	1926427	254
SAMPLE	HILL0047.D	PFOS-2.2.3	042301-PFOS-2.2.3 Trip	THPFOS	5.11	1245602	254	PFBS	4.409	1881971	254
SAMPLE	HILL0048.D	PFOS-2.2.4MS	042301-PFOS-2.2.4 Spike	THPFOS	5.117	1219461	254	PFBS	4.402	1935761	254
SAMPLE	HILL0050.D	PFOS-2.3.1	042301-PFOS-2.3.1 Smp	THPFOS	5.117	1211070	254	PFBS	4.41	1958520	254
SAMPLE	HILL0051.D	PFOS-2.3.2	042301-PFOS-2.3.2 Dup	THPFOS	5.116	1227470	254	PFBS	4.408	1962518	254
SAMPLE	HILL0052.D	PFOS-2.3.3	042301-PFOS-2.3.3 Trip	THPFOS	5.116	1222931	254	PFBS	4.409	1984447	254
SAMPLE	HILL0053.D	PFOS-2.3.4MS	042301-PFOS-2.3.4 Spike	THPFOS	5.111	1223446	254	PFBS	4.397	1937135	254
				Average:		1319470				1806527	
				Std Deviation:		65017				86617	
				%RSD:		4.9%				4.8%	
SAMPLE	HILL0014.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	5.189	7753	0	PFBS	4.418	7561	0
SAMPLE	HILL0015.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0029.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	5.195	8699	0	PFBS	0	0	0
SAMPLE	HILL0030.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0036.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0043.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0044.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0049.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0056.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0057.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0071.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0072.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/PFOS0425.m (ChemStation);
Reprocessing Method: H010501t.m (Target) Quant range: 10-501 ng/mL

Day 2 samples were re-aliquotted from the initial centrifuge tube on 05-01-01 into 1.5 mL centrifuge tubes, centrifuged, and then diluted 1:100 and 1:10 for a final dilution factor of 1:1,000 prior to analysis.

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amount	% Recovery
SAMPLE	HILL0016.D	01003-07-01	0 ng/mL PFOS	PFOS	0	0	0	-
SAMPLE	HILL0017.D	01003-07-02	2.5 ng/mL PFOS	PFOS	5.326	71773	4	-
CALIB_1Disabled	HILL0018.D	01003-07-03	5 ng/mL PFOS	PFOS	5.326	129494	6	-
CALIB_2	HILL0019.D	01003-07-04	10 ng/mL PFOS	PFOS	5.318	230148	10	105%
CALIB_3	HILL0020.D	01003-07-05	25 ng/mL PFOS	PFOS	5.32	628525	26	103%
CALIB_4	HILL0021.D	01003-07-06	40 ng/mL PFOS	PFOS	5.32	994126	40	100%
CALIB_5	HILL0022.D	01003-07-07	50.1 ng/mL PFOS	PFOS	5.32	1255424	50	101%
CALIB_6	HILL0023.D	01003-07-08	75.1 ng/mL PFOS	PFOS	5.321	1866886	75	100%
CALIB_7	HILL0024.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.342	2434000	99	99%
CALIB_8	HILL0025.D	01003-07-10	250.5 ng/mL PFOS	PFOS	5.32	5704519	252	100%
CALIB_9	HILL0026.D	01003-07-11	400.8 ng/mL PFOS	PFOS	5.322	8544962	397	99%
CALIB_10	HILL0027.D	01003-07-12	501 ng/mL PFOS	PFOS	5.318	10504197	506	101%
SAMPLE	HILL0028.D	01003-07-13	1002 ng/mL PFOS	PFOS	5.319	18199294	1041	-
SAMPLE	HILL0041.D	01003-07-03	5 ng/mL PFOS	PFOS	5.332	152981	7	-
SAMPLE	HILL0042.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.32	2399376	99	99%
SAMPLE	HILL0054.D	01003-07-03	5 ng/mL PFOS	PFOS	5.326	158654	7	-
SAMPLE	HILL0055.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.32	2404840	99	99%
SAMPLE	HILL0058.D	01003-07-01	0 ng/mL PFOS	PFOS	0	0	0	-
SAMPLE	HILL0059.D	01003-07-02	2.5 ng/mL PFOS	PFOS	5.311	72552	4	-
CALIB_11 Disabled	HILL0060.D	01003-07-03	5 ng/mL PFOS	PFOS	5.326	130239	6	-
CALIB_12	HILL0061.D	01003-07-04	10 ng/mL PFOS	PFOS	5.32	233273	10	104%
CALIB_13	HILL0062.D	01003-07-05	25 ng/mL PFOS	PFOS	5.32	621213	25	102%
CALIB_14	HILL0063.D	01003-07-06	40 ng/mL PFOS	PFOS	5.334	994794	40	100%
CALIB_15	HILL0064.D	01003-07-07	50.1 ng/mL PFOS	PFOS	5.314	1220007	50	99%
CALIB_16	HILL0065.D	01003-07-08	75.1 ng/mL PFOS	PFOS	5.325	1796101	74	99%
CALIB_17	HILL0066.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.325	2409666	99	99%
CALIB_18	HILL0067.D	01003-07-10	250.5 ng/mL PFOS	PFOS	5.327	5602770	251	100%
CALIB_19	HILL0068.D	01003-07-11	400.8 ng/mL PFOS	PFOS	5.313	8515207	406	101%
CALIB_20	HILL0069.D	01003-07-12	501 ng/mL PFOS	PFOS	5.313	10313769	494	99%
SAMPLE	HILL0070.D	01003-07-13	1002 ng/mL PFOS	PFOS	5.325	17653606	995	-

Two curves were averaged using Target software to quantify data. A standard curve range of only 10 levels is allowed. 5-500 ng/mL was the initial range chosen.
However, the 5 ng/mL std did not meet acceptance criteria, therefore the 5 ng/mL std was disabled and the data was re-quantified using the std curve range 10-501 ng/mL.

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/PFOS0425.m (ChemStation);
Reprocessing Method: H010501t.m (Target) Quant range: 10-501 ng/mL

Day 2 samples were re-aliquotted from the initial centrifuge tube on 05-01-01 into 1.5 mL centrifuge tubes, centrifuged, and then diluted 1:100 and 1:10 for a final dilution factor of 1:1,000 prior to analysis.

								Average µg/mL, dil factor corrected		
								Sample Purity	Std Dev %RSD	
SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amt (ng/mL)	corrected ng/mL	*Spike Recovery*	
SAMPLE	HILL0031.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	PFOS	5.325	2110116	89	87	*89%*	
SAMPLE	HILL0032.D	PFOS-2.4.1blk	042301-PFOS-2.4.1 Mtd Blk	PFOS	0	0	0			
SAMPLE	HILL0033.D	PFOS-2.4.2blk	042301-PFOS-2.4.2 Mtd Blk	PFOS	0	0	0			
SAMPLE	HILL0034.D	PFOS-2.4.3blk	042301-PFOS-2.4.3 Mtd Blk	PFOS	0	0	0			
SAMPLE	HILL0035.D	PFOS-2.4.4blkMS	042301-PFOS-2.4.4 Mtd Blk Spike	PFOS	5.314	2188222	94	92	*94%*	
SAMPLE	HILL0037.D	PFOS-2.1.1	042301-PFOS-2.1.1 Smp	PFOS	5.248	7420988	312	305	358	
SAMPLE	HILL0038.D	PFOS-2.1.2	042301-PFOS-2.1.2 Dup	PFOS	5.248	9885661	421	412	54	
SAMPLE	HILL0039.D	PFOS-2.1.3	042301-PFOS-2.1.3 Trip	PFOS	5.256	8832844	363	356	15%	
SAMPLE	HILL0040.D	PFOS-2.1.4MS	042301-PFOS-2.1.4 Spike	PFOS	5.249	9719956	417	409	*52%*	
SAMPLE	HILL0045.D	PFOS-2.2.1	042301-PFOS-2.2.1 Smp	PFOS	5.263	7021826	295	289	280	
SAMPLE	HILL0046.D	PFOS-2.2.2 .	042301-PFOS-2.2.2 Dup	PFOS	5.255	7094280	294	288	15	
SAMPLE	HILL0047.D	PFOS-2.2.3	042301-PFOS-2.2.3 Trip	PFOS	5.25	6410858	269	263	5%	
SAMPLE	HILL0048.D	PFOS-2.2.4MS	042301-PFOS-2.2.4 Spike	PFOS	5.257	9712496	421	412	*135%*	
SAMPLE	HILL0050.D	PFOS-2.3.1	042301-PFOS-2.3.1 Smp	PFOS	5.257	8177762	339	332	338	
SAMPLE	HILL0051.D	PFOS-2.3.2	042301-PFOS-2.3.2 Dup	PFOS	5.249	8319423	345	338	7	
SAMPLE	HILL0052.D	PFOS-2.3.3	042301-PFOS-2.3.3 Trip	PFOS	5.249	8547947	352	345	2%	
SAMPLE	HILL0053.D	PFOS-2.3.4MS	042301-PFOS-2.3.4 Spike	PFOS	5.251	9269162	398	390	*53%*	
SAMPLE	HILL0014.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.413	24002	below LOQ			
SAMPLE	HILL0015.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0			
SAMPLE	HILL0029.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.419	40197	below LOQ			
SAMPLE	HILL0030.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.403	16788	below LOQ			
SAMPLE	HILL0036.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0			
SAMPLE	HILL0043.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0			
SAMPLE	HILL0044.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0			
SAMPLE	HILL0049.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.34	23584	below LOQ			
SAMPLE	HILL0056.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0			
SAMPLE	HILL0057.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0			
SAMPLE	HILL0071.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.412	38878	below LOQ			
SAMPLE	HILL0072.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.404	14734	below LOQ			

Day 2 results, µg/mL	
average	325
std dev	40
%RSD:	12%

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Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 2 and 3; Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010430.s/PFOS0425.m (ChemStation); Reprocessing Method: H010430t.m (Target)

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amount	Compound Name	RT	Area	Amount
SAMPLE	HILL0016.D	01003-07-01	0 ng/mL PFOS	THPFOS	5.105	1240800	254	PFBS	4.384	1897371	254
SAMPLE	HILL0017.D	01003-07-02	2.5 ng/mL PFOS	THPFOS	5.101	1259512	254	PFBS	4.38	1883485	254
CALIB_1	HILL0018.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.096	1268358	254	PFBS	4.382	1898698	254
CALIB_2	HILL0019.D	01003-07-04	10 ng/mL PFOS	THPFOS	5.104	1273859	254	PFBS	4.382	1894001	254
CALIB_3	HILL0020.D	01003-07-05	25 ng/mL PFOS	THPFOS	5.103	1287880	254	PFBS	4.382	1891481	254
CALIB_4	HILL0021.D	01003-07-06	40 ng/mL PFOS	THPFOS	5.104	1293445	254	PFBS	4.376	1895316	254
CALIB_5	HILL0022.D	01003-07-07	50.1 ng/mL PFOS	THPFOS	5.105	1299052	254	PFBS	4.376	1887750	254
CALIB_6	HILL0023.D	01003-07-08	75.1 ng/mL PFOS	THPFOS	5.101	1297549	254	PFBS	4.38	1894332	254
CALIB_7	HILL0024.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.105	1310823	254	PFBS	4.376	1899388	254
CALIB_8	HILL0025.D	01003-07-10	250.5 ng/mL PFOS	THPFOS	5.103	1305549	254	PFBS	4.375	1877817	254
CALIB_9	HILL0026.D	01003-07-11	400.8 ng/mL PFOS	THPFOS	5.103	1299241	254	PFBS	4.375	1884885	254
CALIB_10	HILL0027.D	01003-07-12	501 ng/mL PFOS	THPFOS	5.095	1306826	254	PFBS	4.374	1906259	254
SAMPLE	HILL0028.D	01003-07-13	1002 ng/mL PFOS	THPFOS	5.109	1302797	254	PFBS	4.381	1898513	254
SAMPLE	HILL0041.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.1	1390044	254	PFBS	4.386	1873917	254
SAMPLE	HILL0042.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.103	1386912	254	PFBS	4.381	1895596	254
SAMPLE	HILL0054.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.103	1417554	254	PFBS	4.381	1870203	254
SAMPLE	HILL0055.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.101	1416339	254	PFBS	4.373	1863988	254
SAMPLE	HILL0068.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.11	1463633	254	PFBS	4.381	1941991	254
SAMPLE	HILL0069.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.103	1453681	254	PFBS	4.382	1934618	254
SAMPLE	HILL0081.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.098	1482897	254	PFBS	4.391	1898558	254
SAMPLE	HILL0082.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.101	1463161	254	PFBS	4.401	1912978	254
SAMPLE	HILL0085.D	01003-07-01	0 ng/mL PFOS	THPFOS	5.101	1470609	254	PFBS	4.394	1882104	254
SAMPLE	HILL0086.D	01003-07-02	2.5 ng/mL PFOS	THPFOS	5.095	1481303	254	PFBS	4.394	1898530	254
CALIB_11	HILL0087.D	01003-07-03	5 ng/mL PFOS	THPFOS	5.097	1492012	254	PFBS	4.396	1905370	254
CALIB_12	HILL0088.D	01003-07-04	10 ng/mL PFOS	THPFOS	5.095	1477732	254	PFBS	4.394	1894008	254
CALIB_13	HILL0089.D	01003-07-05	25 ng/mL PFOS	THPFOS	5.112	1481121	254	PFBS	4.397	1892338	254
CALIB_14	HILL0090.D	01003-07-06	40 ng/mL PFOS	THPFOS	5.095	1479643	254	PFBS	4.394	1891942	254
CALIB_15	HILL0091.D	01003-07-07	50.1 ng/mL PFOS	THPFOS	5.095	1475404	254	PFBS	4.388	1897650	254
CALIB_16	HILL0092.D	01003-07-08	75.1 ng/mL PFOS	THPFOS	5.094	1469508	254	PFBS	4.394	1911398	254
CALIB_17	HILL0093.D	01003-07-09	100.2 ng/mL PFOS	THPFOS	5.096	1496019	254	PFBS	4.395	1928086	254
CALIB_18	HILL0094.D	01003-07-10	250.5 ng/mL PFOS	THPFOS	5.109	1476250	254	PFBS	4.395	1869148	254
CALIB_19	HILL0095.D	01003-07-11	400.8 ng/mL PFOS	THPFOS	5.097	1466888	254	PFBS	4.396	1898244	254
CALIB_20	HILL0096.D	01003-07-12	501 ng/mL PFOS	THPFOS	5.109	1475286	254	PFBS	4.38	1911397	254
SAMPLE	HILL0097.D	01003-07-13	1002 ng/mL PFOS	THPFOS	5.103	1440061	254	PFBS	4.382	1902178	254
SAMPLE	HILL0031.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	THPFOS	5.095	1325456	254	PFBS	4.381	1823070	254
SAMPLE	HILL0032.D	PFOS-2.4.1blk	042301-PFOS-2.4.1 Mtd Blk	THPFOS	5.096	1344481	254	PFBS	4.375	1834828	254
SAMPLE	HILL0033.D	PFOS-2.4.2blk	042301-PFOS-2.4.2 Mtd Blk	THPFOS	5.101	1347423	254	PFBS	4.379	1867721	254
SAMPLE	HILL0034.D	PFOS-2.4.3blk	042301-PFOS-2.4.3 Mtd Blk	THPFOS	5.103	1347097	254	PFBS	4.389	1835565	254
SAMPLE	HILL0035.D	PFOS-2.4.4blkMS	042301-PFOS-2.4.4 Mtd Blk Spike	THPFOS	5.095	1324124	254	PFBS	4.38	1847790	254
SAMPLE	HILL0037.D	PFOS-2.1.1	042301-PFOS-2.1.1 Smp	THPFOS	5.101	1163460	254	PFBS	4.387	1968845	254
SAMPLE	HILL0038.D	PFOS-2.1.2	042301-PFOS-2.1.2 Dup	THPFOS	5.102	1199081	254	PFBS	4.388	2112105	254
SAMPLE	HILL0039.D	PFOS-2.1.3	042301-PFOS-2.1.3 Trip	THPFOS	5.102	1228695	254	PFBS	4.387	2098049	254
SAMPLE	HILL0040.D	PFOS-2.1.4MS	042301-PFOS-2.1.4 Spike	THPFOS	5.104	1219919	254	PFBS	4.383	2057424	254
SAMPLE	HILL0045.D	PFOS-2.2.1	042301-PFOS-2.2.1 Smp	THPFOS	5.101	1364440	254	PFBS	4.387	1826163	254
SAMPLE	HILL0046.D	PFOS-2.2.2	042301-PFOS-2.2.2 Dup	THPFOS	5.096	1385823	254	PFBS	4.382	1818895	254
SAMPLE	HILL0047.D	PFOS-2.2.3	042301-PFOS-2.2.3 Trip	THPFOS	5.102	1392690	254	PFBS	4.381	1822627	254
SAMPLE	HILL0048.D	PFOS-2.2.4MS	042301-PFOS-2.2.4 Spike	THPFOS	5.102	1376257	254	PFBS	4.374	1820132	254
SAMPLE	HILL0050.D	PFOS-2.3.1	042301-PFOS-2.3.1 Smp	THPFOS	5.103	1367578	254	PFBS	4.374	1841611	254
SAMPLE	HILL0051.D	PFOS-2.3.2	042301-PFOS-2.3.2 Dup	THPFOS	5.097	1373402	254	PFBS	4.376	1797985	254
SAMPLE	HILL0052.D	PFOS-2.3.3	042301-PFOS-2.3.3 Trip	THPFOS	5.096	1382100	254	PFBS	4.374	1816031	254
SAMPLE	HILL0053.D	PFOS-2.3.4MS	042301-PFOS-2.3.4 Spike	THPFOS	5.096	1373073	254	PFBS	4.374	1843565	254

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Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 2 and 3; Analytical Instrument: HPLC/MSD "Hillary";

SAMPLE	HILL0058.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	THPFOS	5.095	1394750	254	PFBS	4.367	1828732	254
SAMPLE	HILL0059.D	PFOS-3.4.1blk	042301-PFOS-3.4.1 Mtd Blk	THPFOS	5.098	1419875	254	PFBS	4.363	1870275	254
SAMPLE	HILL0060.D	PFOS-3.4.2blk	042301-PFOS-3.4.2 Mtd Blk	THPFOS	5.096	1420804	254	PFBS	4.368	1875725	254
SAMPLE	HILL0061.D	PFOS-3.4.3blk	042301-PFOS-3.4.3 Mtd Blk	THPFOS	5.098	1433374	254	PFBS	4.369	1883710	254
SAMPLE	HILL0062.D	PFOS-3.4.4blkMS	042301-PFOS-3.4.4 Mtd Blk Spike	THPFOS	5.103	1406537	254	PFBS	4.375	1858704	254
SAMPLE	HILL0064.D	PFOS-3.1.1	042301-PFOS-3.1.1 Smp	THPFOS	5.097	1310521	254	PFBS	4.376	2034585	254
SAMPLE	HILL0065.D	PFOS-3.1.2	042301-PFOS-3.1.2 Dup	THPFOS	5.109	1294952	254	PFBS	4.38	2079222	254
SAMPLE	HILL0066.D	PFOS-3.1.3	042301-PFOS-3.1.3 Trip	THPFOS	5.102	1296261	254	PFBS	4.38	2046016	254
SAMPLE	HILL0067.D	PFOS-3.1.4MS	042301-PFOS-3.1.4 Spike	THPFOS	5.107	1334559	254	PFBS	4.379	2057849	254
SAMPLE	HILL0072.D	PFOS-3.2.1	042301-PFOS-3.2.1 Smp	THPFOS	5.105	1305210	254	PFBS	4.384	2128725	254
SAMPLE	HILL0073.D	PFOS-3.2.2	042301-PFOS-3.2.2 Dup	THPFOS	5.109	1308424	254	PFBS	4.388	2170045	254
SAMPLE	HILL0074.D	PFOS-3.2.3	042301-PFOS-3.2.3 Trip	THPFOS	5.111	1312470	254	PFBS	4.39	2160742	254
SAMPLE	HILL0075.D	PFOS-3.2.4MS	042301-PFOS-3.2.4 Spike	THPFOS	5.104	1289916	254	PFBS	4.397	2146697	254
SAMPLE	HILL0077.D	PFOS-3.3.1	042301-PFOS-3.3.1 Smp	THPFOS	5.104	1325618	254	PFBS	4.397	2084860	254
SAMPLE	HILL0078.D	PFOS-3.3.2	042301-PFOS-3.3.2 Dup	THPFOS	5.11	1332228	254	PFBS	4.389	2030571	254
SAMPLE	HILL0079.D	PFOS-3.3.3	042301-PFOS-3.3.3 Trip	THPFOS	5.101	1339789	254	PFBS	4.386	2064866	254
SAMPLE	HILL0080.D	PFOS-3.3.4MS	042301-PFOS-3.3.4 Spike	THPFOS	5.098	1327428	254	PFBS	4.383	2068536	254
					Average:	1364258.28				1925085.4	
					Std Deviation:	82080				95672	
					%RSD:	6.0%				5.0%	
SAMPLE	HILL0014.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0015.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0029.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0030.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0036.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0043.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.409	7786	0
SAMPLE	HILL0044.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0049.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	5.178	7634	0	PFBS	0	0	0
SAMPLE	HILL0056.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0057.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0063.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0070.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0071.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0076.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	4.423	7887	0
SAMPLE	HILL0083.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	5.172	8030	0	PFBS	0	0	0
SAMPLE	HILL0084.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0098.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	5.2	7970	0	PFBS	0	0	0
SAMPLE	HILL0099.D	TN-A 4802 MeOH	Solvent Blank	THPFOS	0	0	0	PFBS	0	0	0

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 2 and 3; Analytical Instrument: HPLC/MSD "Hillary";

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amt (ng/mL)	% Recovery
SAMPLE	HILL0016.D	01003-07-01	0 ng/mL PFOS	PFOS	0	0	0.0	-
SAMPLE	HILL0017.D	01003-07-02	2.5 ng/mL PFOS	PFOS	5.311	77068	3.4	-
CALIB_1	HILL0018.D	01003-07-03	5 ng/mL PFOS	PFOS	5.306	140922	5.7	114%
CALIB_2	HILL0019.D	01003-07-04	10 ng/mL PFOS	PFOS	5.314	250842	9.8	98%
CALIB_3	HILL0020.D	01003-07-05	25 ng/mL PFOS	PFOS	5.313	644213	24.7	99%
CALIB_4	HILL0021.D	01003-07-06	40 ng/mL PFOS	PFOS	5.307	1035507	39.7	99%
CALIB_5	HILL0022.D	01003-07-07	50.1 ng/mL PFOS	PFOS	5.308	1297981	50.1	100%
CALIB_6	HILL0023.D	01003-07-08	75.1 ng/mL PFOS	PFOS	5.311	1923999	74.8	100%
CALIB_7	HILL0024.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.308	2507092	98.2	98%
CALIB_8	HILL0025.D	01003-07-10	250.5 ng/mL PFOS	PFOS	5.306	5879693	248.2	99%
CALIB_9	HILL0026.D	01003-07-11	400.8 ng/mL PFOS	PFOS	5.306	8875074	394.1	98%
CALIB_10	HILL0027.D	01003-07-12	501 ng/mL PFOS	PFOS	5.305	10870332	493.2	98%
SAMPLE	HILL0028.D	01003-07-13	1002 ng/mL PFOS	PFOS	5.312	18717748	968.5	-
SAMPLE	HILL0041.D	01003-07-03	5 ng/mL PFOS	PFOS	5.31	163873	6.7	133%
SAMPLE	HILL0042.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.306	2519548	98.9	99%
SAMPLE	HILL0054.D	01003-07-03	5 ng/mL PFOS	PFOS	5.313	136445	5.6	113%
SAMPLE	HILL0055.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.312	2484938	99.2	99%
SAMPLE	HILL0068.D	01003-07-03	5 ng/mL PFOS	PFOS	5.32	166490	6.5	100%
SAMPLE	HILL0069.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.306	2588211	99.5	99%
SAMPLE	HILL0081.D	01003-07-03	5 ng/mL PFOS	PFOS	5.301	162640	6.5	130%
SAMPLE	HILL0082.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.304	2575229	100.2	100%
SAMPLE	HILL0085.D	01003-07-01	0 ng/mL PFOS	PFOS	0	0	0.0	-
SAMPLE	HILL0086.D	01003-07-02	2.5 ng/mL PFOS	PFOS	5.298	74899	3.3	-
CALIB_11	HILL0087.D	01003-07-03	5 ng/mL PFOS	PFOS	5.3	138171	5.6	112%
CALIB_12	HILL0088.D	01003-07-04	10 ng/mL PFOS	PFOS	5.298	248359	9.7	97%
CALIB_13	HILL0089.D	01003-07-05	25 ng/mL PFOS	PFOS	5.322	648497	24.9	100%
CALIB_14	HILL0090.D	01003-07-06	40 ng/mL PFOS	PFOS	5.298	1030084	39.6	99%
CALIB_15	HILL0091.D	01003-07-07	50.1 ng/mL PFOS	PFOS	5.298	1323882	50.9	102%
CALIB_16	HILL0092.D	01003-07-08	75.1 ng/mL PFOS	PFOS	5.304	1963056	75.6	101%
CALIB_17	HILL0093.D	01003-07-09	100.2 ng/mL PFOS	PFOS	5.299	2594831	100.2	100%
CALIB_18	HILL0094.D	01003-07-10	250.5 ng/mL PFOS	PFOS	5.312	6013719	255.9	102%
CALIB_19	HILL0095.D	01003-07-11	400.8 ng/mL PFOS	PFOS	5.3	9177063	406.4	101%
CALIB_20	HILL0096.D	01003-07-12	501 ng/mL PFOS	PFOS	5.319	11174689	508.1	101%
SAMPLE	HILL0097.D	01003-07-13	1002 ng/mL PFOS	PFOS	5.313	18959651	982.2	-

Two curves were averaged using Target software to quantify data. A standard curve range of only 10 levels is allowed. 5-500 ng/mL was the range chosen.

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 2 and 3; Analytical Instrument: HPLC/MSD "Hillary";

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amt (ng/mL)	Sample Purity corrected ng/mL	Average µg/mL Std Dev %RSD Spike Recovery
SAMPLE	HILL0031.D	042301-PFOS-LCS		PFOS	5.305	2196915	89.3	87	*89%*
SAMPLE	HILL0032.D	PFOS-2.4.1blk	042301-PFOS-2.4.1 Mtd Blk	PFOS	0	0	0.0		
SAMPLE	HILL0033.D	PFOS-2.4.2blk	042301-PFOS-2.4.2 Mtd Blk	PFOS	0	0	0.0		
SAMPLE	HILL0034.D	PFOS-2.4.3blk	042301-PFOS-2.4.3 Mtd Blk	PFOS	0	0	0.0		
SAMPLE	HILL0035.D	PFOS-2.4.4blkMS	042301-PFOS-2.4.4 Mtd Blk Spike	PFOS	5.305	2461943	99.1	97	*99%*
SAMPLE	HILL0037.D	PFOS-2.1.1	042301-PFOS-2.1.1 Smp	PFOS	5.235	7481346	308.4	302	322
SAMPLE	HILL0038.D	PFOS-2.1.2	042301-PFOS-2.1.2 Dup	PFOS	5.242	9178901	359.3	352	26.5
SAMPLE	HILL0039.D	PFOS-2.1.3	042301-PFOS-2.1.3 Trip	PFOS	5.242	8215875	319.1	312	8%
SAMPLE	HILL0040.D	PFOS-2.1.4MS	042301-PFOS-2.1.4 Spike	PFOS	5.237	9849022	401.7	393	*72%*
SAMPLE	HILL0045.D	PFOS-2.2.1	042301-PFOS-2.2.1 Smp	PFOS	0	0	0.0		
SAMPLE	HILL0046.D	PFOS-2.2.2	042301-PFOS-2.2.2 Dup	PFOS	0	0	0.0		
SAMPLE	HILL0047.D	PFOS-2.2.3	042301-PFOS-2.2.3 Trip	PFOS	0	0	0.0		
SAMPLE	HILL0048.D	PFOS-2.2.4MS	042301-PFOS-2.2.4 Spike	PFOS	5.305	2441252	99.8	98	*100%*
SAMPLE	HILL0050.D	PFOS-2.3.1	042301-PFOS-2.3.1 Smp	PFOS	0	0	0.0		
SAMPLE	HILL0051.D	PFOS-2.3.2	042301-PFOS-2.3.2 Dup	PFOS	0	0	0.0		
SAMPLE	HILL0052.D	PFOS-2.3.3	042301-PFOS-2.3.3 Trip	PFOS	0	0	0.0		
SAMPLE	HILL0053.D	PFOS-2.3.4MS	042301-PFOS-2.3.4 Spike	PFOS	5.306	2368154	95.4	93	*95%*
SAMPLE	HILL0058.D	042301-PFOS-LCS	PFOS Solvent Blank Spike	PFOS	5.305	2208565	89.5	88	*90%*
SAMPLE	HILL0059.D	PFOS-3.4.1blk	042301-PFOS-3.4.1 Mtd Blk	PFOS	0	0	0.0		
SAMPLE	HILL0060.D	PFOS-3.4.2blk	042301-PFOS-3.4.2 Mtd Blk	PFOS	0	0	0.0		
SAMPLE	HILL0061.D	PFOS-3.4.3blk	042301-PFOS-3.4.3 Mtd Blk	PFOS	0	0	0.0		
SAMPLE	HILL0062.D	PFOS-3.4.4blkMS	042301-PFOS-3.4.4 Mtd Blk Spike	PFOS	5.313	2459314	98.4	96	*98%*
SAMPLE	HILL0064.D	PFOS-3.1.1	042301-PFOS-3.1.1 Smp	PFOS	5.237	6815087	267.6	262	269
SAMPLE	HILL0065.D	PFOS-3.1.2	042301-PFOS-3.1.2 Dup	PFOS	5.242	7544482	292.7	287	15.4
SAMPLE	HILL0066.D	PFOS-3.1.3	042301-PFOS-3.1.3 Trip	PFOS	5.242	6794540	265.0	259	6%
SAMPLE	HILL0067.D	PFOS-3.1.4MS	042301-PFOS-3.1.4 Spike	PFOS	5.247	8532934	340.5	333	*65%*
SAMPLE	HILL0072.D	PFOS-3.2.1	042301-PFOS-3.2.1 Smp	PFOS	5.245	8117378	308.4	302	322
SAMPLE	HILL0073.D	PFOS-3.2.2	042301-PFOS-3.2.2 Dup	PFOS	5.249	8786445	359.3	352	26.5
SAMPLE	HILL0074.D	PFOS-3.2.3	042301-PFOS-3.2.3 Trip	PFOS	5.251	9095599	319.1	312	8%
SAMPLE	HILL0075.D	PFOS-3.2.4MS	042301-PFOS-3.2.4 Spike	PFOS	5.237	11117925	401.7	393	*72%*
SAMPLE	HILL0077.D	PFOS-3.3.1	042301-PFOS-3.3.1 Smp	PFOS	5.238	7558687	292.4	286	271
SAMPLE	HILL0078.D	PFOS-3.3.2	042301-PFOS-3.3.2 Dup	PFOS	5.243	6689145	262.7	257	14.5
SAMPLE	HILL0079.D	PFOS-3.3.3	042301-PFOS-3.3.3 Trip	PFOS	5.234	7103605	275.7	270	5%
SAMPLE	HILL0080.D	PFOS-3.3.4MS	042301-PFOS-3.3.4 Spike	PFOS	5.238	9203477	369.2	361	*92%*

	Day 3 results
average	287
std dev	30
%RSD:	10.4%

3M Environmental Laboratory
Project # E01-0768

Solubility Study, Shake Flask Method; Test Compound: PFOS; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 2 and 3; Analytical Instrument: HPLC/MSD "Hillary";

SAMPLE	HILL0014.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.398	27635	Below LOQ
SAMPLE	HILL0015.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0029.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0030.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0036.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0043.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0044.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0049.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0056.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0057.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0063.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0070.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0071.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0076.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.333	30130	Below LOQ
SAMPLE	HILL0083.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0084.D	TN-A 4802 MeOH	Solvent Blank	PFOS	0	0	0
SAMPLE	HILL0098.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.424	47439	Below LOQ
SAMPLE	HILL0099.D	TN-A 4802 MeOH	Solvent Blank	PFOS	5.404	15154	Below LOQ

BACK TO MAIN

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Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/FOSA0425.m (ChemStation);
Reprocessing Method: H010426a.m (Target)

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amount	Compound Name	RT	Area	Amount
SAMPLE	HILL0105.D	01003-25-01	0 Std	THPFOS	5.123	1465515	254	PFBS	4.394	2007446	254
SAMPLE	HILL0106.D	01003-25-02	2.5 ng/mL FOSA	THPFOS	5.111	1447576	254	PFBS	4.39	2027800	254
CALIB_1 XX	HILL0107.D	01003-25-03	5.3 ng/mL FOSA	THPFOS	5.116	1439545	254	PFBS	4.395	2026487	254
CALIB_2	HILL0108.D	01003-25-04	10.1 ng/mL FOSA	THPFOS	5.116	1445222	254	PFBS	4.401	2021996	254
CALIB_3	HILL0109.D	01003-25-05	25 ng/mL FOSA	THPFOS	5.109	1441747	254	PFBS	4.387	2015250	254
CALIB_4	HILL0110.D	01003-25-06	40.3 ng/mL FOSA	THPFOS	5.118	1427400	254	PFBS	4.389	2007486	254
CALIB_5	HILL0111.D	01003-25-07	49.9 ng/mL FOSA	THPFOS	5.115	1441987	254	PFBS	4.386	2028225	254
CALIB_6	HILL0112.D	01003-25-08	74.9 ng/mL FOSA	THPFOS	5.116	1447199	254	PFBS	4.395	2012838	254
CALIB_7	HILL0113.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.112	1441812	254	PFBS	4.39	1997221	254
CALIB_8	HILL0114.D	01003-25-10	249.6 ng/mL FOSA	THPFOS	5.115	1459549	254	PFBS	4.394	1999512	254
CALIB_9	HILL0115.D	01003-25-11	403.2 ng/mL FOSA	THPFOS	5.116	1432834	254	PFBS	4.395	2008100	254
CALIB_10	HILL0116.D	01003-25-12	499.2 ng/mL FOSA	THPFOS	5.112	1428416	254	PFBS	4.39	1999395	254
SAMPLE	HILL0117.D	01003-25-13	1008 ng/mL FOSA	THPFOS	5.116	1447369	254	PFBS	4.387	1988333	254
SAMPLE	HILL0131.D	01003-25-03	5.3 ng/mL FOSA	THPFOS	5.11	1265387	254	PFBS	4.381	1993211	254
SAMPLE	HILL0132.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.119	1298469	254	PFBS	4.39	1945472	254
SAMPLE	HILL0146.D	01003-25-03	5.3 ng/mL FOSA	THPFOS	5.11	1170043	254	PFBS	4.382	1974246	254
SAMPLE	HILL0147.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.111	1177767	254	PFBS	4.389	1924449	254
SAMPLE	HILL0161.D	01003-25-03	5.3 ng/mL FOSA	THPFOS	5.111	1145578	254	PFBS	4.389	1984007	254
SAMPLE	HILL0162.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.115	1120490	254	PFBS	4.393	1933728	254
SAMPLE	HILL0171.D	01003-25-01	0 Std	THPFOS	5.115	1122982	254	PFBS	4.394	1989610	254
SAMPLE	HILL0172.D	01003-25-02	2.5 ng/mL FOSA	THPFOS	5.116	1107259	254	PFBS	4.388	1975741	254
CALIB_11 XX	HILL0173.D	01003-25-03	5.3 ng/mL FOSA	THPFOS	5.118	1096459	254	PFBS	4.383	1962904	254
CALIB_12	HILL0174.D	01003-25-04	10.1 ng/mL FOSA	THPFOS	5.109	1125849	254	PFBS	4.388	1986462	254
CALIB_13	HILL0175.D	01003-25-05	25 ng/mL FOSA	THPFOS	5.115	1101993	254	PFBS	4.387	1965181	254
CALIB_14	HILL0176.D	01003-25-06	40.3 ng/mL FOSA	THPFOS	5.11	1104242	254	PFBS	4.389	1958152	254
CALIB_15	HILL0177.D	01003-25-07	49.9 ng/mL FOSA	THPFOS	5.109	1111672	254	PFBS	4.388	1969738	254
CALIB_16	HILL0178.D	01003-25-08	74.9 ng/mL FOSA	THPFOS	5.107	1107520	254	PFBS	4.386	1941113	254
CALIB_17	HILL0179.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.112	1113145	254	PFBS	4.391	1940454	254
CALIB_18	HILL0180.D	01003-25-10	249.6 ng/mL FOSA	THPFOS	5.124	1124167	254	PFBS	4.395	1955186	254
CALIB_19	HILL0181.D	01003-25-11	403.2 ng/mL FOSA	THPFOS	5.117	1114186	254	PFBS	4.389	1940675	254
CALIB_20	HILL0182.D	01003-25-12	499.2 ng/mL FOSA	THPFOS	5.123	1126570	254	PFBS	4.387	1933249	254
SAMPLE	HILL0183.D	01003-25-13	1008 ng/mL FOSA	THPFOS	5.119	1146487	254	PFBS	4.383	1964405	254
SAMPLE	HILL0120.D	042301-FOSA-LCS		THPFOS	5.109	1387790	254	PFBS	4.381	1875014	254
SAMPLE	HILL0121.D	FOSA-1.4.1blk		THPFOS	5.108	1379938	254	PFBS	4.38	1893699	254
SAMPLE	HILL0122.D	FOSA-1.4.2blk		THPFOS	5.115	1385495	254	PFBS	4.387	1872319	254
SAMPLE	HILL0123.D	FOSA-1.4.3blk		THPFOS	5.117	1413232	254	PFBS	4.381	1876934	254
SAMPLE	HILL0124.D	FOSA-1.4.4blkMS		THPFOS	5.116	1388787	254	PFBS	4.387	1861408	254
SAMPLE	HILL0126.D	FOSA-1.1.1		THPFOS	5.112	1223896	254	PFBS	4.391	1863247	254
SAMPLE	HILL0127.D	FOSA-1.1.2		THPFOS	5.112	1207970	254	PFBS	4.384	1889163	254
SAMPLE	HILL0128.D	FOSA-1.1.3		THPFOS	5.115	1198241	254	PFBS	4.387	1858991	254
SAMPLE	HILL0129.D	FOSA-1.1.4MS		THPFOS	5.11	1184556	254	PFBS	4.381	1860013	254
SAMPLE	HILL0130.D	FOSA-1.2.1		THPFOS	5.118	1223960	254	PFBS	4.383	1887871	254
SAMPLE	HILL0135.D	FOSA-1.2.2		THPFOS	5.108	1227152	254	PFBS	4.379	1901597	254
SAMPLE	HILL0136.D	FOSA-1.2.3		THPFOS	5.11	1210108	254	PFBS	4.375	1890574	254
SAMPLE	HILL0137.D	FOSA-1.2.4MS		THPFOS	5.11	1202734	254	PFBS	4.382	1875132	254

Exact Copy of Original

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3M Environmental Laboratory
Project # E01-0768

Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/FOSA0425.m (ChemStation);
Reprocessing Method: H010426a.m (Target)

SAMPLE	HILL0138.D	FOSA-1.3.1	THPFOS	5.112	1177514	254	PFBS	4.39	1900215	254
SAMPLE	HILL0139.D	FOSA-1.3.2	THPFOS	5.109	1188089	254	PFBS	4.381	1882956	254
SAMPLE	HILL0141.D	FOSA-1.3.3	THPFOS	5.116	1173989	254	PFBS	4.381	1853824	254
SAMPLE	HILL0142.D	FOSA-1.3.4MS	THPFOS	5.116	1179158	254	PFBS	4.388	1873732	254
SAMPLE	HILL0143.D	042301-FOSA-LCS	THPFOS	5.116	1152061	254	PFBS	4.395	1836551	254
SAMPLE	HILL0144.D	FOSA-2.4.1blk	THPFOS	5.112	1308838	254	PFBS	4.383	1875879	254
SAMPLE	HILL0145.D	FOSA-2.4.2blk	THPFOS	5.115	1332634	254	PFBS	4.386	1889627	254
SAMPLE	HILL0150.D	FOSA-2.4.3blk	THPFOS	5.109	1307643	254	PFBS	4.38	1868213	254
SAMPLE	HILL0151.D	FOSA-2.4.4blkMS	THPFOS	5.116	1312788	254	PFBS	4.387	1876885	254
SAMPLE	HILL0152.D	FOSA-2.1.1	THPFOS	5.116	1110585	254	PFBS	4.387	1783250	254
SAMPLE	HILL0153.D	FOSA-2.1.2	THPFOS	5.109	1194990	254	PFBS	4.388	1910331	254
SAMPLE	HILL0154.D	FOSA-2.1.3	THPFOS	5.116	1181747	254	PFBS	4.388	1870518	254
SAMPLE	HILL0156.D	FOSA-2.1.4MS	THPFOS	5.116	1165222	254	PFBS	4.388	1853384	254
SAMPLE	HILL0157.D	FOSA-2.2.1	THPFOS	5.116	1202436	254	PFBS	4.388	1863768	254
SAMPLE	HILL0158.D	FOSA-2.2.2	THPFOS	5.115	1233448	254	PFBS	4.387	1900272	254
SAMPLE	HILL0159.D	FOSA-2.2.3	THPFOS	5.109	1228256	254	PFBS	4.388	1907383	254
SAMPLE	HILL0160.D	FOSA-2.2.4MS	THPFOS	5.11	1224603	254	PFBS	4.389	1899528	254
SAMPLE	HILL0165.D	FOSA-2.3.1	THPFOS	5.116	1158902	254	PFBS	4.395	1883593	254
SAMPLE	HILL0166.D	FOSA-2.3.2	THPFOS	5.111	1162327	254	PFBS	4.39	1866181	254
SAMPLE	HILL0167.D	FOSA-2.3.3	THPFOS	5.117	1177491	254	PFBS	4.388	1891600	254
SAMPLE	HILL0168.D	FOSA-2.3.4MS	THPFOS	5.116	1181915	254	PFBS	4.387	1875392	254
				Average:	1249014				1926471	
				Std Deviation:	125832				60064	
				%RSD:	10.1%				3.1%	
SAMPLE	HILL0103.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.426	10752	0
SAMPLE	HILL0104.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0118.D	TN-A 4802 MeOH	THPFOS	5.187	8097	0	PFBS	4.403	9817	0
SAMPLE	HILL0119.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0125.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.41	10811	0
SAMPLE	HILL0133.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.409	10138	0
SAMPLE	HILL0134.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.408	7466	0
SAMPLE	HILL0140.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.403	11533	0
SAMPLE	HILL0148.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.41	10126	0
SAMPLE	HILL0149.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0155.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.41	10883	0
SAMPLE	HILL0163.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.417	9378	0
SAMPLE	HILL0164.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0169.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.403	9038	0
SAMPLE	HILL0170.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0184.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	4.415	9491	0
SAMPLE	HILL0185.D	TN-A 4802 MeOH	THPFOS	0	0	0	PFBS	0	0	0

Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/FOSA0425.m (ChemStation);
Reprocessing Method: H010426a.m (Target)

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amount	% Recovery
SAMPLE	HILL0105.D	01003-25-01	0 Std	FOSA	0	0	0.0	Excluded
SAMPLE	HILL0106.D	01003-25-02	2.5 ng/mL FOSA	FOSA	5.973	92513	1.8	Excluded
CALIB_1 XX	HILL0107.D	01003-25-03	5.3 ng/mL FOSA	FOSA	5.977	175825	4.5	Excluded
CALIB_2	HILL0108.D	01003-25-04	10.1 ng/mL FOSA	FOSA	5.977	333744	9.5	94.1%
CALIB_3	HILL0109.D	01003-25-05	25 ng/mL FOSA	FOSA	5.977	832878	25.6	102.4%
CALIB_4	HILL0110.D	01003-25-06	40.3 ng/mL FOSA	FOSA	5.979	1346447	42.5	105.4%
CALIB_5	HILL0111.D	01003-25-07	49.9 ng/mL FOSA	FOSA	5.976	1622197	51.0	102.2%
CALIB_6	HILL0112.D	01003-25-08	74.9 ng/mL FOSA	FOSA	5.978	2412994	77.6	103.6%
CALIB_7	HILL0113.D	01003-25-09	100.8 ng/mL FOSA	FOSA	5.98	3249134	106.6	105.8%
CALIB_8	HILL0114.D	01003-25-10	249.6 ng/mL FOSA	FOSA	5.976	7439185	255.5	102.4%
CALIB_9	HILL0115.D	01003-25-11	403.2 ng/mL FOSA	FOSA	5.978	11510014	409.4	101.5%
CALIB_10	HILL0116.D	01003-25-12	499.2 ng/mL FOSA	FOSA	5.973	13881086	507.2	101.6%
SAMPLE	HILL0117.D	01003-25-13	1008 ng/mL FOSA	FOSA	5.977	24702641	998.1	Excluded
SAMPLE	HILL0131.D	01003-25-03	5.3 ng/mL FOSA	FOSA	5.971	200577	5.3	Excluded
SAMPLE	HILL0132.D	01003-25-09	100.8 ng/mL FOSA	FOSA	5.973	3024244	101.7	100.9%
SAMPLE	HILL0146.D	01003-25-03	5.3 ng/mL FOSA	FOSA	5.965	157381	4.0	Excluded
SAMPLE	HILL0147.D	01003-25-09	100.8 ng/mL FOSA	FOSA	5.965	2919745	99.1	98.4%
SAMPLE	HILL0161.D	01003-25-03	5.3 ng/mL FOSA	FOSA	5.972	190625	5.1	Excluded
SAMPLE	HILL0162.D	01003-25-09	100.8 ng/mL FOSA	FOSA	5.969	2792405	94.2	93.4%
SAMPLE	HILL0171.D	01003-25-01	0 Std	FOSA	0	0	0.0	Excluded
SAMPLE	HILL0172.D	01003-25-02	2.5 ng/mL FOSA	FOSA	5.964	75933	1.4	Excluded
CALIB_11 XX	HILL0173.D	01003-25-03	5.3 ng/mL FOSA	FOSA	5.973	152433	3.9	Excluded
CALIB_12	HILL0174.D	01003-25-04	10.1 ng/mL FOSA	FOSA	5.971	297834	8.5	84.4%
CALIB_13	HILL0175.D	01003-25-05	25 ng/mL FOSA	FOSA	5.969	743034	23.3	93.2%
CALIB_14	HILL0176.D	01003-25-06	40.3 ng/mL FOSA	FOSA	5.958	1216120	39.2	97.3%
CALIB_15	HILL0177.D	01003-25-07	49.9 ng/mL FOSA	FOSA	5.964	1485755	48.0	96.1%
CALIB_16	HILL0178.D	01003-25-08	74.9 ng/mL FOSA	FOSA	5.969	2183173	72.6	97.0%
CALIB_17	HILL0179.D	01003-25-09	100.8 ng/mL FOSA	FOSA	5.966	2971186	100.1	99.3%
CALIB_18	HILL0180.D	01003-25-10	249.6 ng/mL FOSA	FOSA	5.971	6959290	243.7	97.6%
CALIB_19	HILL0181.D	01003-25-11	403.2 ng/mL FOSA	FOSA	5.972	10720207	393.0	97.5%
CALIB_20	HILL0182.D	01003-25-12	499.2 ng/mL FOSA	FOSA	5.977	13077641	492.6	98.7%
SAMPLE	HILL0183.D	01003-25-13	1008 ng/mL FOSA	FOSA	5.973	23820299	969.4	Excluded

** Only 10 levels may be quantified due to software limitations, therefore 5-500 ng/mL was chosen. However, since not all blank samples had a FOSA response of less than half the 5 ng/mL response, the 5 ng/mL standard was disabled and the LOQ becam

Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/FOSA0425.m (ChemStation);
Reprocessing Method: H010426a.m (Target)

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	on-column Amount	dil factor corrected µg/mL FOSA	Caluculations Average (µg/mL) Std Dev % RSD *Spike % Recov.*		
SAMPLE	HILL0120.D	042301-FOSA-LCS	FOSA Solvent Blank Spike	FOSA	5.971	3056177	107		*111%*		
SAMPLE	HILL0121.D	FOSA-1.4.1blk	042301-FOSA-1.4.1 Mtd Blk	FOSA	5.893	106755	3	0.00			
SAMPLE	HILL0122.D	FOSA-1.4.2blk	042301-FOSA-1.4.2 Mtd Blk	FOSA	0	0	0	0.00			
SAMPLE	HILL0123.D	FOSA-1.4.3blk	042301-FOSA-1.4.3 Mtd Blk	FOSA	5.894	135380	4	0.04			
SAMPLE	HILL0124.D	FOSA-1.4.4blkMS	42301-FOSA-1.4.4 Mtd Blk Spike	FOSA	5.977	2921968	103		*107%*		
SAMPLE	HILL0126.D	~~FOSA-1.1.1~~	042301-FOSA-1.1.1 Smp	~~FOSA~~	~~5.89~~	~~9090670~~	~~343~~	~~3.43~~	2.60	2.18	
SAMPLE	HILL0127.D	FOSA-1.1.2	042301-FOSA-1.1.2 Dup	FOSA	5.89	6180725	223	2.23	0.7	4.4%	Rep2,3 RPD
SAMPLE	HILL0128.D	FOSA-1.1.3	042301-FOSA-1.1.3 Trip	FOSA	5.886	5838914	213	2.13	28%		
SAMPLE	HILL0129.D	FOSA-1.1.4MS	042301-FOSA-1.1.4 Spike	FOSA	5.887	8313598	312	3.12	*98%*	*-based on reps 2&3 only*	
SAMPLE	HILL0130.D	FOSA-1.2.1	042301-FOSA-1.2.1 Smp	FOSA	5.889	8385467	309	3.09	3.11		
SAMPLE	HILL0135.D	FOSA-1.2.2	042301-FOSA-1.2.2 Dup	FOSA	5.885	8541720	313	3.13	0.0		
SAMPLE	HILL0136.D	FOSA-1.2.3	042301-FOSA-1.2.3 Trip	FOSA	5.888	8450504	312	3.12	1%		Day 1 results, µg/mL
SAMPLE	HILL0137.D	FOSA-1.2.4MS	042301-FOSA-1.2.4 Spike	FOSA	5.887	10811323	412	4.12	*105%*		average 2.56
SAMPLE	HILL0138.D	FOSA-1.3.1	042301-FOSA-1.3.1 Smp	FOSA	5.882	6883587	248	2.48	2.37		std dev 0.38
SAMPLE	HILL0139.D	FOSA-1.3.2	042301-FOSA-1.3.2 Dup	FOSA	5.886	6433005	233	2.33	0.1		%RSD: 15%
SAMPLE	HILL0141.D	FOSA-1.3.3	042301-FOSA-1.3.3 Trip	FOSA	5.886	6255010	230	2.30	4%		
SAMPLE	HILL0142.D	FOSA-1.3.4MS	042301-FOSA-1.3.4 Spike	FOSA	5.886	9067750	340	3.40	*107%*		
SAMPLE	HILL0143.D	042301-FOSA-LCS	FOSA Solvent Blank Spike	FOSA	5.97	2756837	98		*102%*		
SAMPLE	HILL0144.D	FOSA-2.4.1blk	042301-FOSA-2.4.1 Mtd Blk	FOSA	0	0	0				
SAMPLE	HILL0145.D	FOSA-2.4.2blk	042301-FOSA-2.4.2 Mtd Blk	FOSA	0	0	0				
SAMPLE	HILL0150.D	FOSA-2.4.3blk	042301-FOSA-2.4.3 Mtd Blk	FOSA	0	0	0				
SAMPLE	HILL0151.D	FOSA-2.4.4blkMS	42301-FOSA-2.4.4 Mtd Blk Spike	FOSA	5.97	2811726	98		*102%*		
SAMPLE	HILL0152.D	FOSA-2.1.1	042301-FOSA-2.1.1 Smp	FOSA	5.879	8103858	317	3.17	3.02		
SAMPLE	HILL0153.D	FOSA-2.1.2	042301-FOSA-2.1.2 Dup	FOSA	5.879	8151376	296	2.96	0.1		
SAMPLE	HILL0154.D	FOSA-2.1.3	042301-FOSA-2.1.3 Trip	FOSA	5.88	7853370	291	2.91	5%		
SAMPLE	HILL0156.D	FOSA-2.1.4MS	042301-FOSA-2.1.4 Spike	FOSA	5.887	10247004	393	3.93	*96%*		
SAMPLE	HILL0157.D	FOSA-2.2.1	042301-FOSA-2.2.1 Smp	FOSA	5.887	7993752	298	2.98	2.82		
SAMPLE	HILL0158.D	FOSA-2.2.2	042301-FOSA-2.2.2 Dup	FOSA	5.886	7697039	280	2.80	0.1		
SAMPLE	HILL0159.D	FOSA-2.2.3	042301-FOSA-2.2.3 Trip	FOSA	5.887	7443513	269	2.69	5%		Day 2 results, µg/mL
SAMPLE	HILL0160.D	FOSA-2.2.4MS	042301-FOSA-2.2.4 Spike	FOSA	5.888	9662387	359	3.59	*80%*		average 2.88
SAMPLE	HILL0165.D	FOSA-2.3.1	042301-FOSA-2.3.1 Smp	FOSA	5.879	7850050	289	2.89	2.81		std dev 0.12
SAMPLE	HILL0166.D	FOSA-2.3.2	042301-FOSA-2.3.2 Dup	FOSA	5.882	7536620	279	2.79	0.1		%RSD: 4%
SAMPLE	HILL0167.D	FOSA-2.3.3	042301-FOSA-2.3.3 Trip	FOSA	5.88	7535404	275	2.75	3%		
SAMPLE	HILL0168.D	FOSA-2.3.4MS	042301-FOSA-2.3.4 Spike	FOSA	5.886	9615944	362	3.62	*84%*		

Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Days 1 and 2
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010426.s/FOSA0425.m (ChemStation);
Reprocessing Method: H010426a.m (Target)

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amount
SAMPLE	HILL0103.D	TN-A 4802 MeOH		FOSA	5.981	143948	<1/2 LOQ resp
SAMPLE	HILL0104.D	TN-A 4802 MeOH		FOSA	5.976	47424	<1/2 LOQ resp
SAMPLE	HILL0118.D	TN-A 4802 MeOH		FOSA	5.979	129092	<1/2 LOQ resp
SAMPLE	HILL0119.D	TN-A 4802 MeOH		FOSA	5.978	53533	<1/2 LOQ resp
SAMPLE	HILL0125.D	TN-A 4802 MeOH		FOSA	0	0	<1/2 LOQ resp
SAMPLE	HILL0133.D	TN-A 4802 MeOH		FOSA	5.978	24760	<1/2 LOQ resp
SAMPLE	HILL0134.D	TN-A 4802 MeOH		FOSA	0	0	<1/2 LOQ resp
SAMPLE	HILL0140.D	TN-A 4802 MeOH		FOSA	5.972	40478	<1/2 LOQ resp
SAMPLE	HILL0148.D	TN-A 4802 MeOH		FOSA	0	0	<1/2 LOQ resp
SAMPLE	HILL0149.D	TN-A 4802 MeOH		FOSA	0	0	<1/2 LOQ resp
SAMPLE	HILL0155.D	TN-A 4802 MeOH		FOSA	5.972	38428	<1/2 LOQ resp
SAMPLE	HILL0163.D	TN-A 4802 MeOH		FOSA	0	0	<1/2 LOQ resp
SAMPLE	HILL0164.D	TN-A 4802 MeOH		FOSA	0	0	<1/2 LOQ resp
SAMPLE	HILL0169.D	TN-A 4802 MeOH		FOSA	5.986	48316	<1/2 LOQ resp
SAMPLE	HILL0170.D	TN-A 4802 MeOH		FOSA	5.984	17972	<1/2 LOQ resp
SAMPLE	HILL0184.D	TN-A 4802 MeOH		FOSA	5.977	136896	<1/2 LOQ resp
SAMPLE	HILL0185.D	TN-A 4802 MeOH		FOSA	5.971	49865	<1/2 LOQ resp

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Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 3
***Analytical Instrument:* HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/FOSA0425.m (ChemStation);**
Reprocessing Method: H010501a.m (Target)

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amount	Compound Name	RT	Area	Amount
SAMPLE	HILL0078.D	01003-25-01	0 Std	THPFOS	5.115	1381461	254	PFBS	4.408	1798374	254
SAMPLE	HILL0079.D	01003-25-02	2.5 ng/mL FOSA	THPFOS	5.116	1374933	254	PFBS	4.401	1777751	254
CALIB_1	HILL0080.D	01003-25-03	5.3 ng/mL FOSA	THPFOS	5.117	1393833	254	PFBS	4.41	1801694	254
CALIB_2	HILL0081.D	01003-25-04	10.1 ng/mL FOSA	THPFOS	5.117	1385169	254	PFBS	4.416	1810322	254
CALIB_3	HILL0082.D	01003-25-05	25 ng/mL FOSA	THPFOS	5.113	1380163	254	PFBS	4.42	1800005	254
CALIB_4	HILL0083.D	01003-25-06	40.3 ng/mL FOSA	THPFOS	5.116	1376007	254	PFBS	4.408	1780106	254
CALIB_5	HILL0084.D	01003-25-07	49.9 ng/mL FOSA	THPFOS	5.129	1374674	254	PFBS	4.407	1812881	254
CALIB_6	HILL0085.D	01003-25-08	74.9 ng/mL FOSA	THPFOS	5.11	1374202	254	PFBS	4.41	1766585	254
CALIB_7	HILL0086.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.123	1380970	254	PFBS	4.423	1798232	254
CALIB_8	HILL0087.D	01003-25-10	249.6 ng/mL FOSA	THPFOS	5.111	1382975	254	PFBS	4.411	1773614	254
CALIB_9	HILL0088.D	01003-25-11	403.2 ng/mL FOSA	THPFOS	5.115	1394460	254	PFBS	4.401	1805937	254
CALIB_10	HILL0089.D	01003-25-12	499.2 ng/mL FOSA	THPFOS	5.115	1388280	254	PFBS	4.414	1776350	254
SAMPLE	HILL0090.D	01003-25-13	1008 ng/mL FOSA	THPFOS	5.122	1389397	254	PFBS	4.414	1788258	254
SAMPLE	HILL0104.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.117	1244044	254	PFBS	4.402	1778858	254
SAMPLE	HILL0116.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.125	1247230	254	PFBS	4.41	1778060	254
SAMPLE	HILL0119.D	01003-25-01	0 Std	THPFOS	5.115	1266027	254	PFBS	4.415	1789621	254
SAMPLE	HILL0120.D	01003-25-02	2.5 ng/mL FOSA	THPFOS	5.123	1262927	254	PFBS	4.415	1775266	254
CALIB_11	HILL0121.D	01003-25-03	5.3 ng/mL FOSA	THPFOS	5.126	1263723	254	PFBS	4.418	1808173	254
CALIB_12	HILL0122.D	01003-25-04	10.1 ng/mL FOSA	THPFOS	5.114	1262166	254	PFBS	4.421	1804070	254
CALIB_13	HILL0123.D	01003-25-05	25 ng/mL FOSA	THPFOS	5.122	1280362	254	PFBS	4.414	1774497	254
CALIB_14	HILL0124.D	01003-25-06	40.3 ng/mL FOSA	THPFOS	5.115	1275643	254	PFBS	4.415	1778220	254
CALIB_15	HILL0125.D	01003-25-07	49.9 ng/mL FOSA	THPFOS	5.131	1276350	254	PFBS	4.416	1785753	254
CALIB_16	HILL0126.D	01003-25-08	74.9 ng/mL FOSA	THPFOS	5.124	1274208	254	PFBS	4.424	1775648	254
CALIB_17	HILL0127.D	01003-25-09	100.8 ng/mL FOSA	THPFOS	5.12	1269909	254	PFBS	4.419	1757613	254
CALIB_18	HILL0128.D	01003-25-10	249.6 ng/mL FOSA	THPFOS	5.119	1287101	254	PFBS	4.412	1767480	254
CALIB_19	HILL0129.D	01003-25-11	403.2 ng/mL FOSA	THPFOS	5.117	1292619	254	PFBS	4.416	1778677	254
CALIB_20	HILL0130.D	01003-25-12	499.2 ng/mL FOSA	THPFOS	5.118	1288469	254	PFBS	4.411	1776855	254
SAMPLE	HILL0131.D	01003-25-13	1008 ng/mL FOSA	THPFOS	5.115	1293221	254	PFBS	4.422	1781355	254

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Initial

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Date

Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 3
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/FOSA0425.m (ChemStation);

SAMPLE	HILL0093.D	FOSA-3.4.1blk	042301-FOSA-3.4.1, Mtd Blk	THPFOS	5.118	1236810	254	PFBS	4.396	1629186	254
SAMPLE	HILL0094.D	FOSA-3.4.2blk	042301-FOSA-3.4.2, Mtd Blk	THPFOS	5.123	1299549	254	PFBS	4.402	1708821	254
SAMPLE	HILL0095.D	FOSA-3.4.3blk	042301-FOSA-3.4.3, Mtd Blk	THPFOS	5.124	1331679	254	PFBS	4.41	1727981	254
SAMPLE	HILL0096.D	FOSA-3.4.4blkMS	042301-FOSA-3.4.4, Mtd Blk Spike	THPFOS	5.123	1313977	254	PFBS	4.416	1714291	254
SAMPLE	HILL0097.D	FOSA-3.1.1	042301-FOSA-3.1.1, Smp	THPFOS	5.118	1186398	254	PFBS	4.403	1735419	254
SAMPLE	HILL0099.D	FOSA-3.1.2	042301-FOSA-3.1.2, Dup	THPFOS	5.117	1180945	254	PFBS	4.41	1725206	254
SAMPLE	HILL0100.D	FOSA-3.1.3	042301-FOSA-3.1.3, Trip	THPFOS	5.124	1174802	254	PFBS	4.409	1738131	254
SAMPLE	HILL0101.D	FOSA-3.2.1	042301-FOSA-3.2.1, Smp	THPFOS	5.117	1170460	254	PFBS	4.409	1757667	254
SAMPLE	HILL0102.D	FOSA-3.2.2	042301-FOSA-3.2.2, Dup	THPFOS	5.117	1157352	254	PFBS	4.402	1733154	254
SAMPLE	HILL0103.D	FOSA-3.2.3	042301-FOSA-3.2.3, Trip	THPFOS	5.118	1169370	254	PFBS	4.418	1755402	254
SAMPLE	HILL0107.D	FOSA-3.2.4MS	042301-FOSA-3.2.4, Spike	THPFOS	5.116	1142996	254	PFBS	4.408	1710176	254
SAMPLE	HILL0108.D	FOSA-3.3.1	042301-FOSA-3.3.1, Smp	THPFOS	5.124	1120852	254	PFBS	4.403	1739019	254
SAMPLE	HILL0109.D	FOSA-3.3.2	042301-FOSA-3.3.2, Dup	THPFOS	5.128	1115271	254	PFBS	4.407	1748703	254
SAMPLE	HILL0110.D	FOSA-3.3.3	042301-FOSA-3.3.3, Trip	THPFOS	5.123	1120639	254	PFBS	4.415	1743829	254
SAMPLE	HILL0112.D	FOSA-3.3.4MS	042301-FOSA-3.3.4, Spike	THPFOS	5.111	1102414	254	PFBS	4.404	1709063	254
SAMPLE	HILL0113.D	042301-FOSA-LCS	FOSA Solvent Blank Spike	THPFOS	5.13	1168358	254	PFBS	4.415	1691538	254
SAMPLE	HILL0114.D	Diluter Blk	Diluter blank with IS mix	THPFOS	5.122	1184946	254	PFBS	4.415	1645960	254
SAMPLE	HILL0115.D	MeOH blk w/ IS	Methanol with IS mix	THPFOS	5.115	809160	254	PFBS	4.414	1075206	254
					Average:	1262315				1745413	
					Std Deviation:	112912				108834	
					%RSD:	8.9%				6.2%	
SAMPLE	HILL0077.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0091.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0092.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0098.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0105.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0106.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0111.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0117.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0118.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0132.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0
SAMPLE	HILL0133.D	TN-A 4802 MeOH		THPFOS	0	0	0	PFBS	0	0	0

Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 3
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/FOSA0425.m (ChemStation);

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amount	%Recovery
SAMPLE	HILL0078.D	01003-25-01	0 Std	FOSA	0	0	0	-
SAMPLE	HILL0079.D	01003-25-02	2.5 ng/mL FOSA	FOSA	5.991	84044	3	-
CALIB_1	HILL0080.D	01003-25-03	5.3 ng/mL FOSA	FOSA	5.993	160849	5	-
CALIB_2	HILL0081.D	01003-25-04	10.1 ng/mL FOSA	FOSA	5.985	307301	10	97%
CALIB_3	HILL0082.D	01003-25-05	25 ng/mL FOSA	FOSA	5.982	768189	25	100%
CALIB_4	HILL0083.D	01003-25-06	40.3 ng/mL FOSA	FOSA	5.984	1231657	41	102%
CALIB_5	HILL0084.D	01003-25-07	49.9 ng/mL FOSA	FOSA	5.997	1519482	50	100%
CALIB_6	HILL0085.D	01003-25-08	74.9 ng/mL FOSA	FOSA	5.985	2227975	76	101%
CALIB_7	HILL0086.D	01003-25-09	100.8 ng/mL FOSA	FOSA	5.985	3036314	103	102%
CALIB_8	HILL0087.D	01003-25-10	249.6 ng/mL FOSA	FOSA	5.98	6971036	253	101%
CALIB_9	HILL0088.D	01003-25-11	403.2 ng/mL FOSA	FOSA	6.004	10819514	405	101%
CALIB_10	HILL0089.D	01003-25-12	499.2 ng/mL FOSA	FOSA	5.983	12885941	505	101%
SAMPLE	HILL0090.D	01003-25-13	1008 ng/mL FOSA	FOSA	5.983	23137645	1018	-
SAMPLE	HILL0104.D	01003-25-09	100.8 ng/mL FOSA	FOSA	5.999	2915113	100	102%
SAMPLE	HILL0116.D	01003-25-09	100.8 ng/mL FOSA	FOSA	6	2902871	99	102%
SAMPLE	HILL0119.D	01003-25-01	0 Std	FOSA	0	0	0	-
SAMPLE	HILL0120.D	01003-25-02	2.5 ng/mL FOSA	FOSA	5.984	72740	2	-
CALIB_11	HILL0121.D	01003-25-03	5.3 ng/mL FOSA	FOSA	5.987	152363	5	-
CALIB_12	HILL0122.D	01003-25-04	10.1 ng/mL FOSA	FOSA	5.983	298779	10	95%
CALIB_13	HILL0123.D	01003-25-05	25 ng/mL FOSA	FOSA	5.99	736231	24	97%
CALIB_14	HILL0124.D	01003-25-06	40.3 ng/mL FOSA	FOSA	5.984	1203821	40	99%
CALIB_15	HILL0125.D	01003-25-07	49.9 ng/mL FOSA	FOSA	5.999	1473022	49	98%
CALIB_16	HILL0126.D	01003-25-08	74.9 ng/mL FOSA	FOSA	5.986	2181078	74	99%
CALIB_17	HILL0127.D	01003-25-09	100.8 ng/mL FOSA	FOSA	5.988	2931206	101	101%
CALIB_18	HILL0128.D	01003-25-10	249.6 ng/mL FOSA	FOSA	5.988	6818287	248	99%
CALIB_19	HILL0129.D	01003-25-11	403.2 ng/mL FOSA	FOSA	5.985	10436918	396	98%
CALIB_20	HILL0130.D	01003-25-12	499.2 ng/mL FOSA	FOSA	5.994	12664603	495	99%
SAMPLE	HILL0131.D	01003-25-13	1008 ng/mL FOSA	FOSA	5.984	22575515	992	-

Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 3
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/FOSA0425.m (ChemStation);

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	on-column Amount	dil factor corrected µg/mL FOSA	Caluculations Average (µg/mL) Std Dev % RSD Spike % Recov.
SAMPLE	HILL0097.D	FOSA-3.1.1	042301-FOSA-3.1.1, Smp	FOSA	5.909	7201703	269	2.69	2.60
SAMPLE	HILL0099.D	FOSA-3.1.2	042301-FOSA-3.1.2, Dup	FOSA	5.901	6731194	251	2.51	0.1
SAMPLE	HILL0100.D	FOSA-3.1.3	042301-FOSA-3.1.3, Trip	FOSA	5.908	6998562	260	2.60	3%
Not enough sample was available to prepare the spike replicate for sample #1. Spike results from samples two and three shall suffice to demonstrate spike recoveries.									
SAMPLE	HILL0101.D	FOSA-3.2.1	042301-FOSA-3.2.1, Smp	FOSA	5.908	6990791	257	2.57	2.56
SAMPLE	HILL0102.D	FOSA-3.2.2	042301-FOSA-3.2.2, Dup	FOSA	5.922	7009317	261	2.61	0.1
SAMPLE	HILL0103.D	FOSA-3.2.3	042301-FOSA-3.2.3, Trip	FOSA	5.895	6836606	251	2.51	2%
SAMPLE	HILL0107.D	FOSA-3.2.4MS	042301-FOSA-3.2.4, Spike	FOSA	5.9	9016954	351	3.51	*99%*
SAMPLE	HILL0108.D	FOSA-3.3.1	042301-FOSA-3.3.1, Smp	FOSA	5.916	7065688	263	2.63	2.66
SAMPLE	HILL0109.D	FOSA-3.3.2	042301-FOSA-3.3.2, Dup	FOSA	5.906	7377514	274	2.74	0.1
SAMPLE	HILL0110.D	FOSA-3.3.3	042301-FOSA-3.3.3, Trip	FOSA	5.9	7030743	260	2.60	3%
SAMPLE	HILL0112.D	FOSA-3.3.4MS	042301-FOSA-3.3.4, Spike	FOSA	5.909	9073879	354	3.54	*92%*
SAMPLE	HILL0113.D	042301-FOSA-LCS	FOSA Solvent Blank Spike	FOSA	5.991	2750436	99		*103%*

Day 3 results, µg/mL
average 2.61
std dev 0.05
%RSD: 2%

3M Environmental Laboratory
Project # E01-0768

Solubility Study, Shake Flask Method; Test Compound: FOSA; Test Matrix: Male/Female Normal Human Urine; Timepoint(s): Day 3
Analytical Instrument: HPLC/MSD "Hillary"; Analytical Sequence/Method: H010501.s/FOSA0425.m (ChemStation);

SampType	File	Sample Name	Misc Info	Compound Name	RT	Area	Amt	dil factor corrected µg/mL FOSA	
SAMPLE	HILL0093.D	FOSA-3.4.1blk	042301-FOSA-3.4.1, Mtd Blk	FOSA	6.007	553440	20	0.20	
SAMPLE	HILL0094.D	FOSA-3.4.2blk	042301-FOSA-3.4.2, Mtd Blk	FOSA	5.992	523878	18	0.18	
SAMPLE	HILL0095.D	FOSA-3.4.3blk	042301-FOSA-3.4.3, Mtd Blk	FOSA	5.986	450065	15	0.15	
SAMPLE	HILL0096.D	FOSA-3.4.4blkMS	042301-FOSA-3.4.4, Mtd Blk Spike	FOSA	5.984	3129563	111		*116%*
SAMPLE	HILL0114.D	Diluter Blk	Diluter blank with IS mix	FOSA	0	0	0	**	
SAMPLE	HILL0115.D	MeOH blk w/ IS	Methanol with IS mix	FOSA	0	0	0		
SAMPLE	HILL0077.D	TN-A 4802 MeOH		FOSA	5.988	79511	0		
SAMPLE	HILL0091.D	TN-A 4802 MeOH		FOSA	5.991	166363	0		
SAMPLE	HILL0092.D	TN-A 4802 MeOH		FOSA	5.992	63070	0		
SAMPLE	HILL0098.D	TN-A 4802 MeOH		FOSA	5.99	40614	0		
SAMPLE	HILL0105.D	TN-A 4802 MeOH		FOSA	5.992	30961	0		
SAMPLE	HILL0106.D	TN-A 4802 MeOH		FOSA	5.991	18624	0		
SAMPLE	HILL0111.D	TN-A 4802 MeOH		FOSA	5.916	46114	0		
SAMPLE	HILL0117.D	TN-A 4802 MeOH		FOSA	0	0	0		
SAMPLE	HILL0118.D	TN-A 4802 MeOH		FOSA	0	0	0		
SAMPLE	HILL0132.D	TN-A 4802 MeOH		FOSA	5.984	150108	0		
SAMPLE	HILL0133.D	TN-A 4802 MeOH		FOSA	5.986	56599	0		

**Day 3 samples were initially diluted on 04-25-01 and analyzed for FOSA in sequence H010430.s Method blank samples demonstrated a significant amount of FOSA. The Day 3 samples were again diluted 10X with methanol along with a diluter blank to check for FOSA background in the diluter, and a methanol blank spiked with IS mix to check for FOSA background/contamination in the internal standard mix. Method blank samples analyzed in seq H010501.s still had a background of FOSA, but it was determined that the FOSA was somehow introduced to the urine during the prep procedure prior to dilution since the diluter blank and IS blank came out "clean." Days 1 and 2 method blank samples demonstrate that the urine used for the study had no initial background of FOSA, and so the level of FOSA in Day 3 mtd blks was due to a small amt of FOSA introduced to the test system during sample preparation. This background of FOSA in the Day 3 mtd blks may be disregarded since it was only incurred on the day of test analyte in addition, and was a random, inadvertent error that does not affect the solubility results for the other three Day 3 samples.

ATTACHMENT C: SAMPLE CHROMATOGRAMS
CHROMATOGRAMS INCLUDED FOR BOTH PFOS AND FOSA:
-3 SOLVENT BLANK CHROMATOGRAMS
-3 METHOD BLANK CHROMATOGRAMS
-9 SAMPLE CHROMATOGRAMS (3 CHROM PER DAY)

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0029.D
Lab Smp Id: TN-A 4802 MeOH
Inj Date : 26-APR-2001 22:02
Operator : KLT Inst ID: hillary.i
Smp Info : TN-A 4802 MeOH
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010426.s
Method : D:\chem\Hillary.i\H010426.b\H0100426t.m
Meth Date : 01-May-2001 11:12 terrell Quant Type: ISTD
Cal Date : 26-APR-2001 21:40 Cal File: HILL0027.D
Als bottle: 92
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	Compound Not Detected.					
* 7 PFBS	299	4.389	4.410	(1.000)	9125	254.000	
2 PFOS	499	5.419	5.334	(1.000)	65012	~~682.022~~ 683	

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05-16-01 Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0029.D

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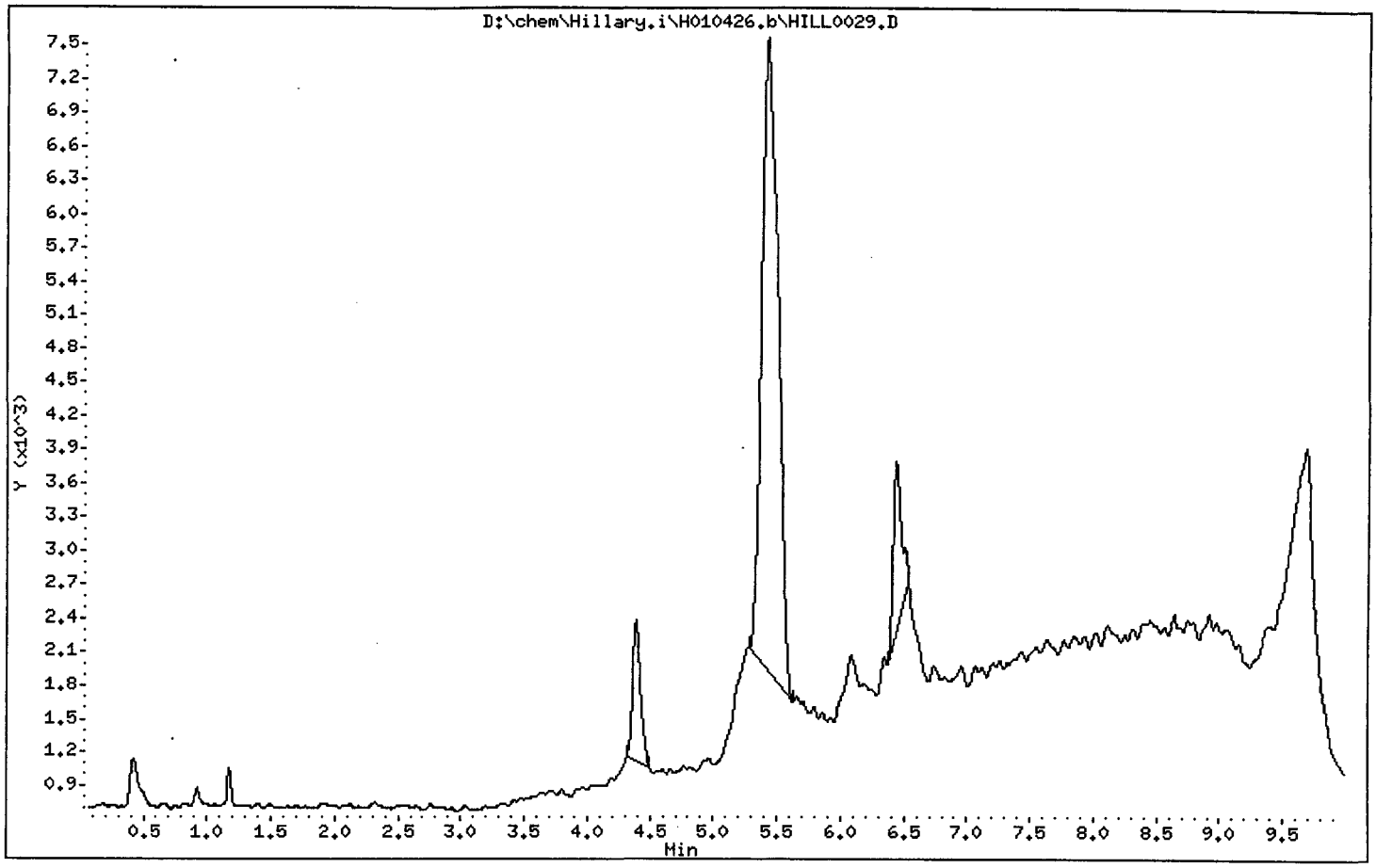
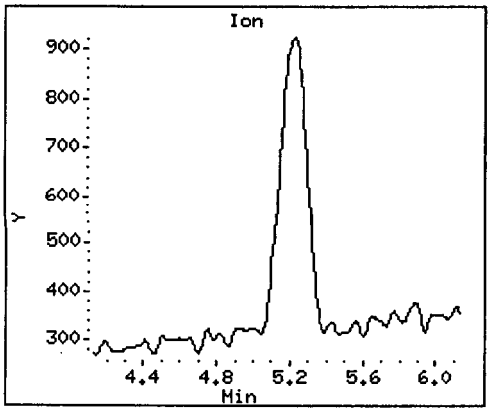
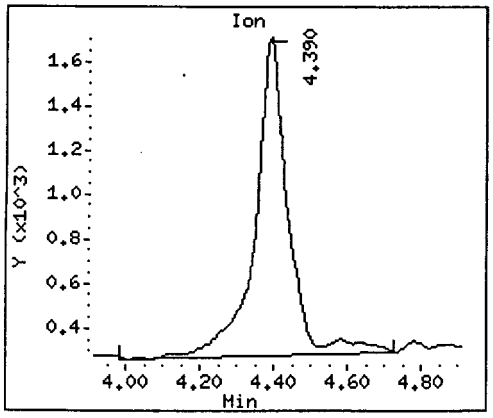


* 1 THPFOS (Undetected)



* 7 PFBS



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Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0029.D

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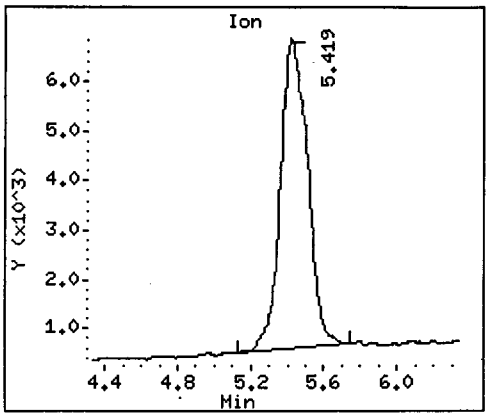
2 PFOS



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Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0036.D
Lab Smp Id: TN-A 4802 MeOH
Inj Date : 01-MAY-2001 22:59
Operator : KLT Inst ID: hillary.i
Smp Info : TN-A 4802 MeOH
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501t.m
Meth Date : 03-May-2001 08:17 terrell Quant Type: ISTD
Cal Date : 01-MAY-2001 21:19 Cal File: HILL0027.D
Als bottle: 93
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	Compound Not Detected.					
* 2 PFBS	299	Compound Not Detected.					
3 PFOS	499	Compound Not Detected.					

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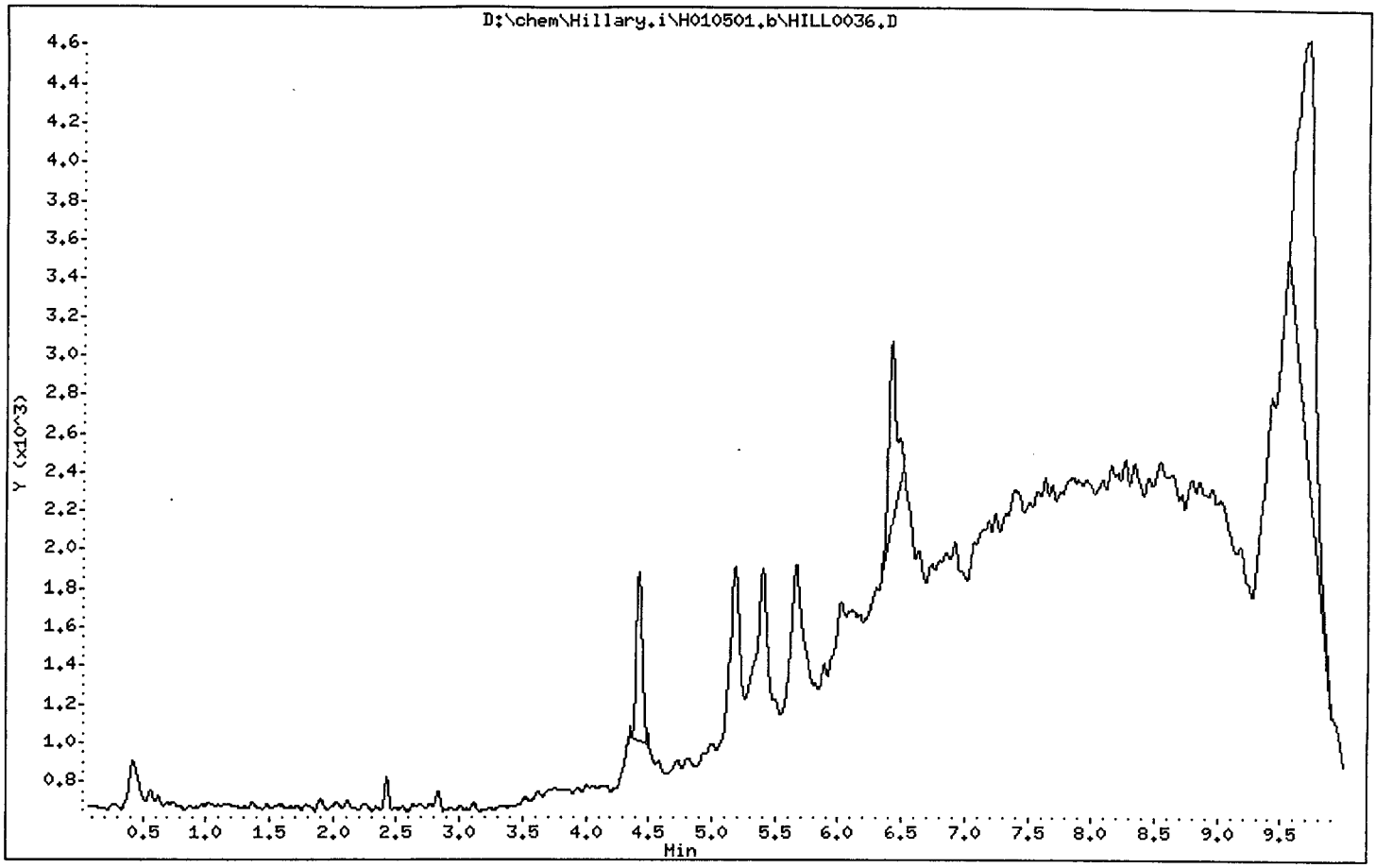
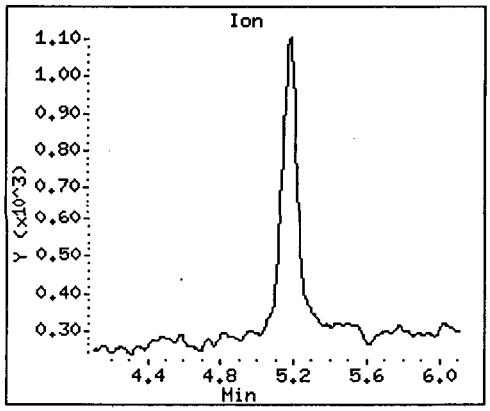
KLT 05/16/01
Initial Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0036.D

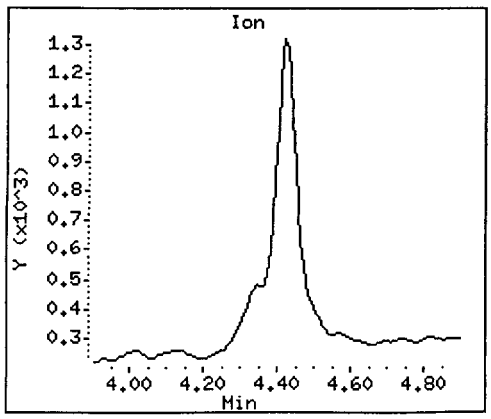
3M Environmental Laboratory
Project # E01-0768



× 1 THPFOS (Undetected)



× 2 PFBS (Undetected)



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05-16-01
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Data File: D:\chem\Hillary.i\H010501.b\HILL0036.D

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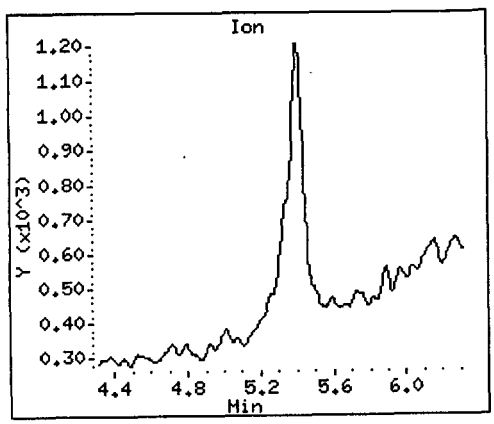
3 PFOS (Undetected)



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Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010430.b\HILL0057.D
Lab Smp Id: TN-A 4802 MeOH
Inj Date : 30-APR-2001 18:57
Operator : KLT Inst ID: hillary.i
Smp Info : TN-A 4802 MeOH
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010430.s
Method : D:\chem\Hillary.i\H010430.b\H0100430t.m
Meth Date : 01-May-2001 12:59 terrell Quant Type: ISTD
Cal Date : 30-APR-2001 13:21 Cal File: HILL0027.D
Als bottle: 93
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	CONCENTRATIONS FINAL (ng/mL)
* 1 THPFOS	427	Compound Not Detected.					
* 7 PFBS	299	Compound Not Detected.					
2 PFOS	499	Compound Not Detected.					

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05-16-01 Date

Data File: D:\chem\Hillary.i\H010430.b\HILL0057.D

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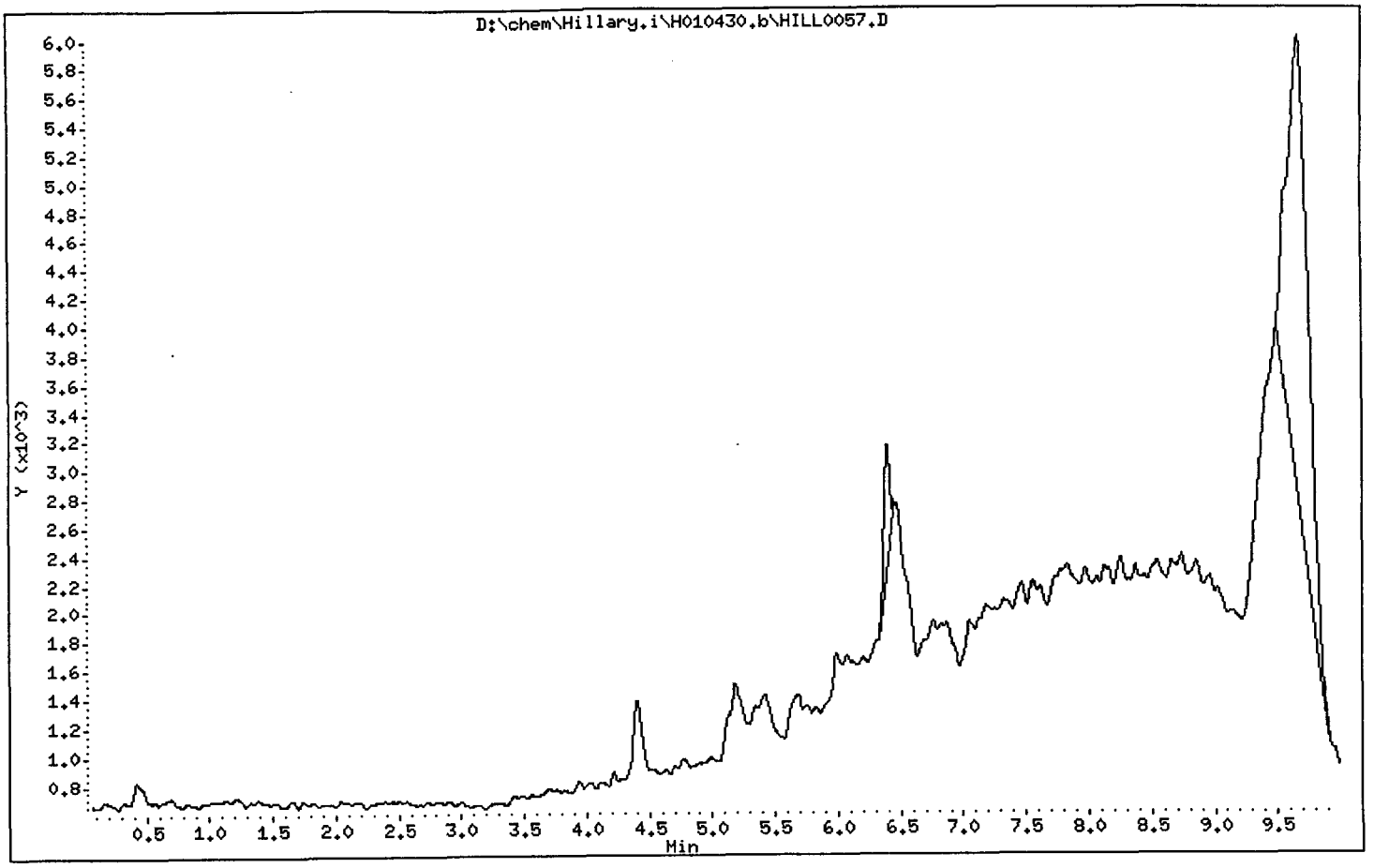
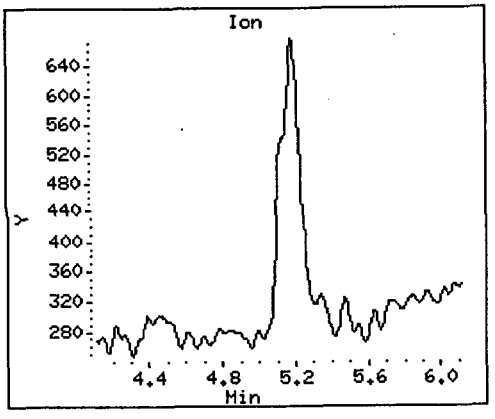
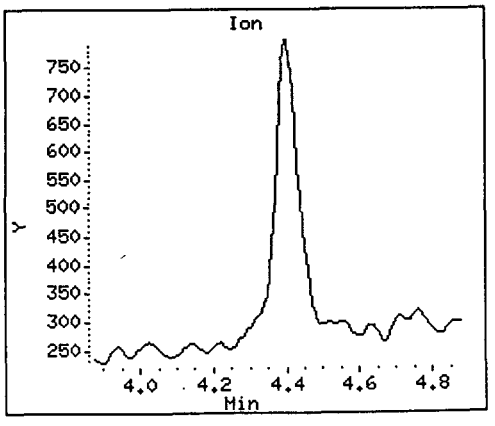


× 1 THPFOS (Undetected)



× 7 PFBS (Undetected)



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Data File: D:\chem\Hillary.i\H010430.b\HILL0057.D

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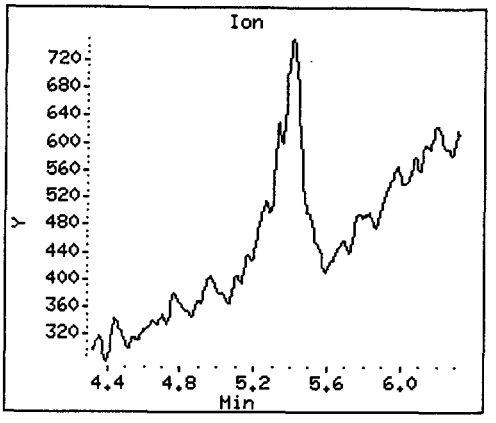
2 PFOS (Undetected)



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3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0032.D
Lab Smp Id: PFOS-1.4.1blk
Inj Date : 26-APR-2001 22:36
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-1
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010426.s
Method : D:\chem\Hillary.i\H010426.b\H0100426t.m
Meth Date : 01-May-2001 11:12 terrell Quant Type: ISTD
Cal Date : 26-APR-2001 21:40 Cal File: HILL0027.D
Als bottle: 22
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.111	5.131	(1.000)	1357667	254.000	
* 7 PFBS	299	4.369	4.410	(1.000)	1900574	254.000	
2 PFOS	499	Compound Not Detected.					

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Data File: D:\chem\Hillary.i\H010426.b\HILL0032.D

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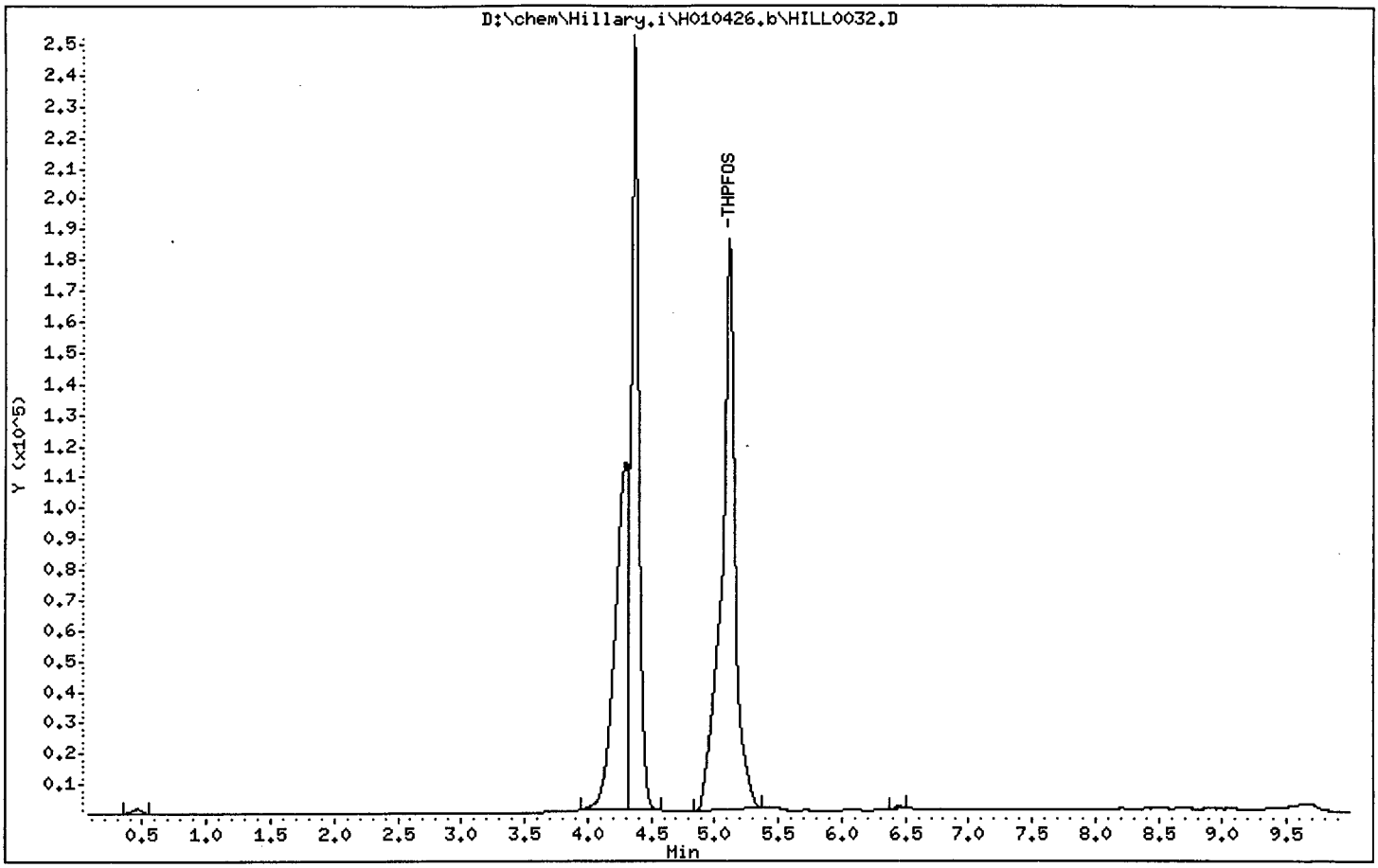
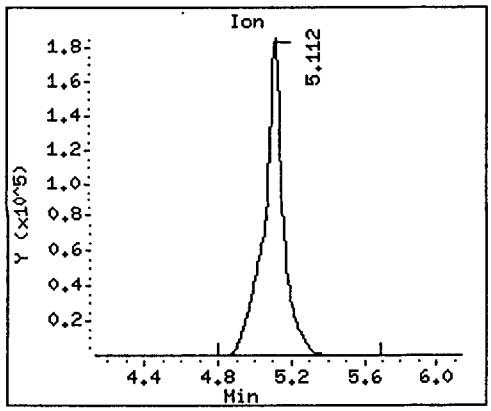
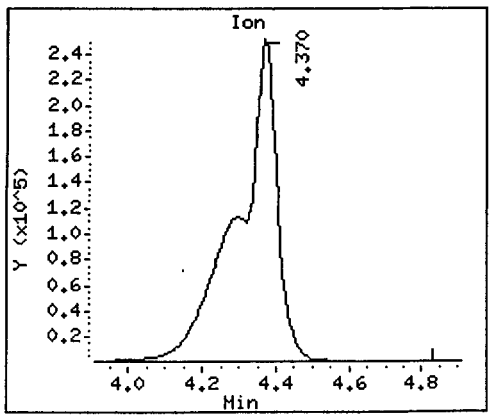
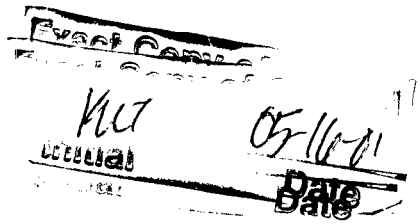


* 1 THPFOS



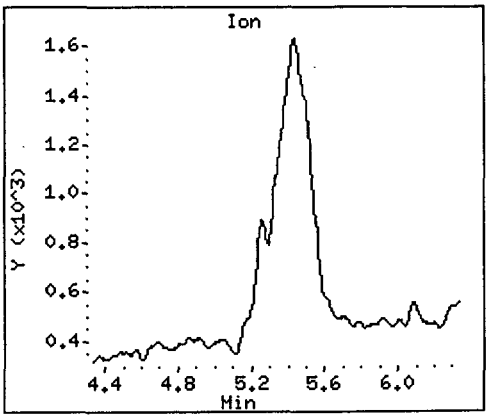
* 7 PFBS





Data File: D:\chem\Hillary.i\H010426.b\HILL0032.D

2 PFOS (Undetected)



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Initial Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0032.D
Lab Smp Id: PFOS-2.4.1blk
Inj Date : 01-MAY-2001 22:15
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-2
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501t.m
Meth Date : 03-May-2001 08:17 terrell Quant Type: ISTD
Cal Date : 01-MAY-2001 21:19 Cal File: HILL0027.D
Als bottle: 22
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	CONCENTRATIONS FINAL (ng/mL)
* 1 THPFOS	427	5.114	5.109	(1.000)	1317272	254.000	
* 2 PFBS	299	4.407	4.402	(1.000)	1718184	254.000	
3 PFOS	499	Compound Not Detected.					

Initial 05/16/01 Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0032.D

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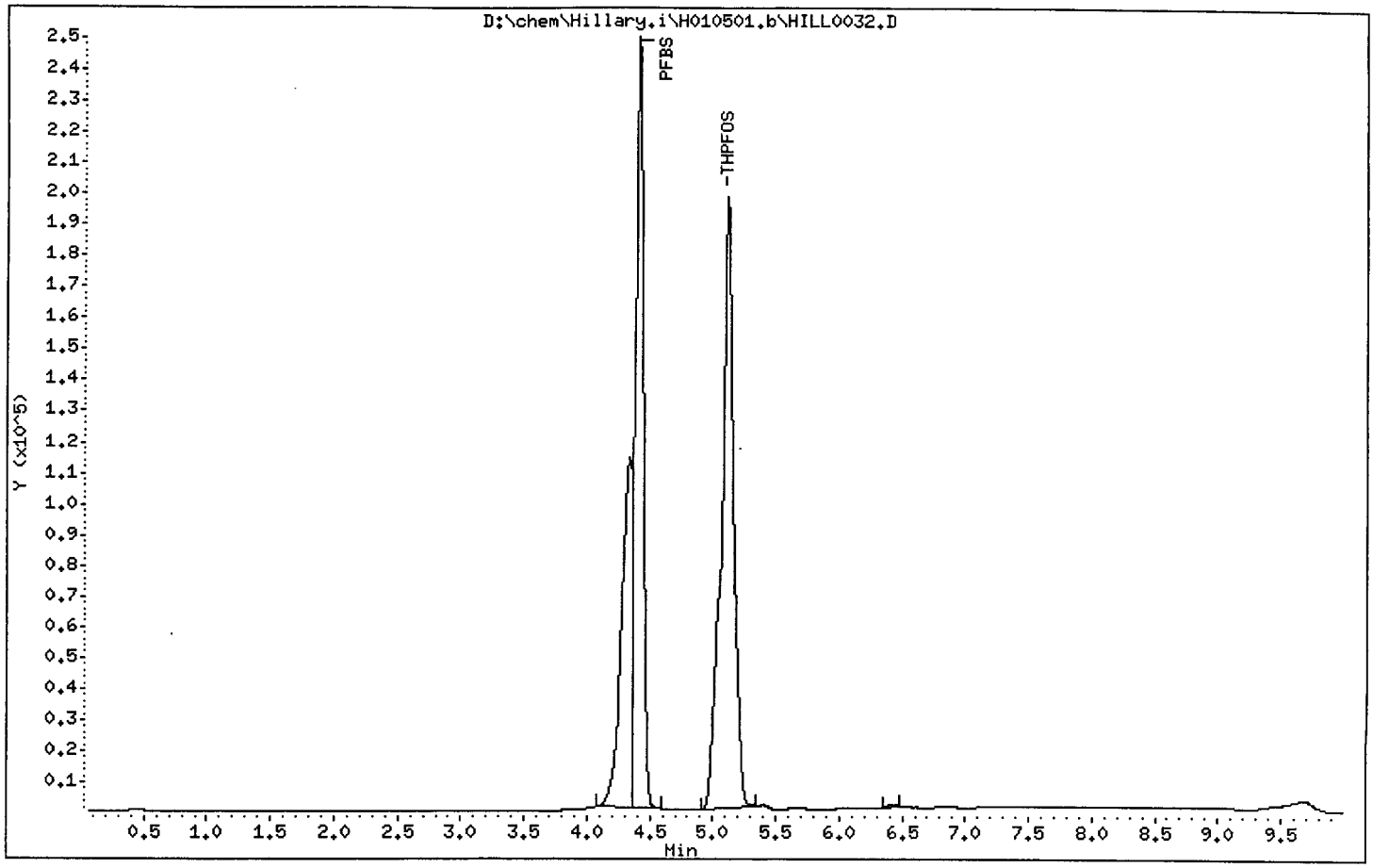
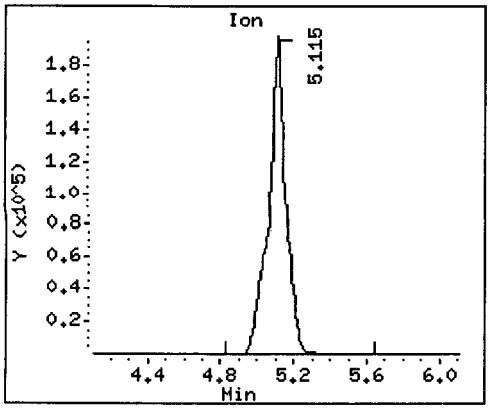
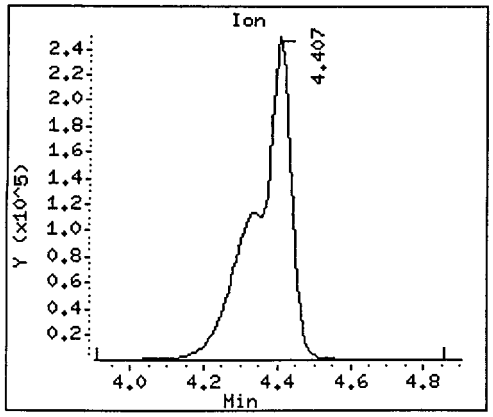
Project # E01-0768 Page 2



× 1 THPFOS



× 2 PFBS



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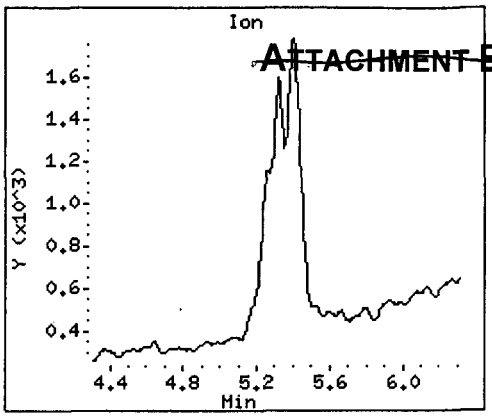
Initial 05-16-01 Date

3 PFOS (Undetected)



~~ATTACHMENT B: DATA SUMMARY TABLES~~ (RE) KLT 5-29-01

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KLT 05-16-01
Initial Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010430.b\HILL0059.D
Lab Smp Id: PFOS-3.4.1blk
Inj Date : 30-APR-2001 19:19
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-3
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010430.s
Method : D:\chem\Hillary.i\H010430.b\H0100430t.m
Meth Date : 01-May-2001 12:59 terrell Quant Type: ISTD
Cal Date : 30-APR-2001 13:21 Cal File: HILL0027.D
Als bottle: 22
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.098	5.108	(1.000)	1419875	254.000	
* 7 PFBS	299	4.363	4.380	(1.000)	1870275	254.000	
2 PFOS	499	Compound Not Detected.					

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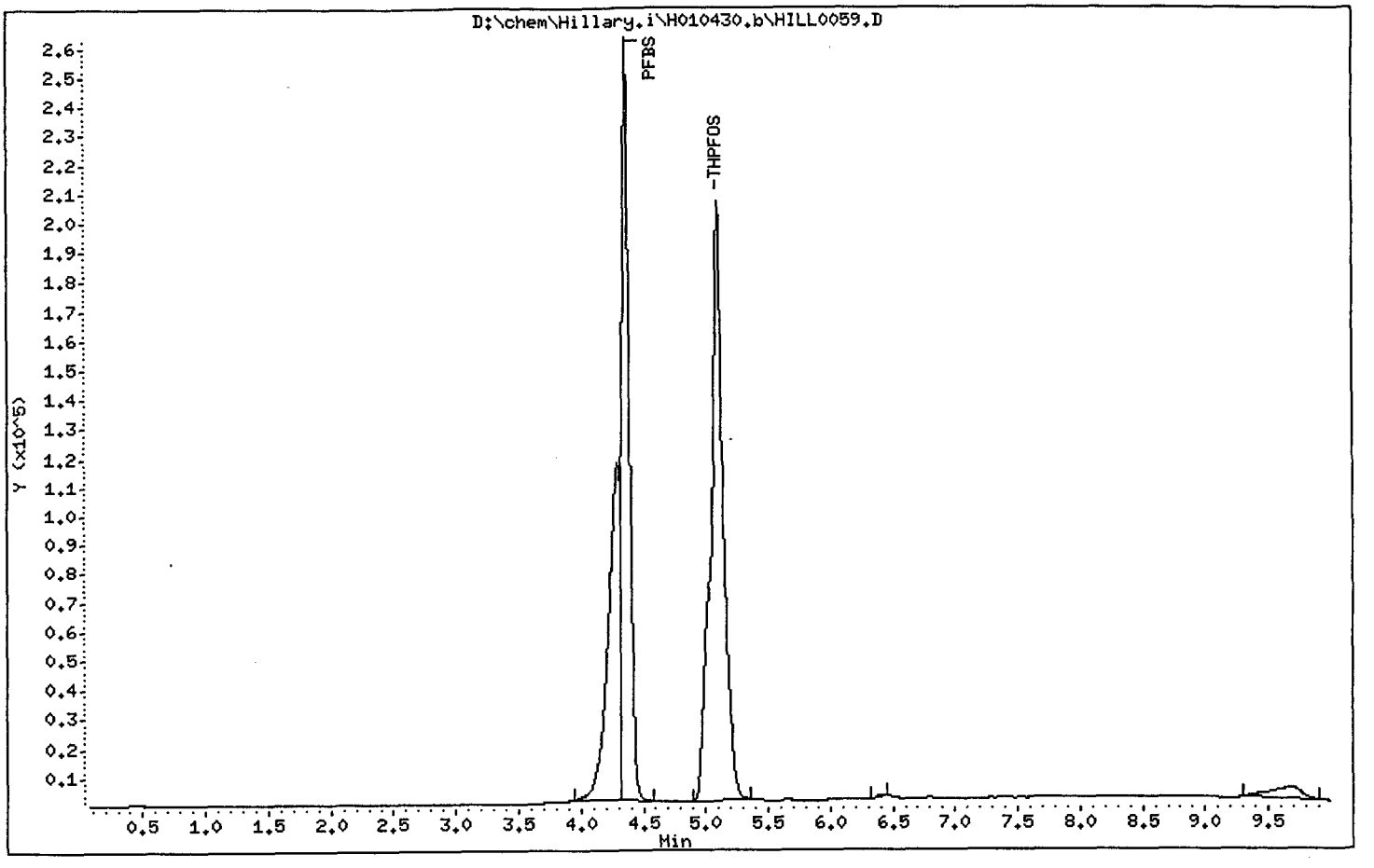
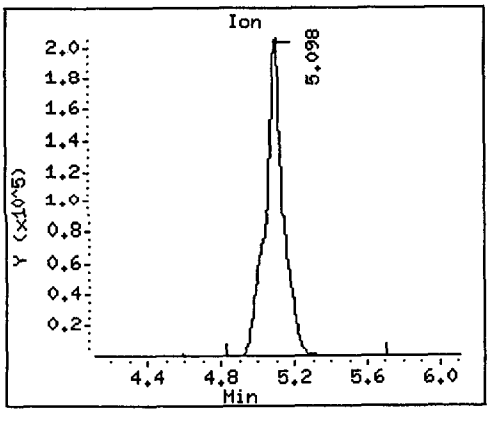
KLT Initial
5/16/01 Date

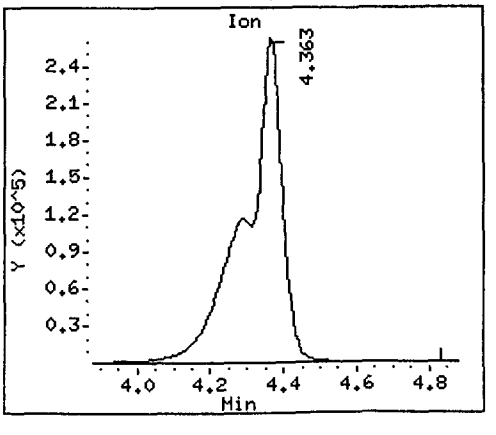
Data File: D:\chem\Hillary.i\H010430.b\HILL0059.D



* 1 THPFOS



* 7 PFBS



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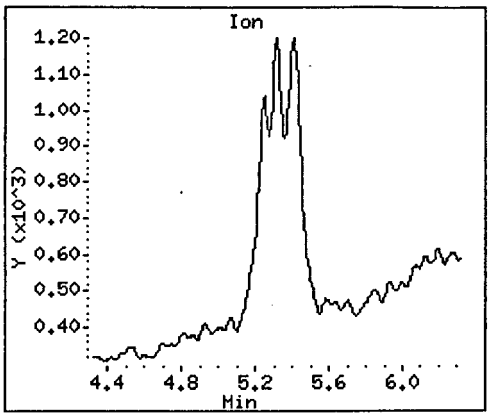
Date

Data File: D:\chem\Hillary.i\H010430.b\HILL0059.D

3M Environmental Laboratory
Project # E01-0768

2 PFOS (Undetected)



KU Initial 05-16-01 Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0037.D
Lab Smp Id: PFOS-1.1.1
Inj Date : 26-APR-2001 23:32
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-1
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010426.s
Method : D:\chem\Hillary.i\H010426.b\H0100426t.m
Meth Date : 01-May-2001 11:12 terrell Quant Type: ISTD
Cal Date : 26-APR-2001 21:40 Cal File: HILL0027.D
Als bottle: 26
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.123	5.131	(1.000)	1239933	254.000	
* 7 PFBS	299	4.388	4.410	(1.000)	2179012	254.000	
2 PFOS	499	5.270	5.334	(1.029)	7570273	292.634	293

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KLT
Initial

05-10-07
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0037.D

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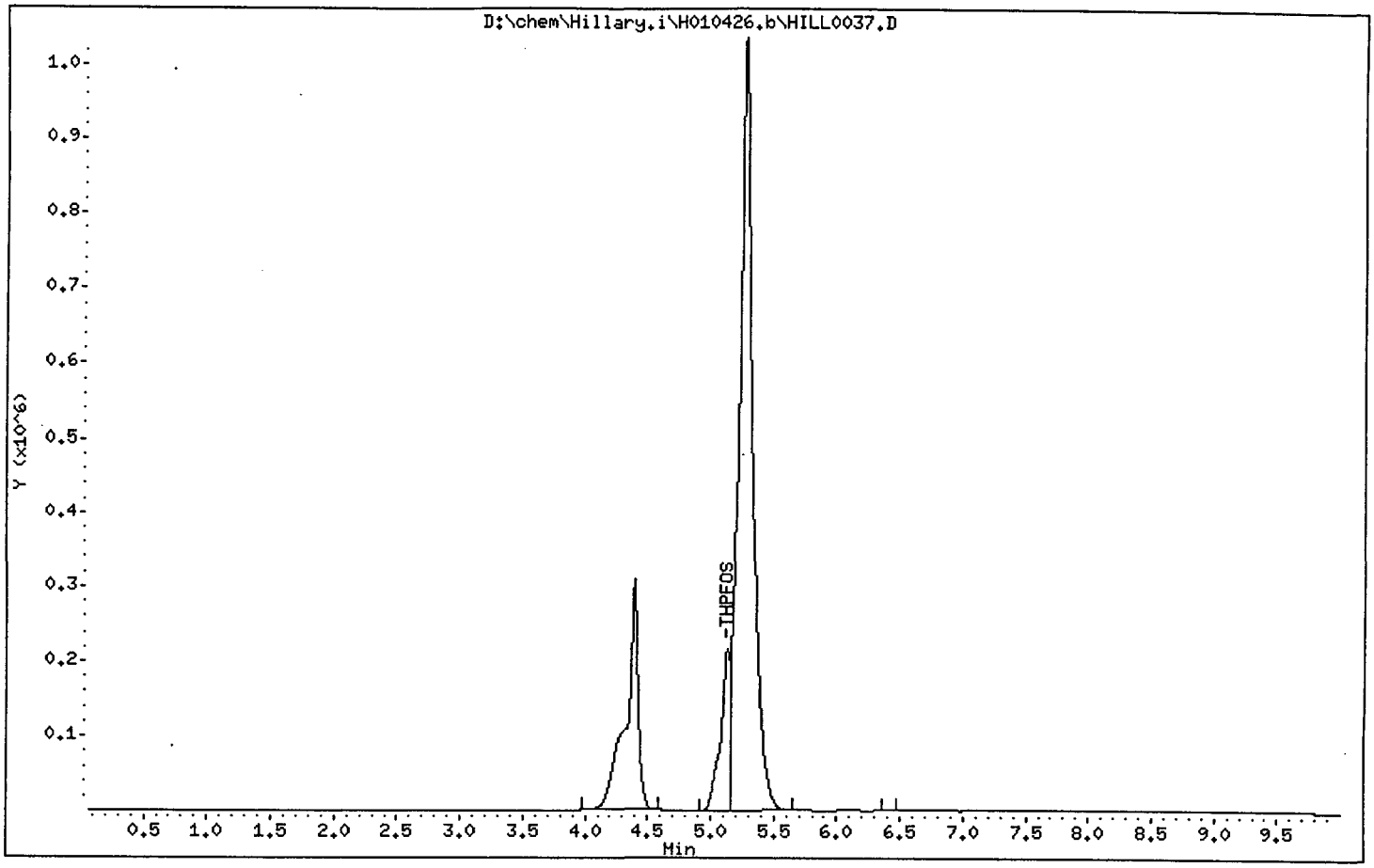
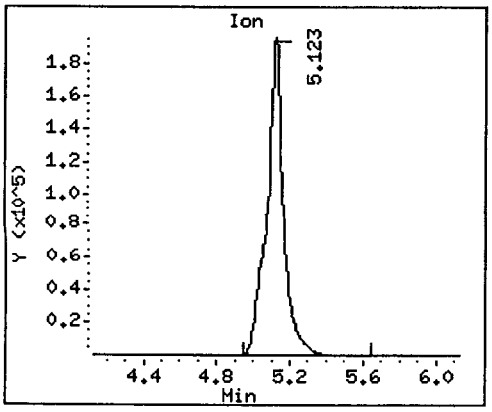
Project # E01-0768 Page 2



* 1 THPFOS



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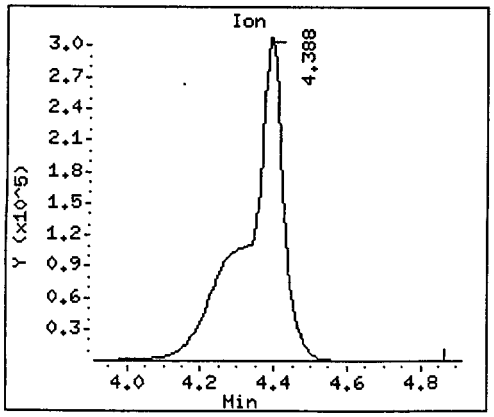

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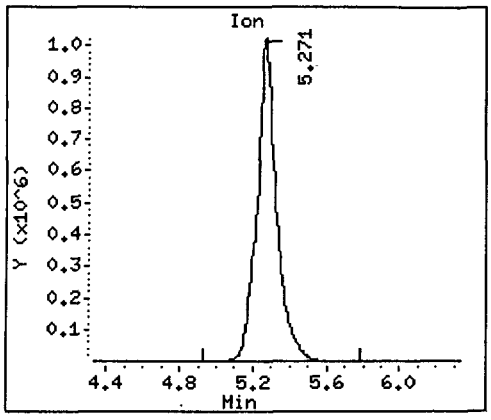
05-IV-01

Data File: D:\chem\Hillary.i\H010426.b\HILL0037.D

2 PFOS



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KH
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05-16-01
Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0041.D
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Inj Date : 27-APR-2001 00:16
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-1
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010426.s
Method : D:\chem\Hillary.i\H010426.b\H0100426t.m
Meth Date : 01-May-2001 11:12 terrell Quant Type: ISTD
Cal Date : 26-APR-2001 21:40 Cal File: HILL0027.D
Als bottle: 30
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.110	5.131	(1.000)	1262274	254.000	
* 7 PFBS	299	4.396	4.410	(1.000)	2148762	254.000	
2 PFOS	499	5.251	5.334	(1.027)	7600633	298.657	299

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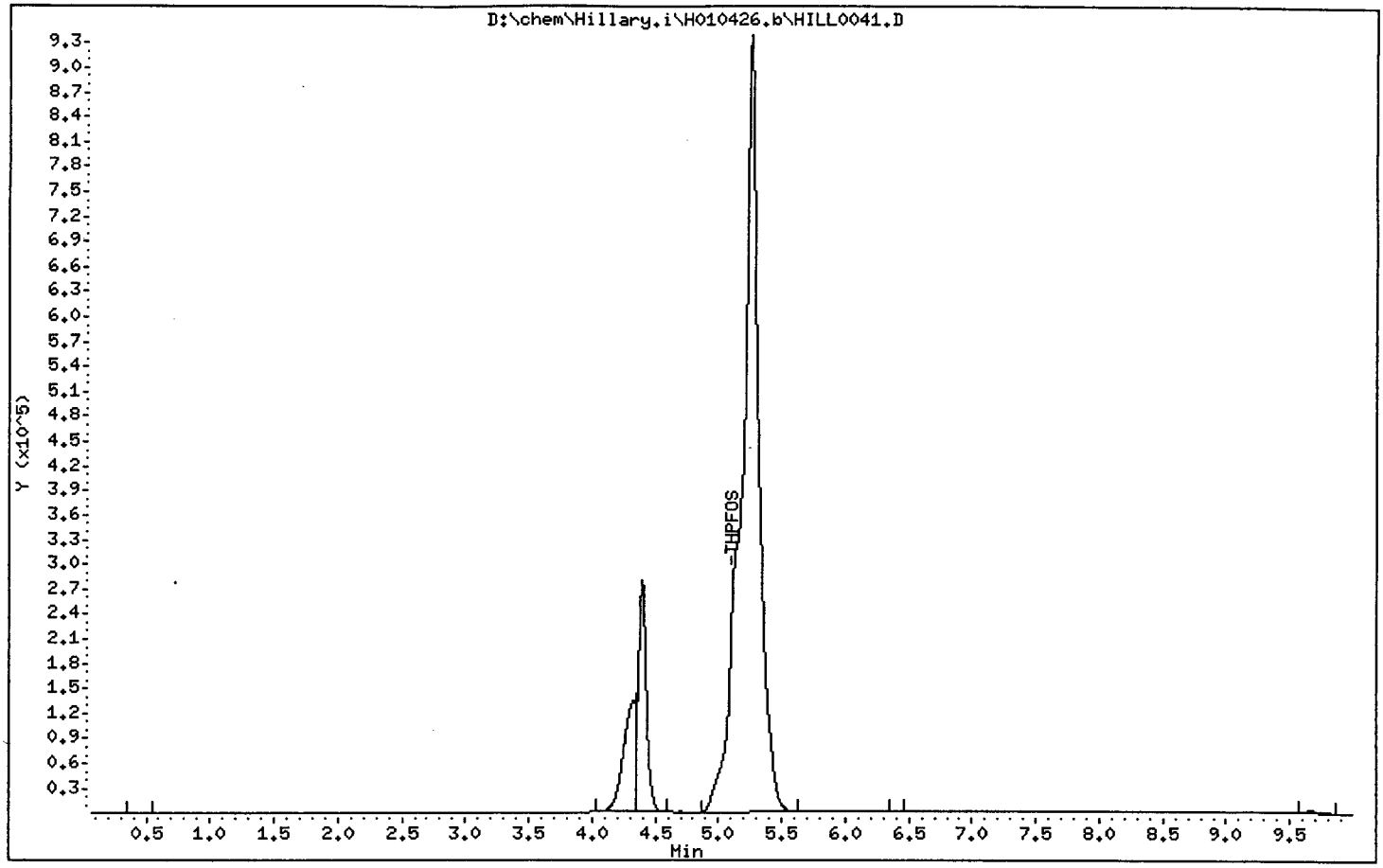
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0041.D

3M Environmental Laboratory
Project # E01-0768 Page 2



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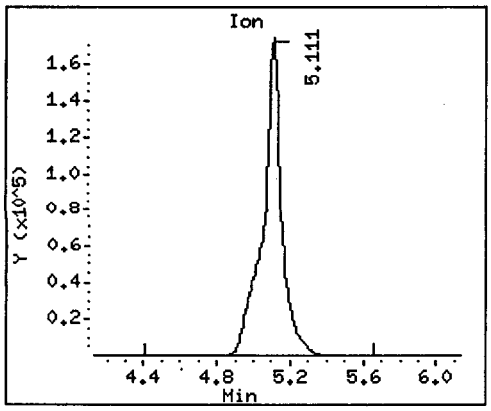
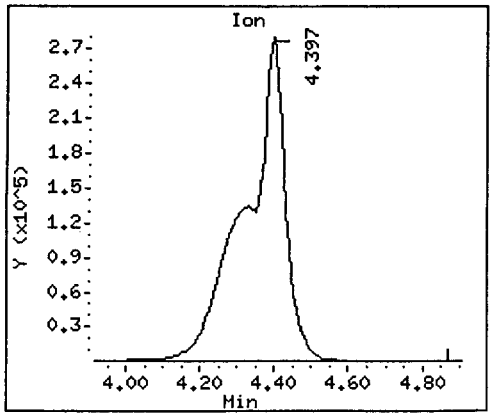


* 7 PFBS



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KLT 05-16-01
Initial Date

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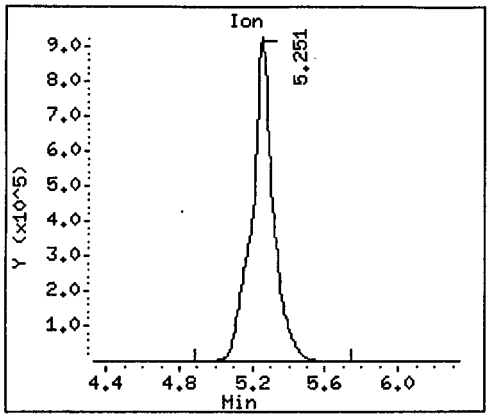
2 PFOS



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KLT
Initial

05/11-01
Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0049.D
Lab Smp Id: PFOS-1.3.1
Inj Date : 27-APR-2001 01:47
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-1
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010426.s
Method : D:\chem\Hillary.i\H010426.b\H0100426t.m
Meth Date : 01-May-2001 11:12 terrell Quant Type: ISTD
Cal Date : 26-APR-2001 21:40 Cal File: HILL0027.D
Als bottle: 34
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.129	5.131	(1.000)	1269547	254.000	
* 7 PFBS	299	4.394	4.410	(1.000)	2195975	254.000	
2 PFOS	499	5.269	5.334	(1.027)	8267658	320.624	321

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Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0049.D

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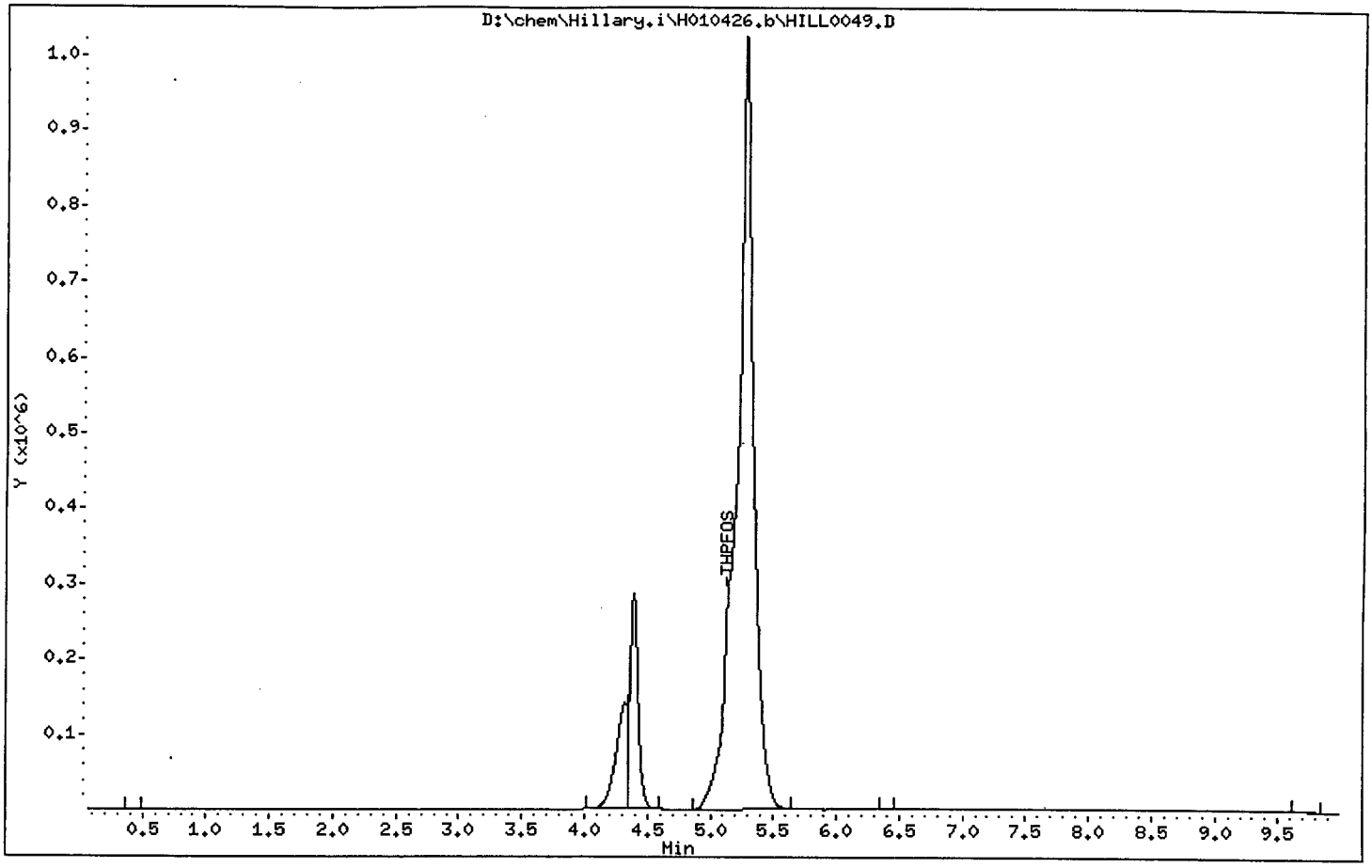

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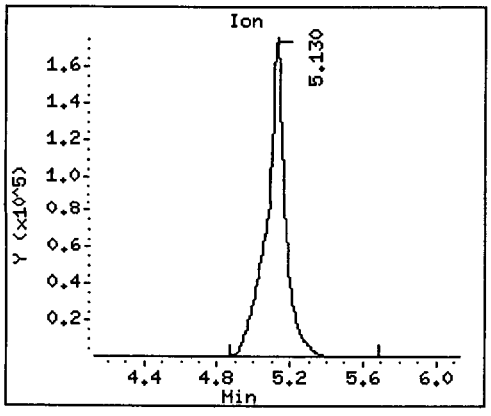

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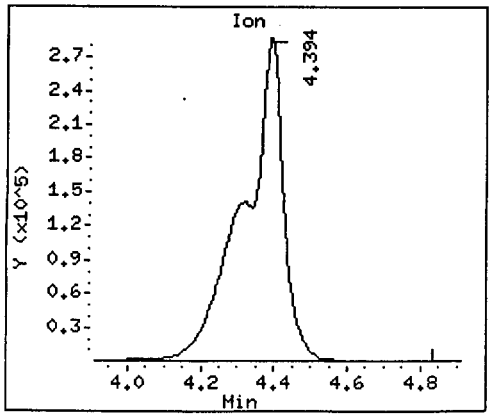

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KT 05/16-01
Initial Date

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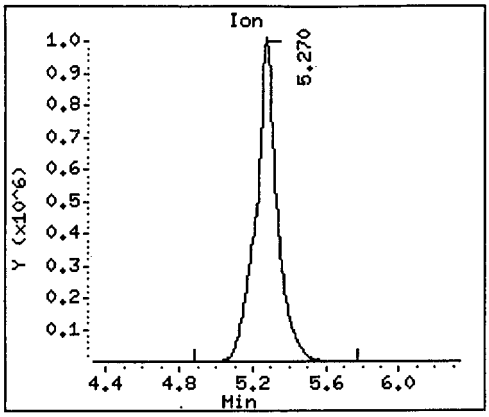
2 PFOS



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KLT
Initial

05-16-01
Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0037.D
Lab Smp Id: PFOS-2.1.1
Inj Date : 01-MAY-2001 23:11
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-2
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501t.m
Meth Date : 03-May-2001 08:17 terrell Quant Type: ISTD
Cal Date : 01-MAY-2001 21:19 Cal File: HILL0027.D
Als bottle: 26
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.115	5.109	(1.000)	1215865	254.000	
* 2 PFBS	299	4.407	4.402	(1.000)	1912250	254.000	
3 PFOS	499	5.248	5.312	(1.191)	7420988	311.873	312

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05/16/01 Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0037.D

3M Environmental Laboratory
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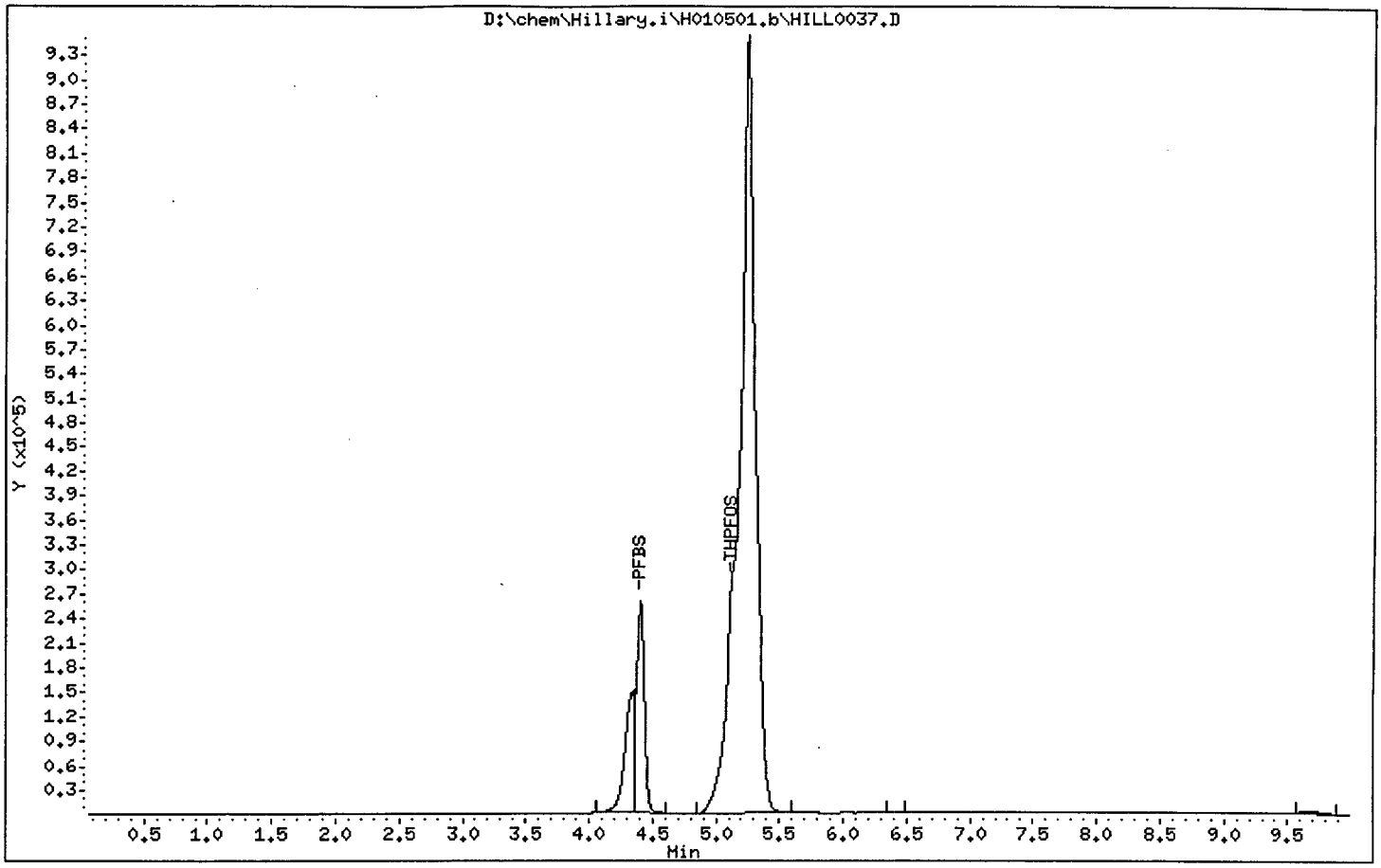

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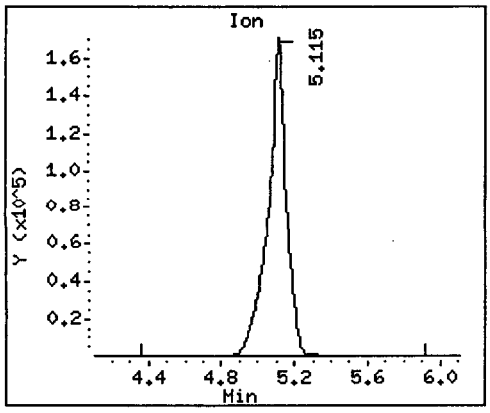

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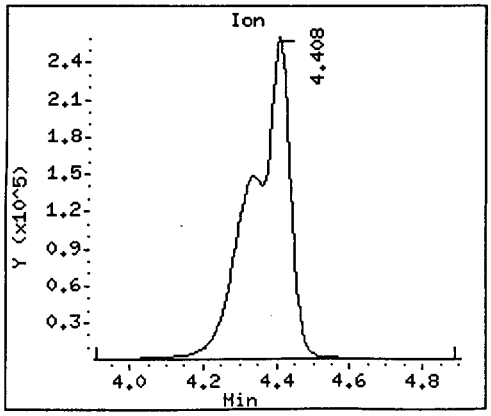

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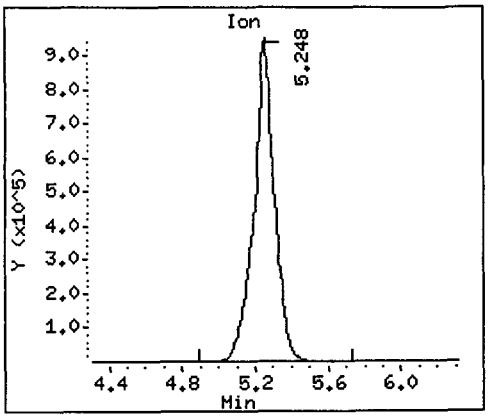
Data File: D:\chem\Hillary.i\H010501.b\HILL0037.D

3M Environmental Laboratory
Project # E01-0768

3 PFOS



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05-16-01
Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0045.D
Lab Smp Id: PFOS-2.2.1
Inj Date : 02-MAY-2001 00:40
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-2
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501t.m
Meth Date : 03-May-2001 08:17 terrell Quant Type: ISTD
Cal Date : 01-MAY-2001 21:19 Cal File: HILL0027.D
Als bottle: 30
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.122	5.109	(1.000)	1256724	254.000	
* 2 PFBS	299	4.408	4.402	(1.000)	1900616	254.000	
3 PFOS	499	5.262	5.312	(1.194)	7021826	294.754	295

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Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0045.D

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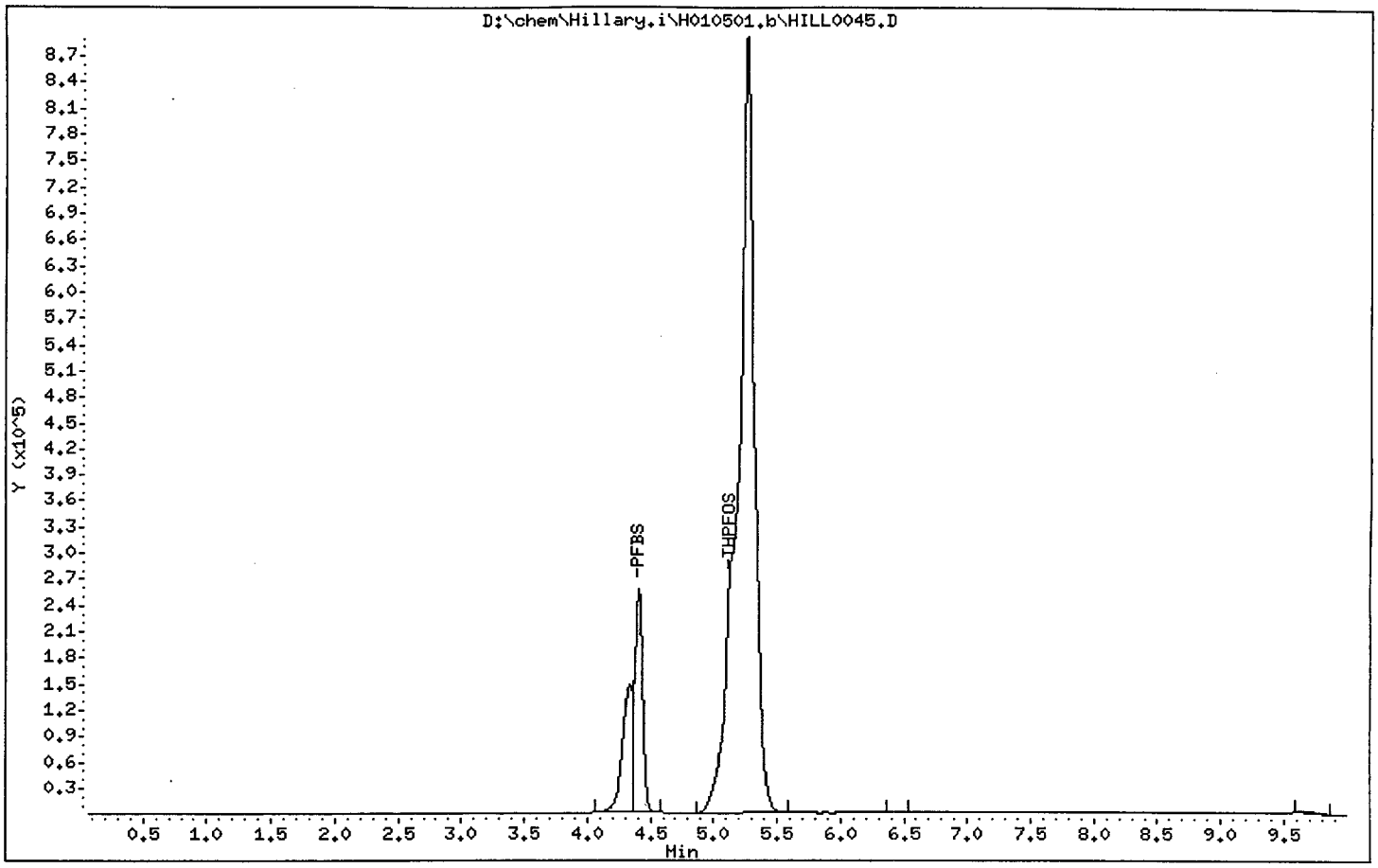
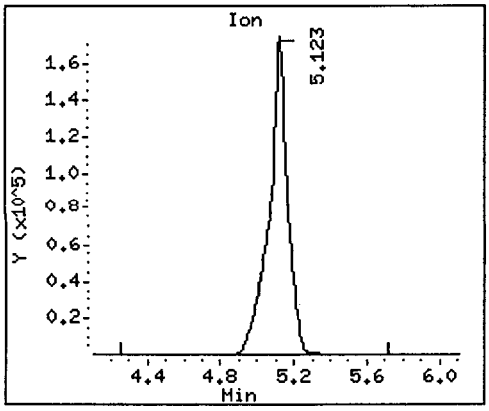


× 1 THPFOS



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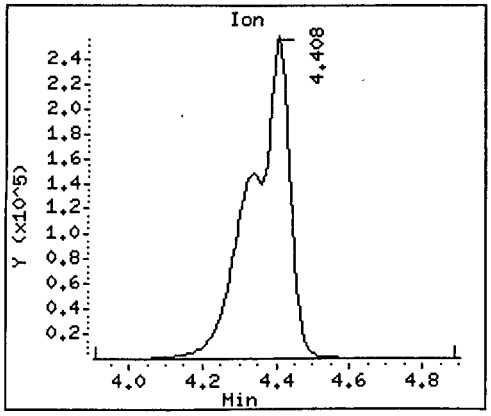

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Date

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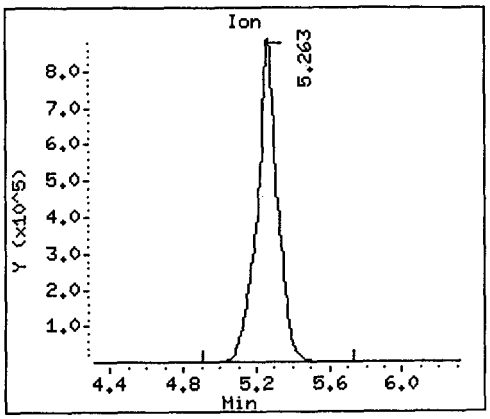
3M Environmental Laboratory
Project # E01-0768

3 PFOS



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05-16-01
Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0050.D
Lab Smp Id: PFOS-2.3.1
Inj Date : 02-MAY-2001 01:36
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-2
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501t.m
Meth Date : 03-May-2001 08:17 terrell Quant Type: ISTD
Cal Date : 01-MAY-2001 21:19 Cal File: HILL0027.D
Als bottle: 34
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.116	5.109	(1.000)	1211070	254.000	
* 2 PFBS	299	4.409	4.402	(1.000)	1958520	254.000	
3 PFOS	499	5.257	5.312	(1.192)	8177762	339.416	339

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KLT Initial
05/16-01 Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0050.D

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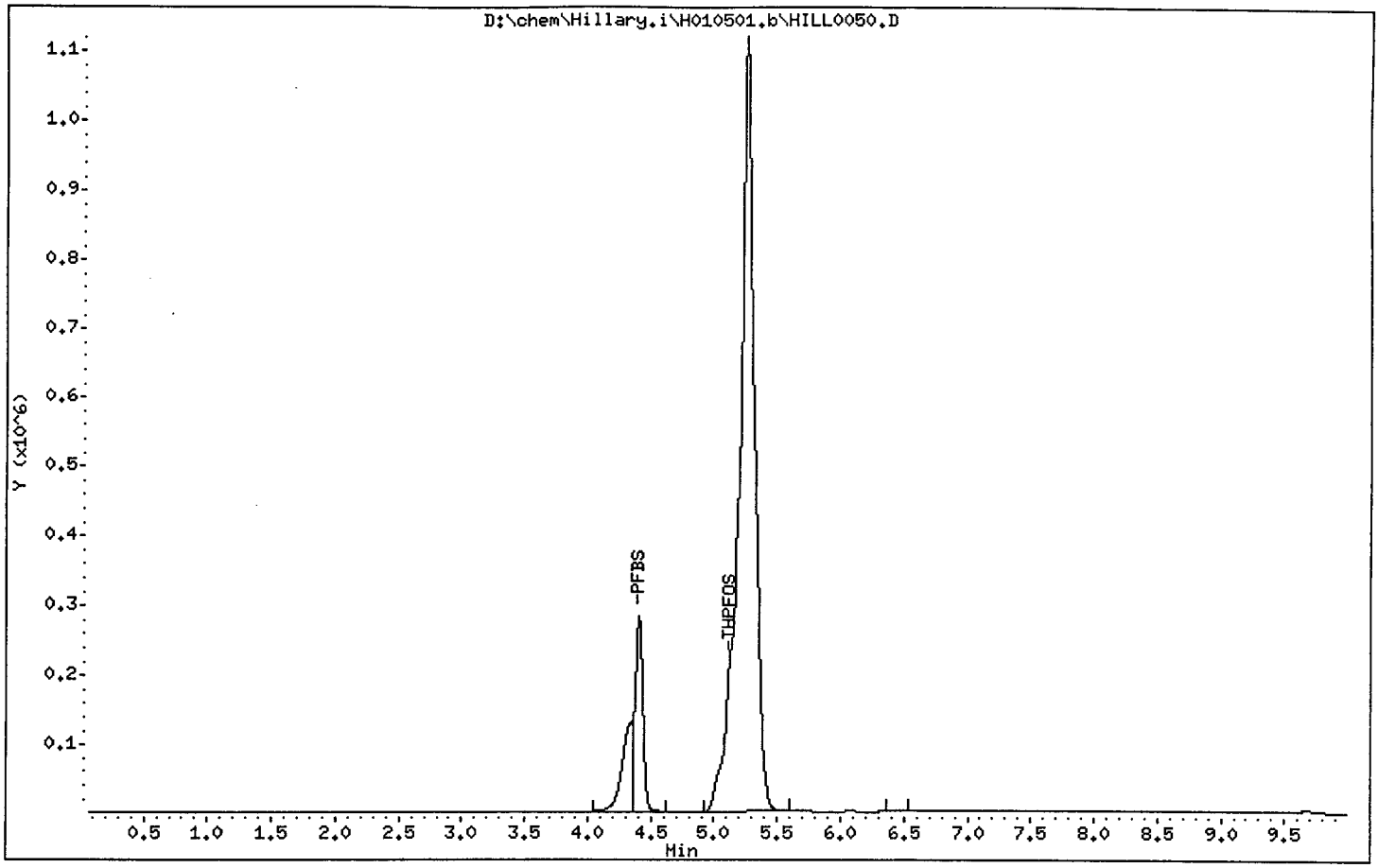

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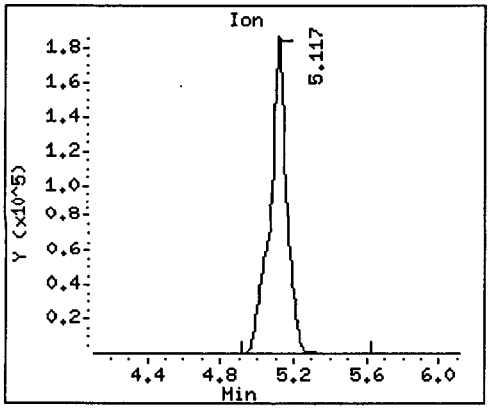

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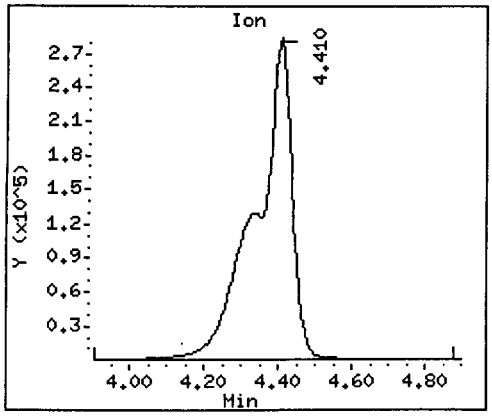

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KLT
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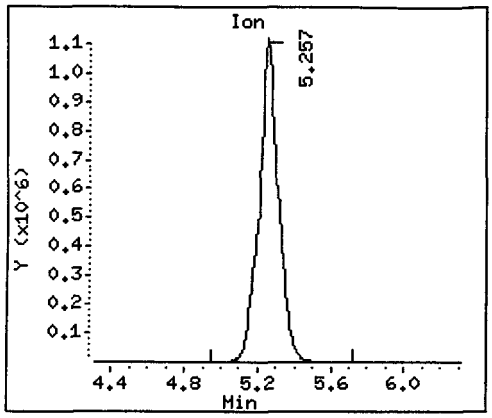
05-16-01
Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0050.D

3M Environmental Laboratory
Project # E01-0768

3 PFOS



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Initial

05-16-01
Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010430.b\HILL0064.D
Lab Smp Id: PFOS-3.1.1
Inj Date : 30-APR-2001 20:15
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-3
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010430.s
Method : D:\chem\Hillary.i\H010430.b\H0100430t.m
Meth Date : 01-May-2001 12:59 terrell Quant Type: ISTD
Cal Date : 30-APR-2001 13:21 Cal File: HILL0027.D
Als bottle: 26
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.097	5.108	(1.000)	1310521	254.000	
* 7 PFBS	299	4.375	4.380	(1.000)	2034585	254.000	
2 PFOS	499	5.237	5.318	(1.197)	6815087	267.605	268

Exact Copy of Original
KLT Initial
05-16-01 Date

Data File: D:\chem\Hillary.i\H010430.b\HILL0064.D

3M Environmental Laboratory
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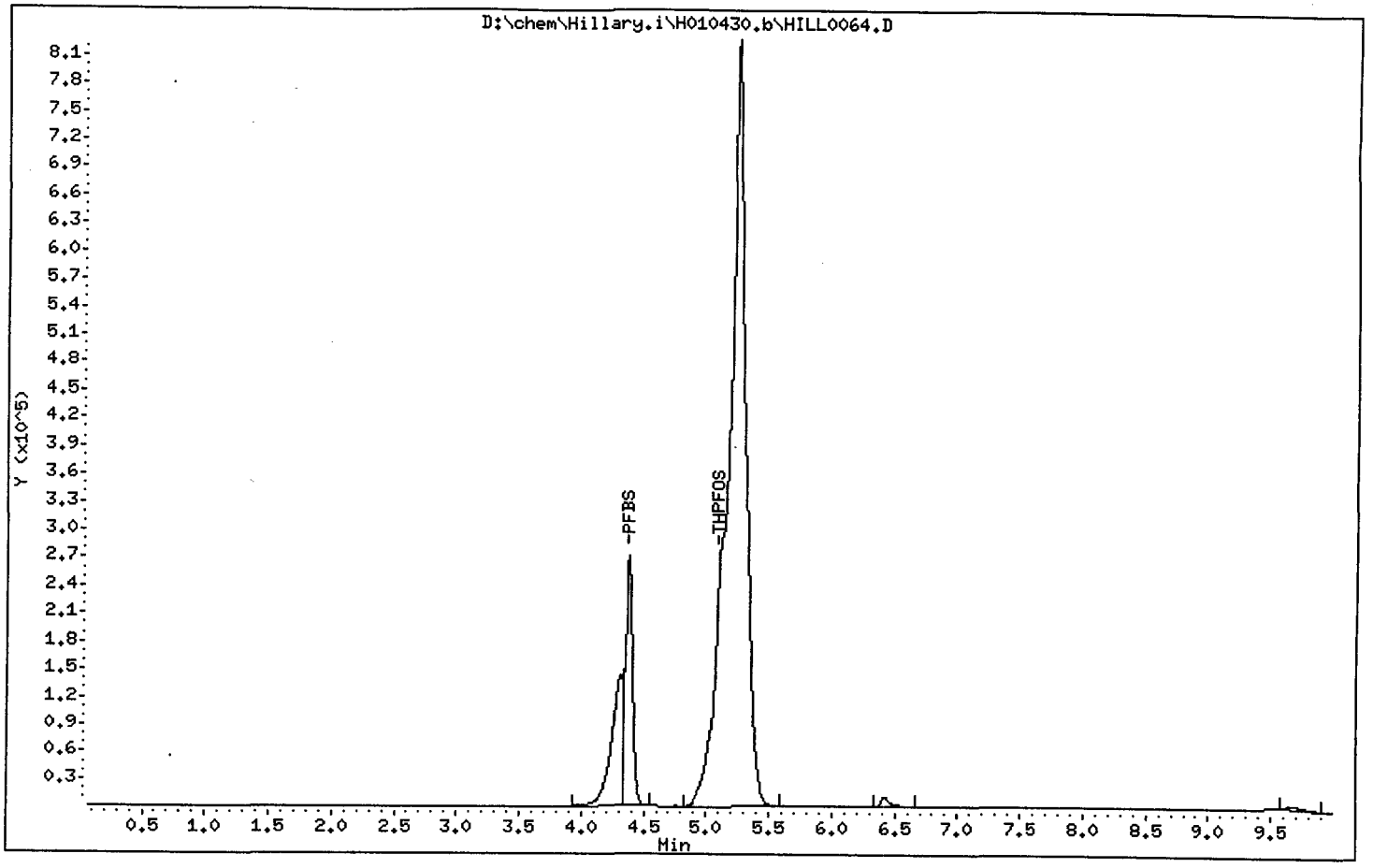
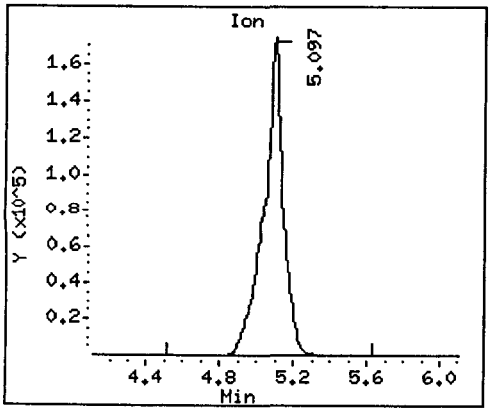


× 1 THPFOS



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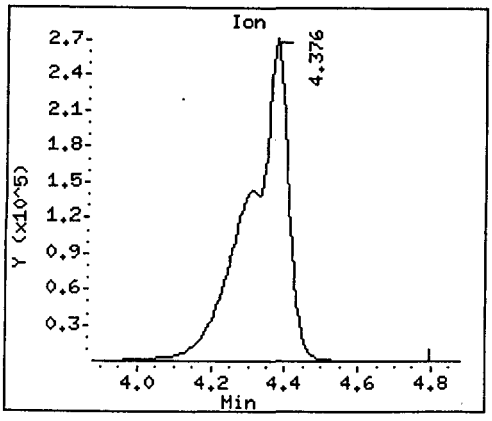

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Date

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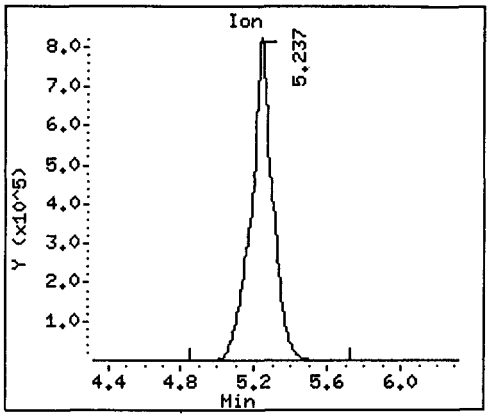
3M Environmental Laboratory
Project # E01-0768 Page 3

2 PFOS



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Initial

05-16-01
Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010430.b\HILL0072.D
Lab Smp Id: PFOS-3.2.1
Inj Date : 30-APR-2001 21:45
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-3
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010430.s
Method : D:\chem\Hillary.i\H010430.b\H0100430t.m
Meth Date : 01-May-2001 12:59 terrell Quant Type: ISTD
Cal Date : 30-APR-2001 13:21 Cal File: HILL0027.D
Als bottle: 30
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.104	5.108	(1.000)	1305210	254.000	
* 7 PFBS	299	4.383	4.380	(1.000)	2128725	254.000	
2 PFOS	499	5.245	5.318	(1.197)	8117378	309.586	310

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KLT
Initial
05-16-01
Date

Data File: D:\chem\Hillary.i\H010430.b\HILL0072.D

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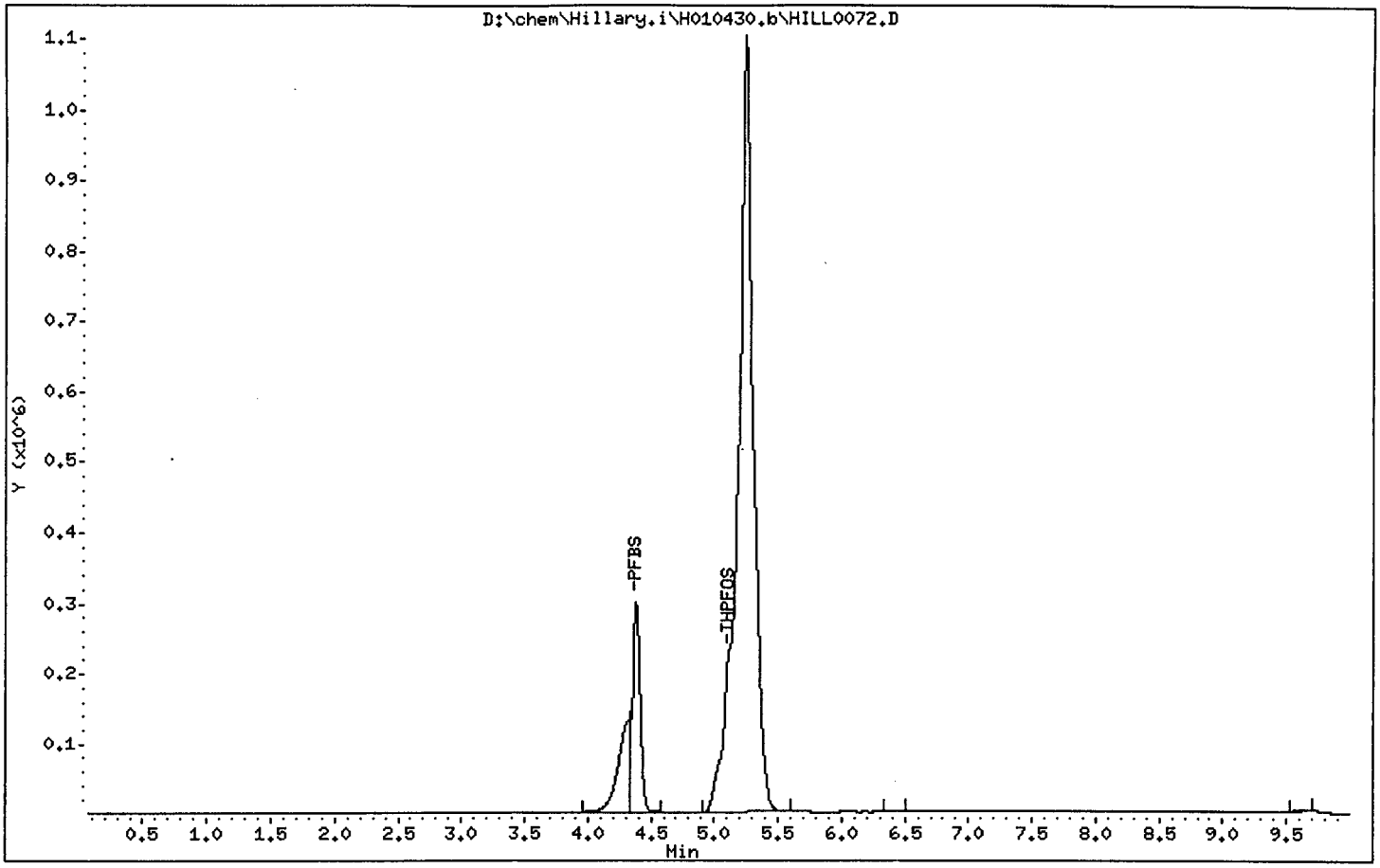
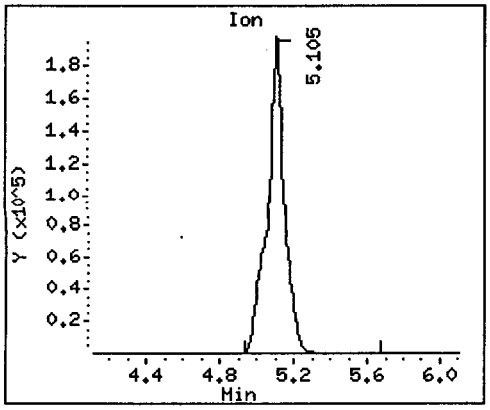


× 1 THPFOS



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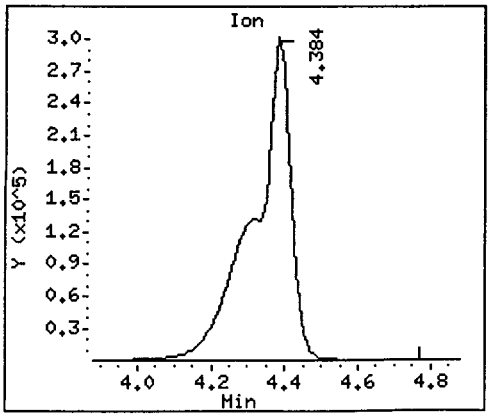

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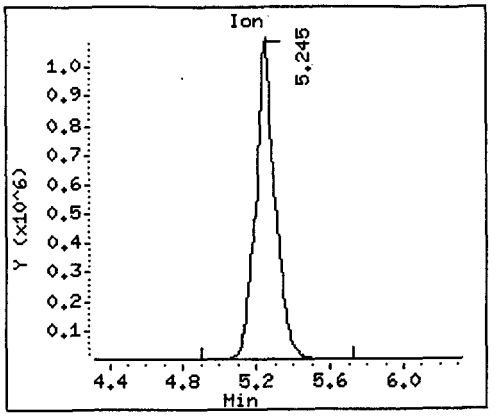
KCJ 05-16-01
Initial Date

Data File: D:\chem\Hillary.i\H010430.b\HILL0072.D

3M Environmental Laboratory
Project # E01-0768

2 PFOS



Exact Copy of Original

KLL
Initial

05-16-01
Date

3M Environmental Laboratory

PFOS, SD-018, Solubility in Urine

Data file : D:\chem\Hillary.i\H010430.b\HILL0077.D
Lab Smp Id: PFOS-3.3.1
Inj Date : 30-APR-2001 22:41
Operator : KLT Inst ID: hillary.i
Smp Info : 1,000X dil 042301-PFOS-3
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary", H010430.s
Method : D:\chem\Hillary.i\H010430.b\H0100430t.m
Meth Date : 01-May-2001 12:59 terrell Quant Type: ISTD
Cal Date : 30-APR-2001 13:21 Cal File: HILL0027.D
Als bottle: 34
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

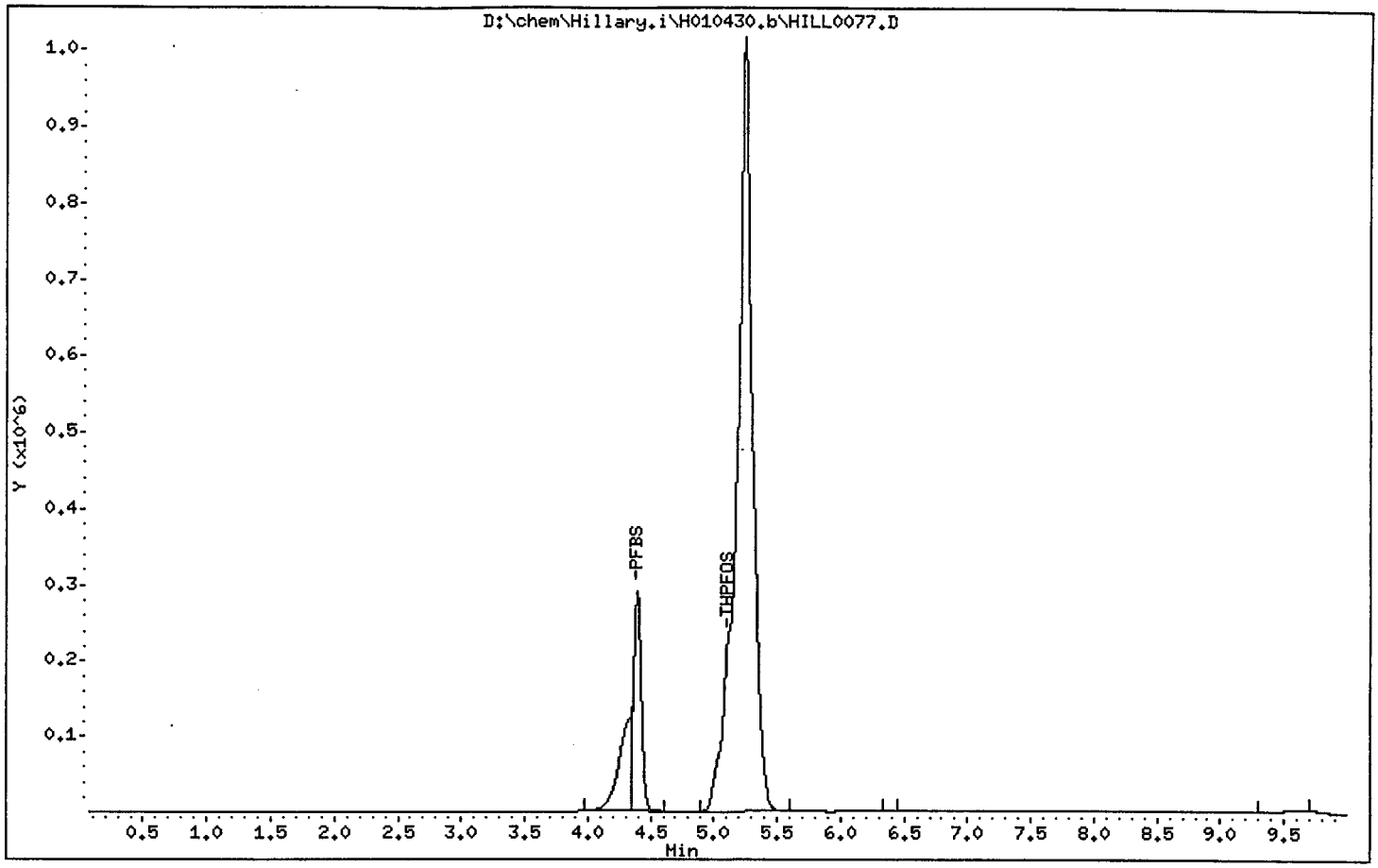
Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.104	5.108	(1.000)	1325618	254.000	
* 7 PFBS	299	4.397	4.380	(1.000)	2084860	254.000	
2 PFOS	499	5.237	5.318	(1.191)	7558687	292.440	292

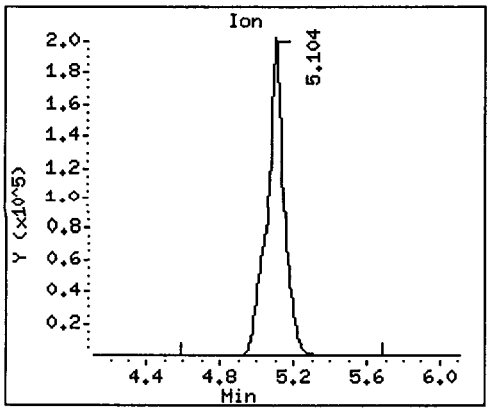
Exact Copy of Original
KLT Initial
05-16-01 Date

Data File: D:\chem\Hillary.i\H010430.b\HILL0077.D

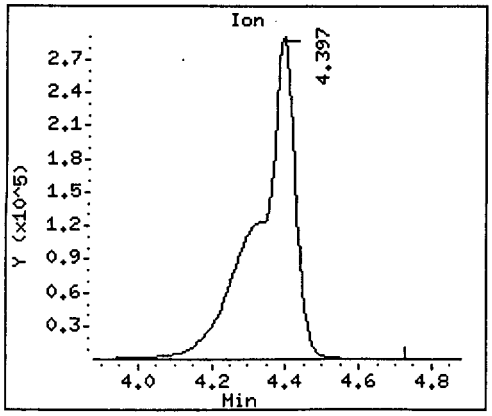
3M Environmental Laboratory
Project # E01-0768 Page 2



* 1 THPFOS



* 7 PFBS



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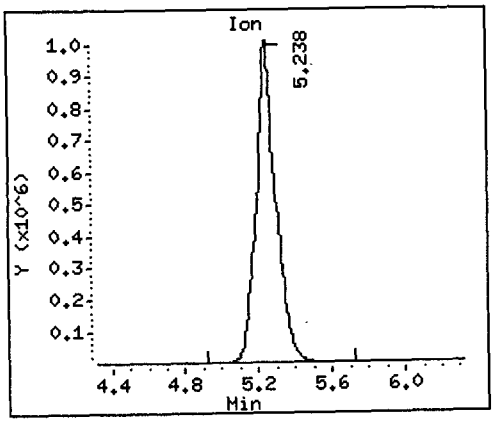
Initial

05-16-01
Date

Data File: D:\chem\Hillary.i\H010430.b\HILL0077.D

2 PFOS



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KG Initial 05-16-01 Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0119.D
Lab Smp Id: TN-A 4802 MeOH
Inj Date : 27-APR-2001 14:51
Operator : KLT Inst ID: hillary.i
Smp Info : TN-A 4802 MeOH
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:05 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 91
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	CONCENTRATIONS FINAL (ng/mL)
* 1 THPFOS	427	Compound Not Detected.					
* 7 PFBS	299	Compound Not Detected.					

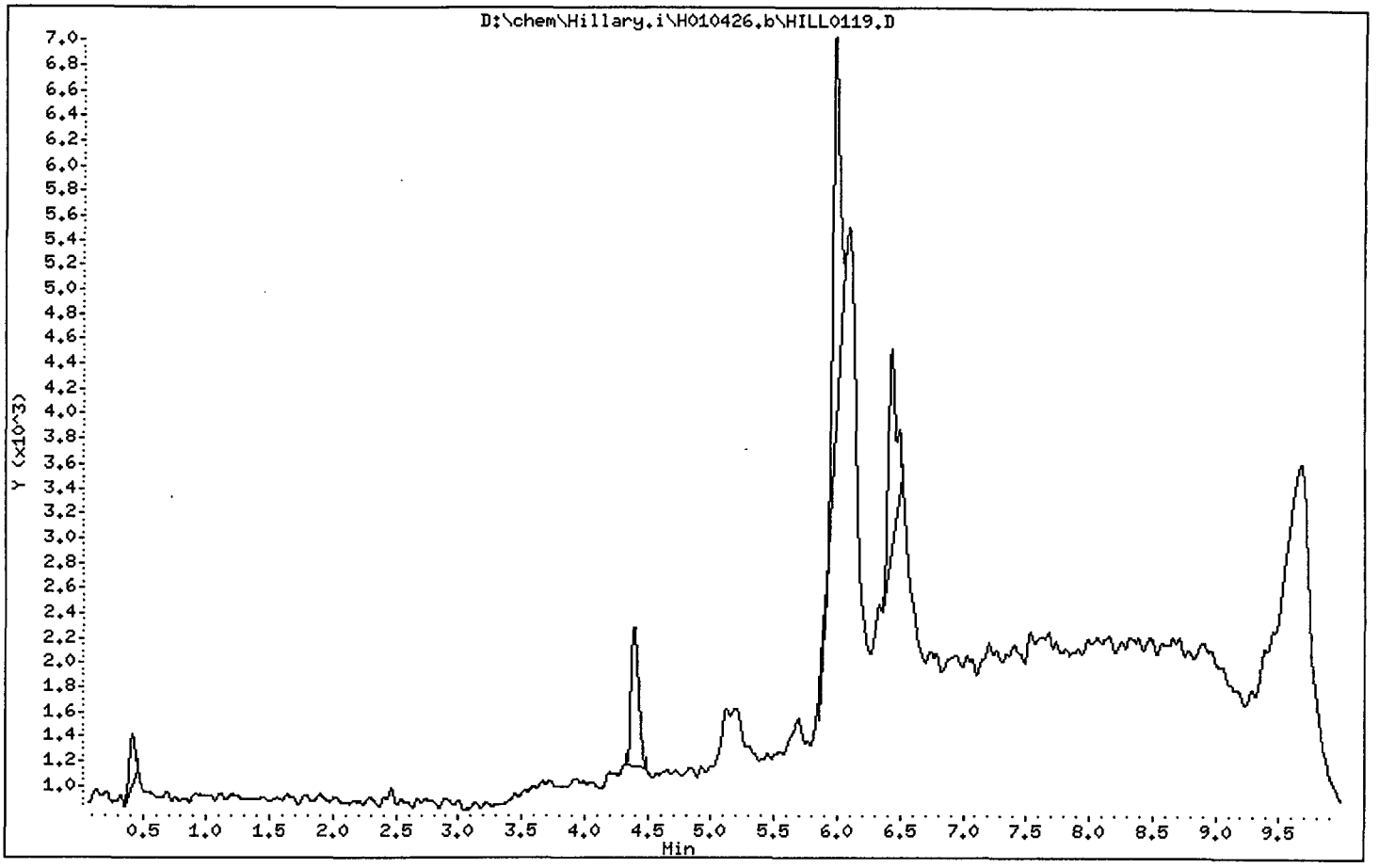
Exact Copy of Original

KL Initial

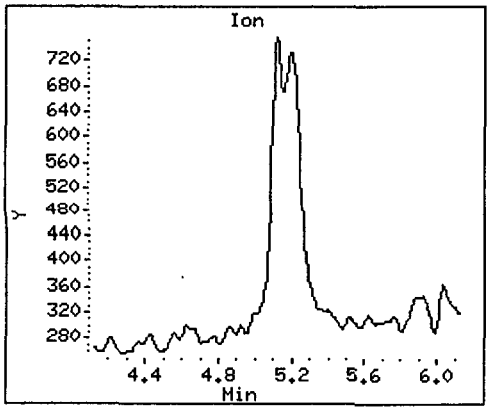
05-16-01 Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0119.D

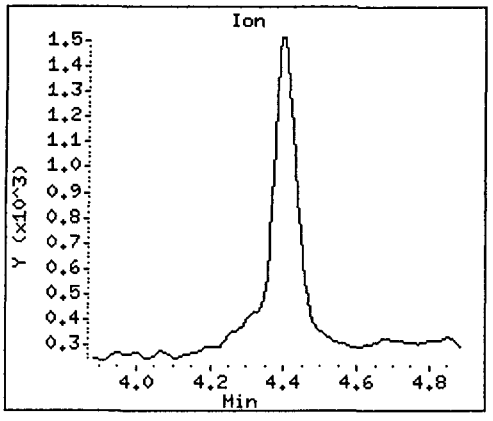
3M Environmental Laboratory
Project # E01-0768 Page 2



* 1 THPFOS (Undetected)



* 7 PFBS (Undetected)

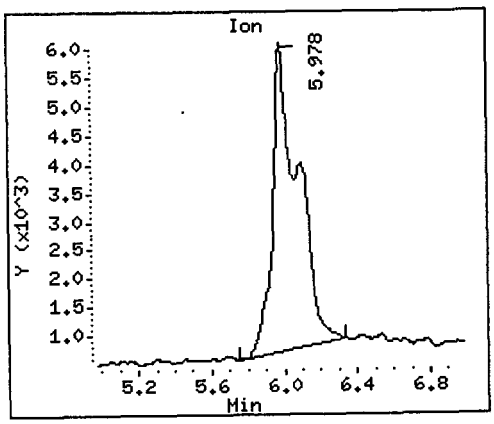


Exact Copy of Original

KC 05-16-01
Initial Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0119.D

2 FOSA



Exact Copy of Original

KG
Initial

05/16/01
Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0148.D
Lab Smp Id: TN-A 4802 MeOH
Inj Date : 27-APR-2001 20:15
Operator : KLT Inst ID: hillary.i
Smp Info : TN-A 4802 MeOH
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:08 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 92
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

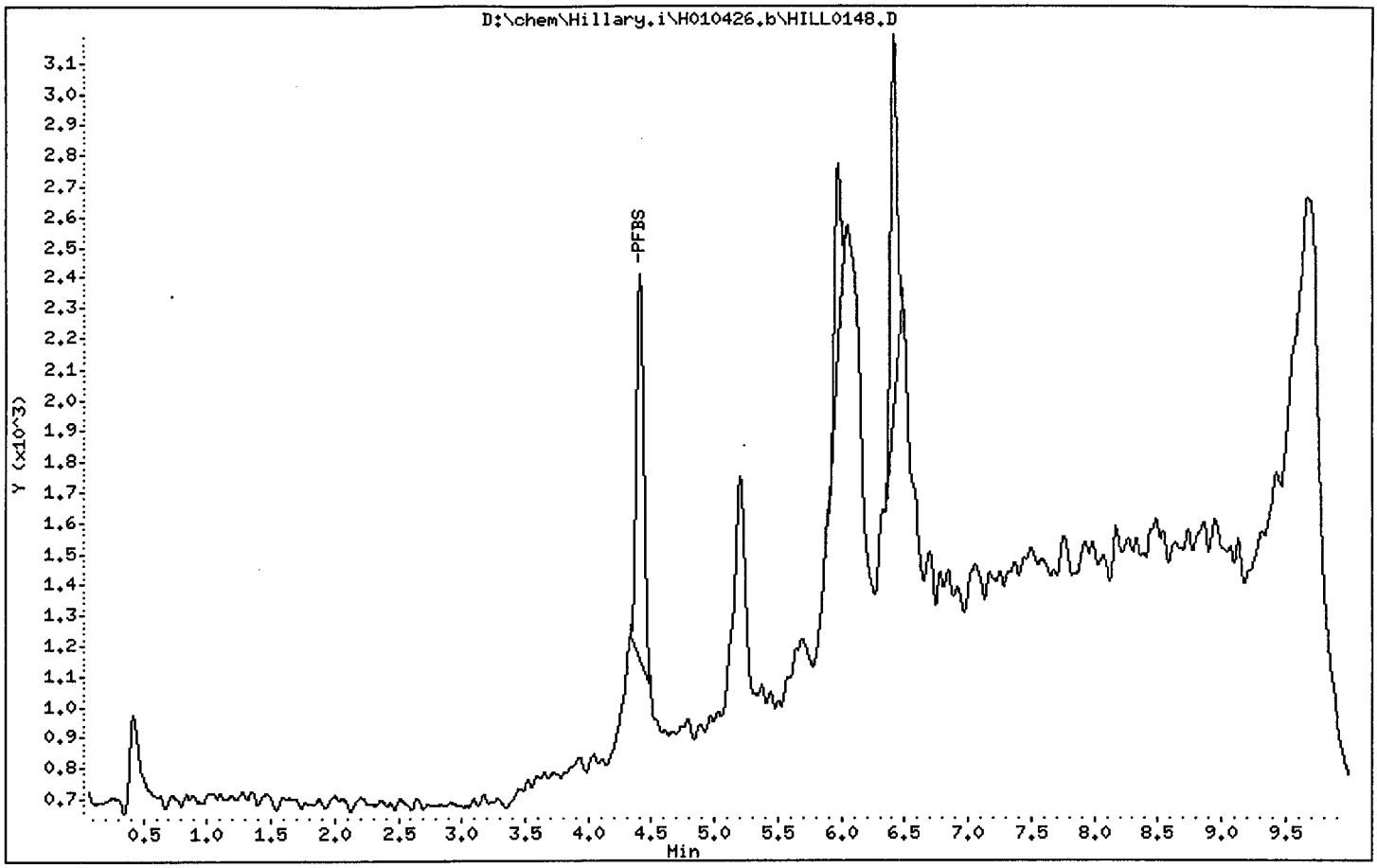
Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	Compound Not Detected.					
* 7 PFBS	299	4.409	4.387	(1.000)	10126	254.000	
2 FOSA	498	Compound Not Detected.					

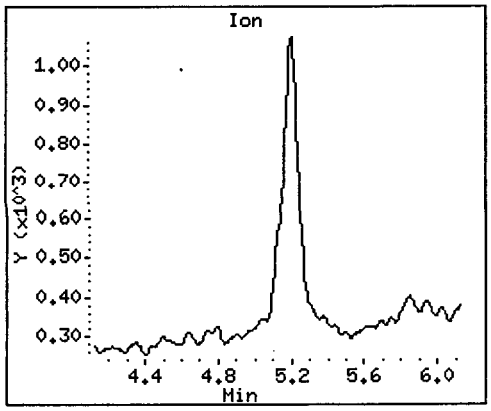
Exact Copy of Original
KLT Initial
05/16/01 Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0148.D

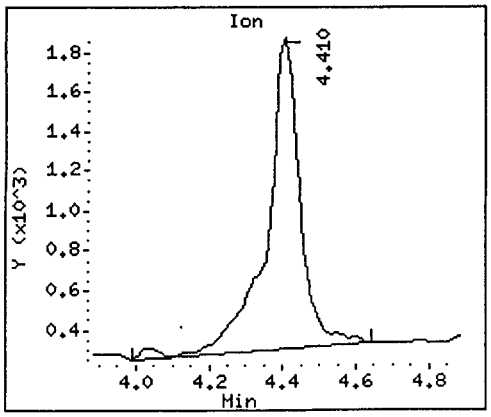
3M Environmental Laboratory
Project # E01-0768 Page 2



* 1 THPFOS (Undetected)



* 7 PFBS



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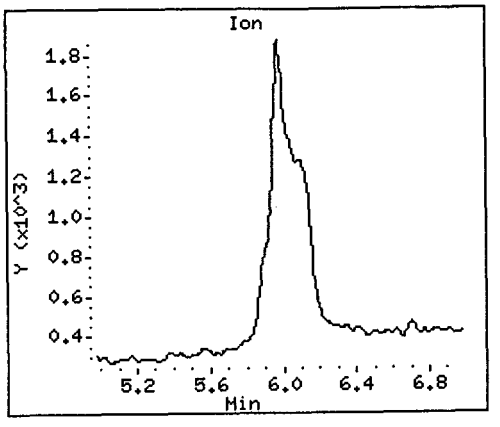
KT Initial

05-16-01 Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0148.D

2 FOSA (Undetected)



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05/16/01

Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0092.D
Lab Smp Id: TN-A 4802 MeOH
Inj Date : 02-MAY-2001 09:26
Operator : KLT Inst ID: hillary.i
Smp Info : TN-A 4802 MeOH
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501a.m
Meth Date : 03-May-2001 11:02 terrell Quant Type: ISTD
Cal Date : 02-MAY-2001 08:53 Cal File: HILL0089.D
Als bottle: 93
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	CONCENTRATIONS FINAL (ng/mL)
* 1 THPFOS	427	Compound Not Detected.					
* 2 PFBS	299	Compound Not Detected.					

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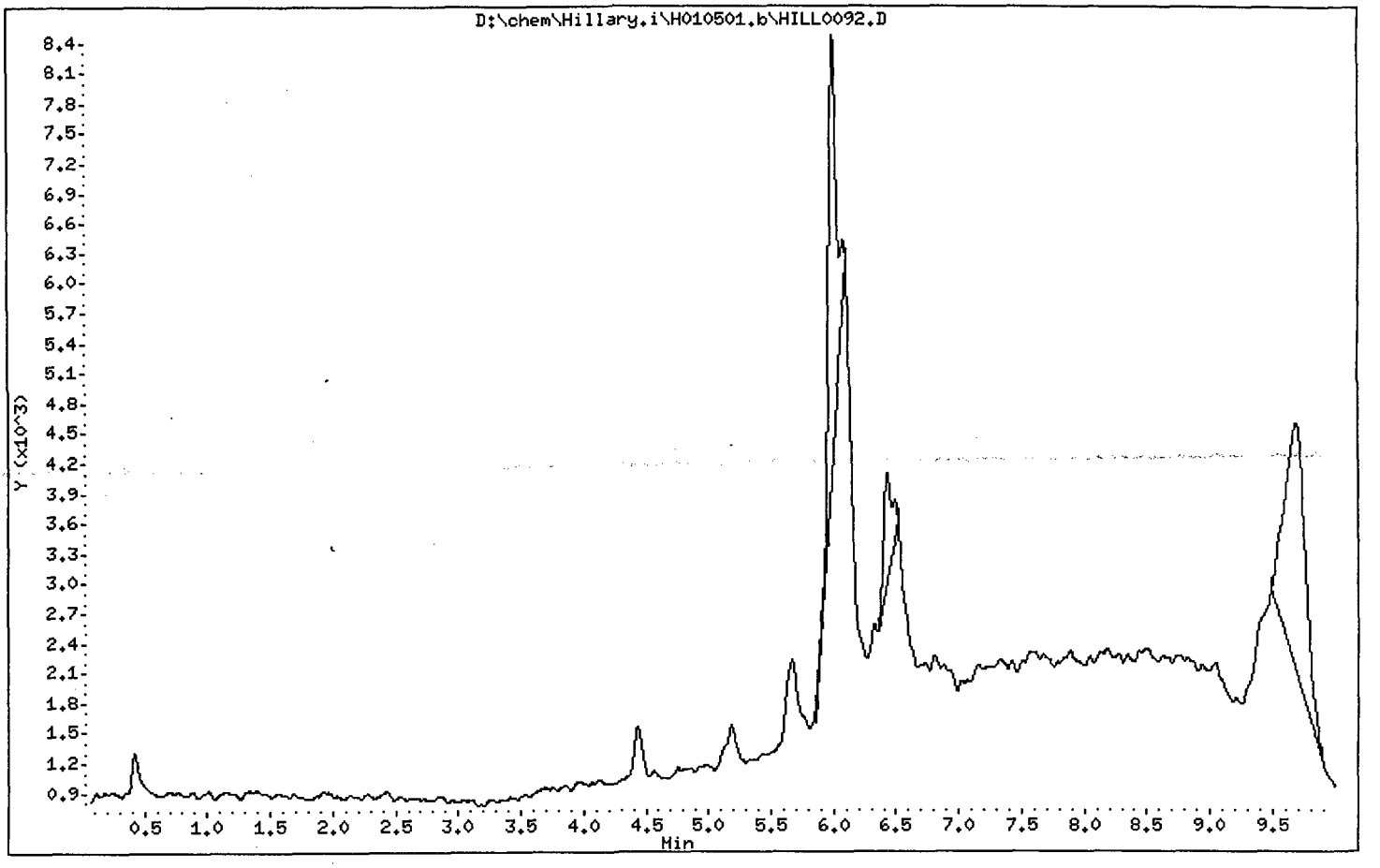
KLT
Initial

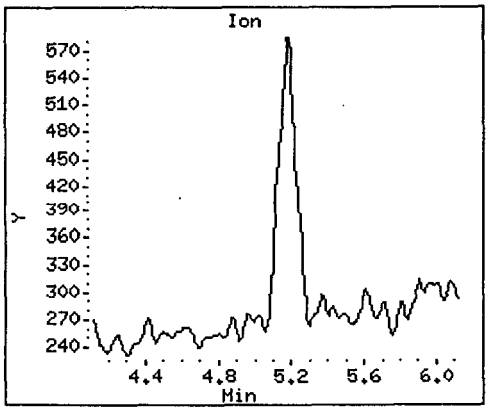
5/16/01
Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0092.D

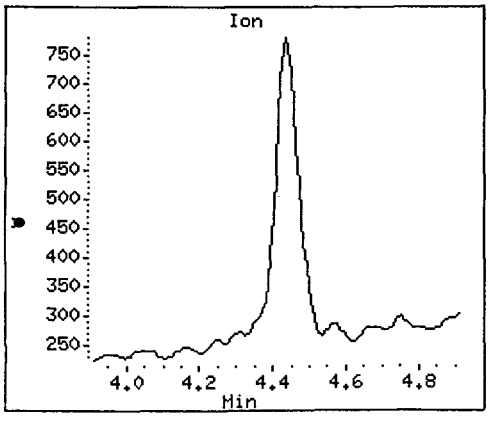
3M Environmental Laboratory
Project # E01-0768



* 1 THPFOS (Undetected)



* 2 PFBS (Undetected)



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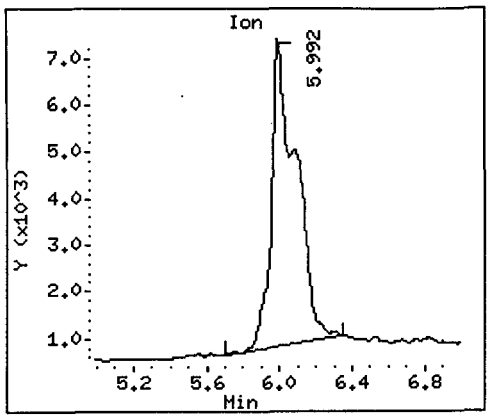
KLJ
Initial

05-Ko-01
Date

8RE KLJ 5-24-01

Data File: D:\chem\Hillary.i\H010501.b\HILL0092.D

3 FOSA



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KH
Initial

05/16/01
Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0121.D
Lab Smp Id: FOSA-1.4.1blk
Inj Date : 27-APR-2001 15:13
Operator : KLT Inst ID: hillary.i
Smp Info : 10X dil 042301-FOSA-1
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:05 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 55
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

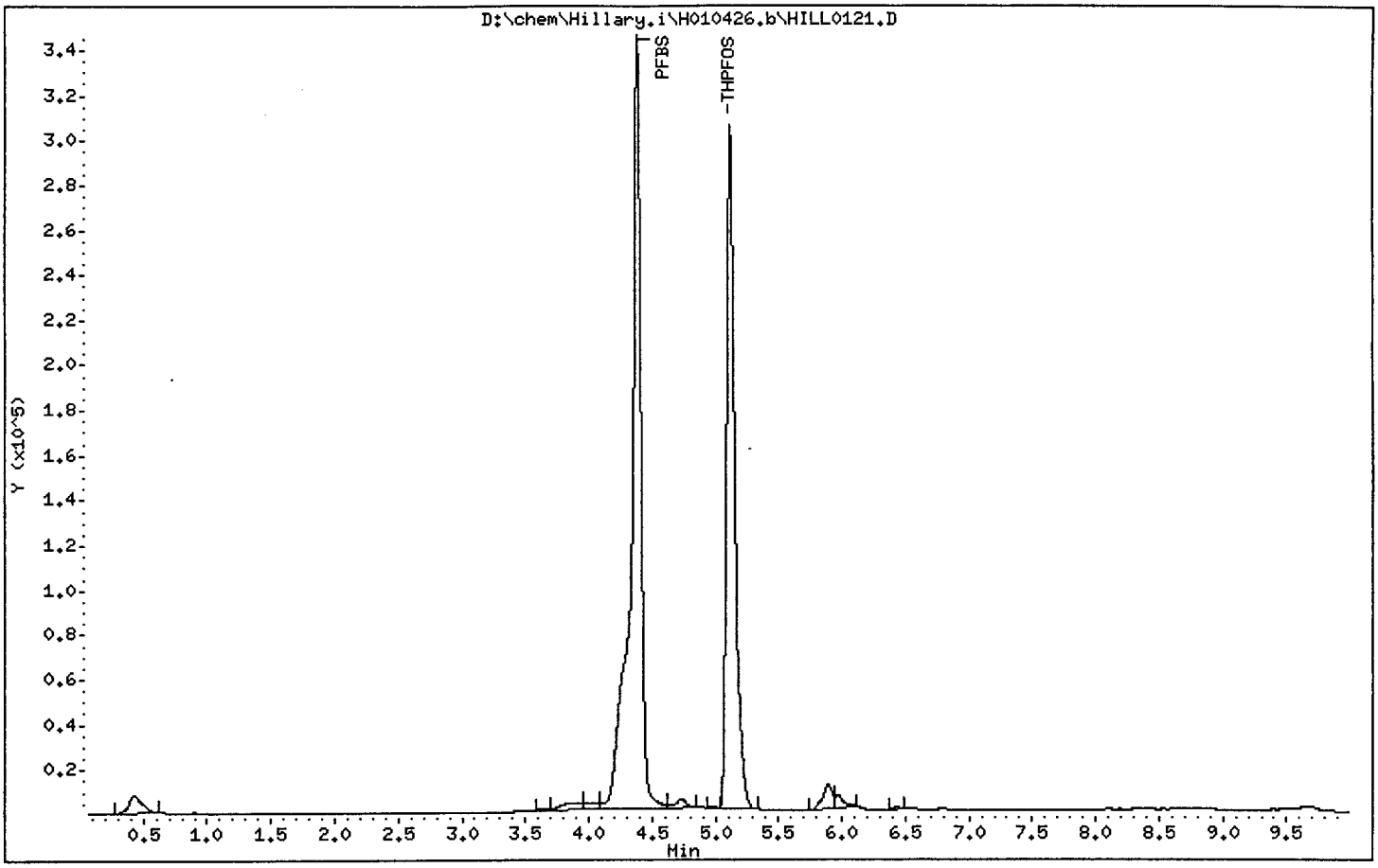
Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.108	5.122	(1.000)	1379938	254.000	
* 7 PFBS	299	4.379	4.387	(1.000)	1893699	254.000	
2 FOSA	498	5.892	5.977	(1.345)	106755	2.50445	2.50

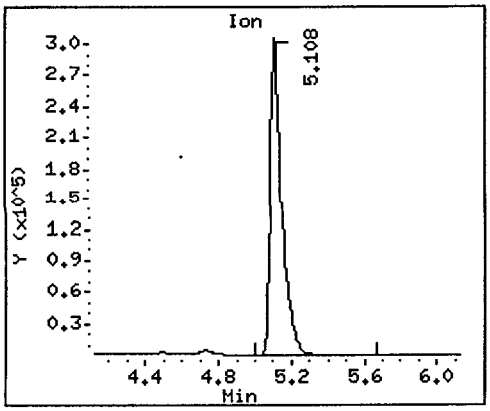
KLT
Initial
05-16-01
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0121.D

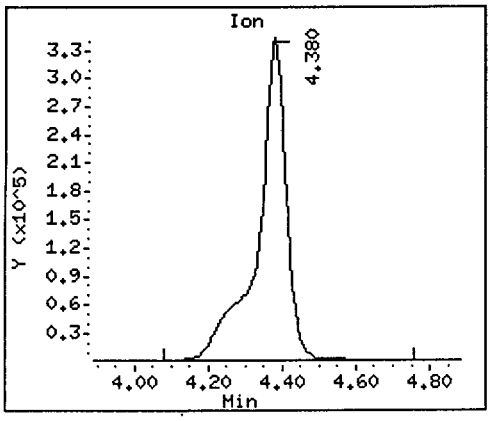
3M Environmental Laboratory
Project # E01-0768 Page 2



× 1 THPFOS



× 7 PFBS



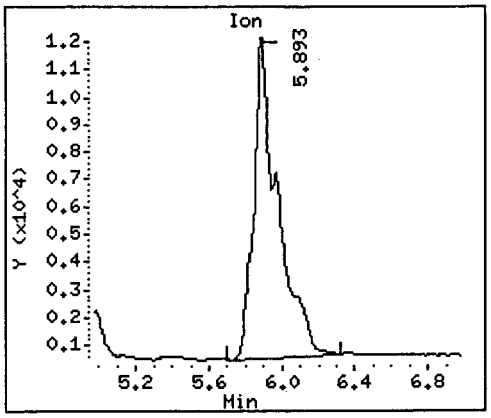
Exact Copy of Original

KLT
Initial

05/16/01
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0121.D

2 FOSA



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05/6-01
Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0144.D
Lab Smp Id: FOSA-2.4.1blk
Inj Date : 27-APR-2001 19:30
Operator : KLT Inst ID: hillary.i
Smp Info : 10X dil 042301-FOSA-2
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:08 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 71
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	CONCENTRATIONS FINAL (ng/mL)
* 1 THPFOS	427	5.111	5.122	(1.000)	1308838	254.000	
* 7 PFBS	299	4.383	4.387	(1.000)	1875879	254.000	
2 FOSA	498	Compound Not Detected.					

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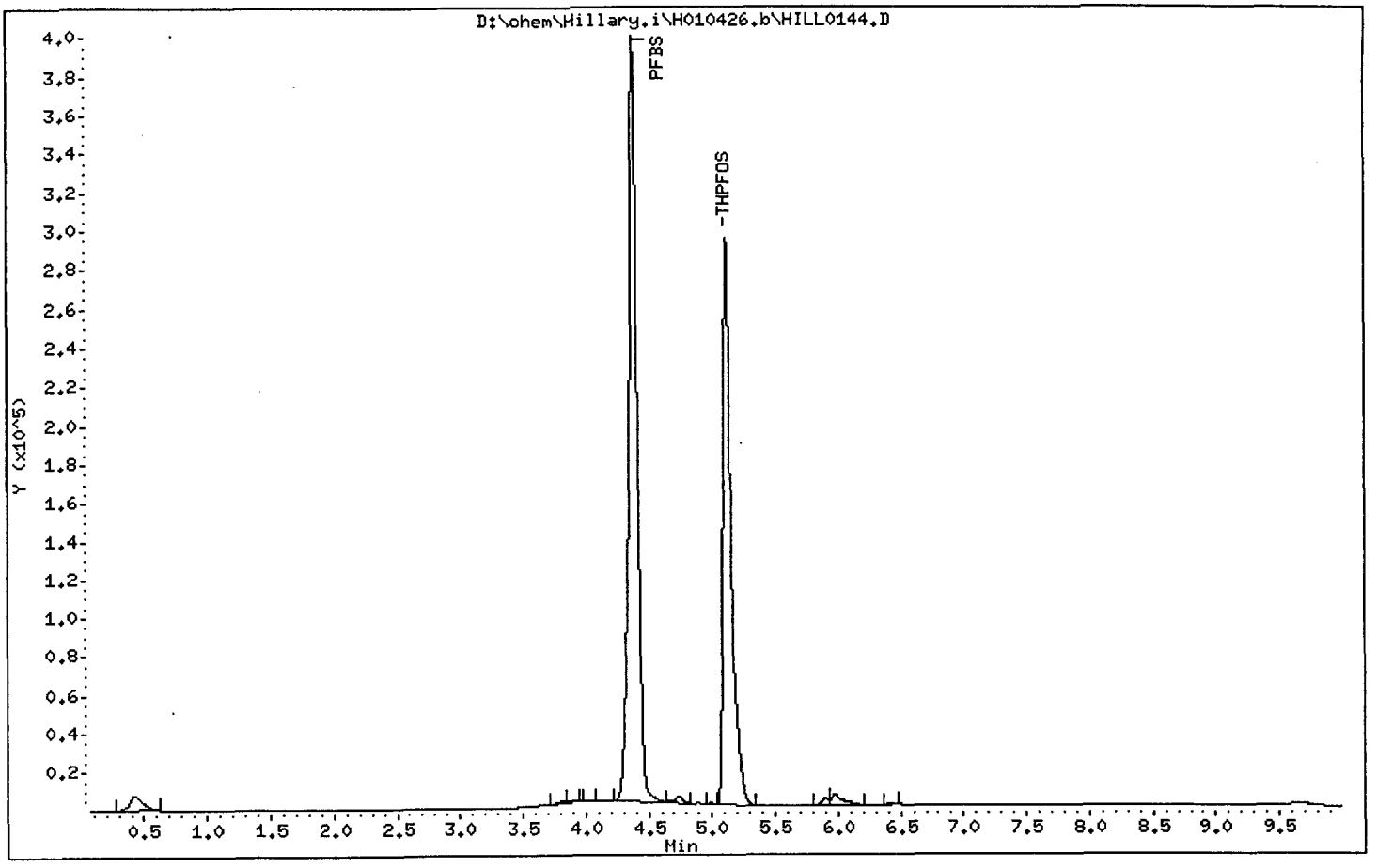
KLT
Analyst

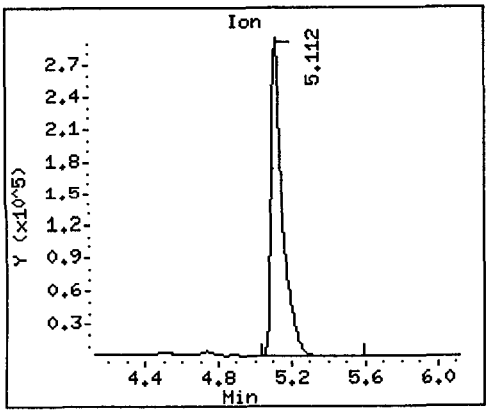
05-16-01
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0144.D

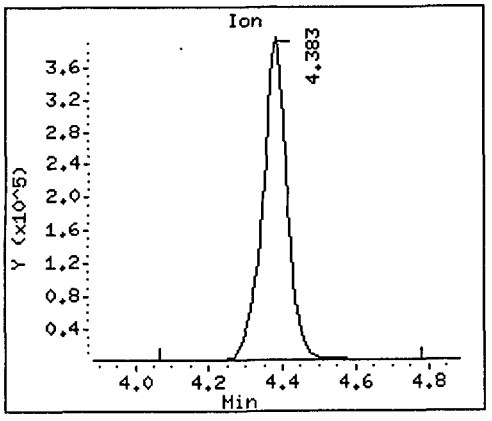
3M Environmental Laboratory
Project # E01-0768 Page 2



× 1 THPFOS



× 7 PFBS



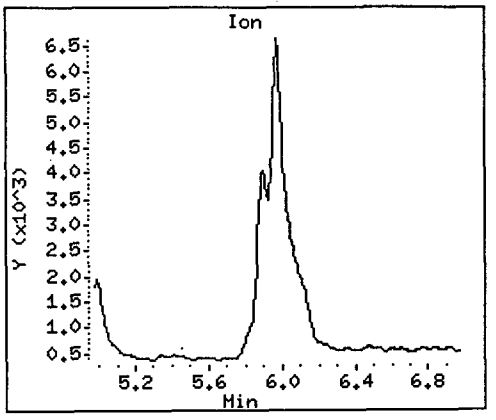
Exact Copy of Original

Initial

Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0144.D

2 FOSA (Undetected)



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Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0093.D
Lab Smp Id: FOSA-3.4.1blk
Inj Date : 02-MAY-2001 09:38
Operator : KLT Inst ID: hillary.i
Smp Info : FOSA Urine Solub Day 3 Re-diluted samples 5-01-01
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501a.m
Meth Date : 03-May-2001 11:02 terrell Quant Type: ISTD
Cal Date : 02-MAY-2001 08:53 Cal File: HILL0089.D
Als bottle: 71
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.117	5.118	(1.000)	1236810	254.000	
* 2 PFBS	299	4.396	4.410	(1.000)	1629186	254.000	
3 FOSA	498	6.007	5.993	(1.366)	553440	19.8742	19.9

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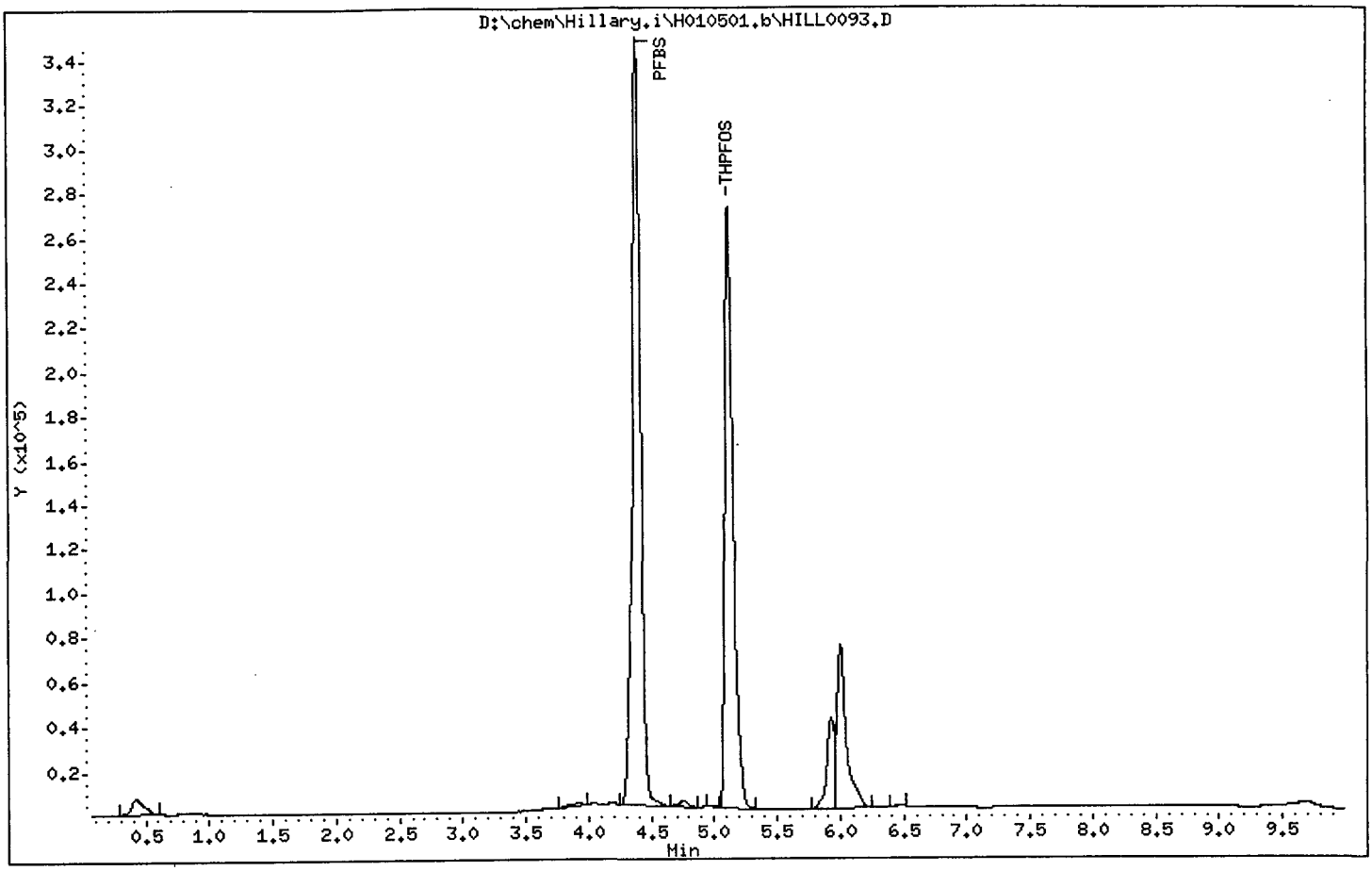
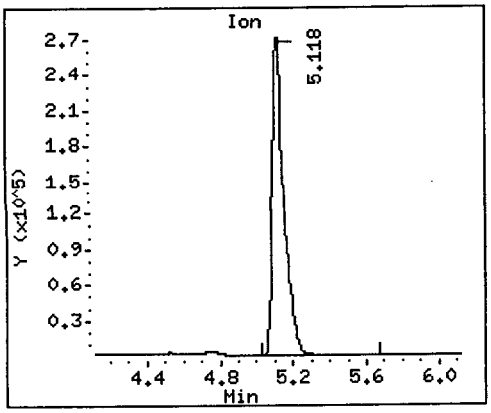
KLT Initial 05/16/01 Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0093.D

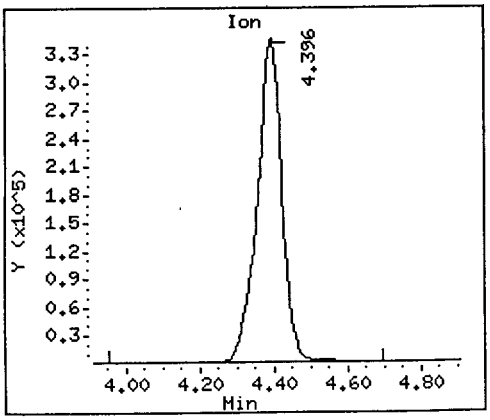
3M Environmental Laboratory
Project # E01-0768 Page 2



* 1 THPFOS



* 2 PFBS



Exact Copy of Original

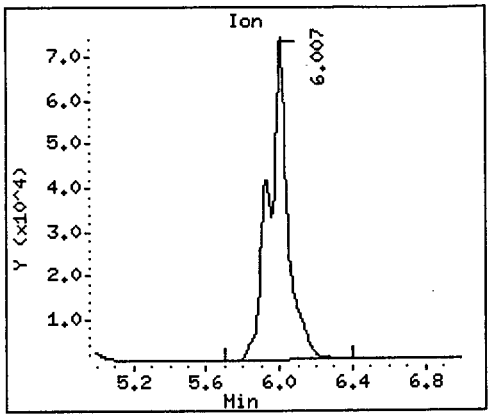
KG Initial

05-16-01 Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0093.D

3M Environmental Laboratory
Project # E01-0768

3 FOSA



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Initial

Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0126.D
Lab Smp Id: FOSA-1.1.1
Inj Date : 27-APR-2001 16:09
Operator : KLT Inst ID: hillary.i
Smp Info : 10X dil 042301-FOSA-1
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:05 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 59
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

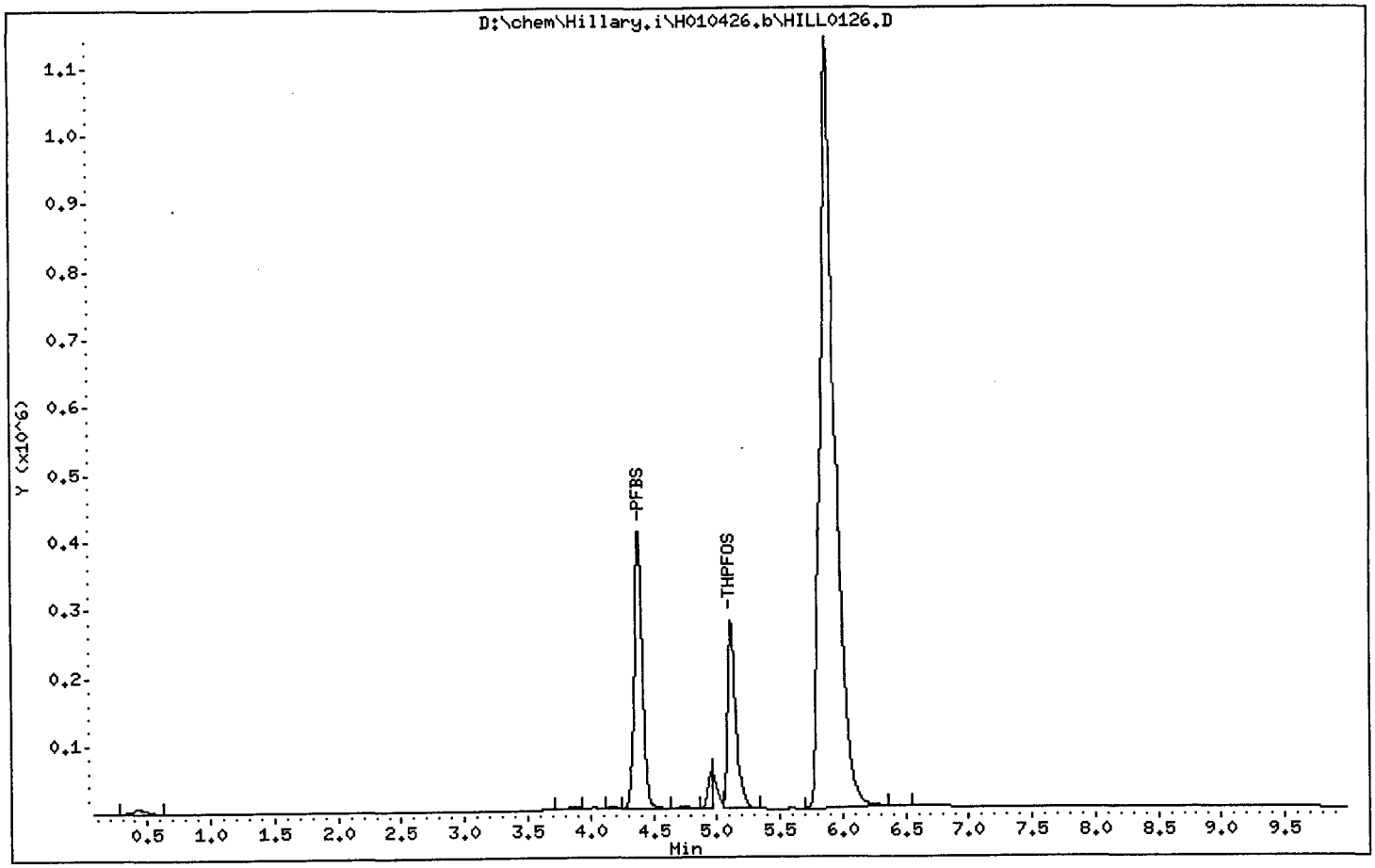
Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.112	5.122	(1.000)	1223896	254.000	
* 7 PFBS	299	4.390	4.387	(1.000)	1863247	254.000	
2 FOSA	498	5.889	5.977	(1.341)	9090670	342.874	343

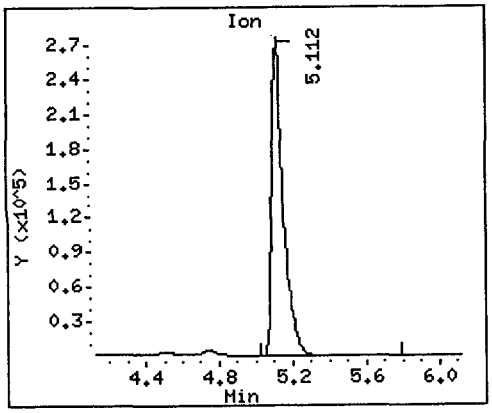
Exact Copy of Original
KLT Initial 05/16/01 Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0126.D

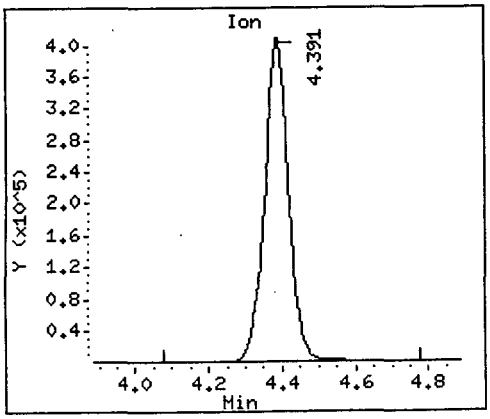
3M Environmental Laboratory
Project # E01-0768



✖ 1 THPFOS



✖ 7 PFBS



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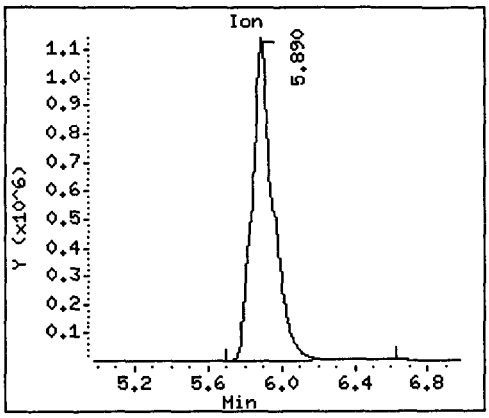
Data File: D:\chem\Hillary.i\H010426.b\HILL0126.D

3M Environmental Laboratory
Project # E01-0768 Page 3

2 FOSA



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Initial Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0130.D
Lab Smp Id: FOSA-1.2.1
Inj Date : 27-APR-2001 16:54
Operator : KLT Inst ID: hillary.i
Smp Info : 10X dil 042301-FOSA-1
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:05 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 63
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.118	5.122	(1.000)	1223960	254.000	
* 7 PFBS	299	4.382	4.387	(1.000)	1887871	254.000	
2 FOSA	498	5.888	5.977	(1.344)	8385467	309.497	309

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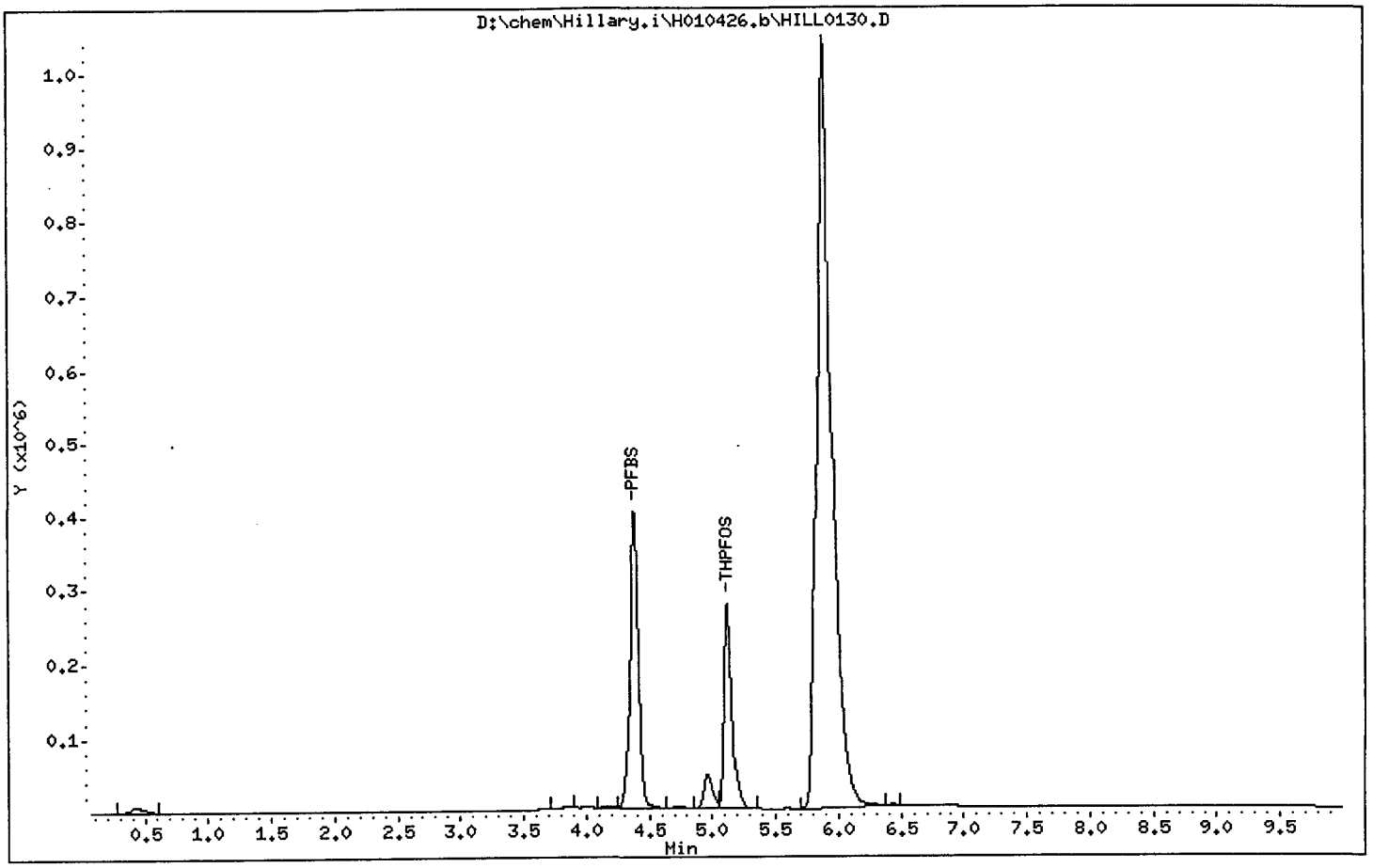
Data File: D:\chem\Hillary.i\H010426.b\HILL0130.D

3M Environmental Laboratory
Project # E01-0768 Page 2



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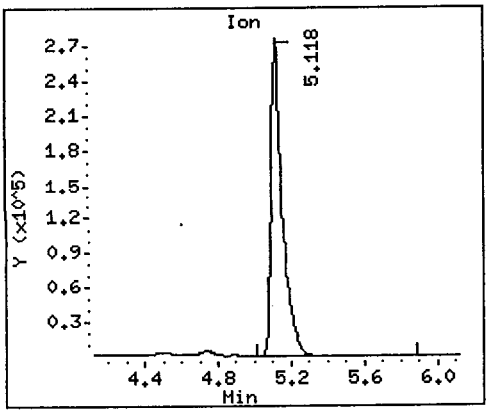

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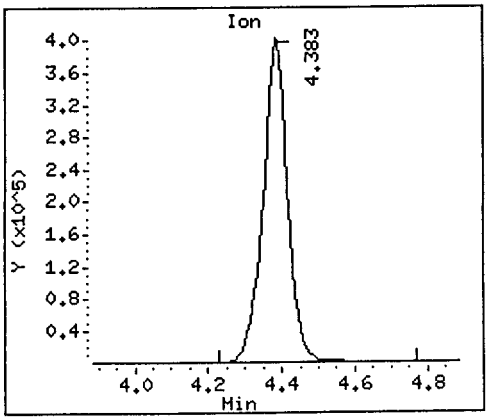

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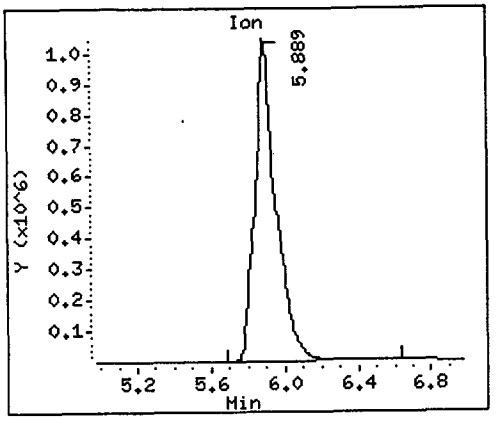
Initial

05-16-01
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0130.D

2 FOSA



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05/16/01
Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0138.D
Lab Smp Id: FOSA-1.3.1
Inj Date : 27-APR-2001 18:23
Operator : KLT Inst ID: hillary.i
Smp Info : 10X dil 042301-FOSA-1
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:05 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 67
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.111	5.122	(1.000)	1177514	254.000	
* 7 PFBS	299	4.390	4.387	(1.000)	1900215	254.000	
2 FOSA	498	5.881	5.977	(1.340)	6883587	248.307	248

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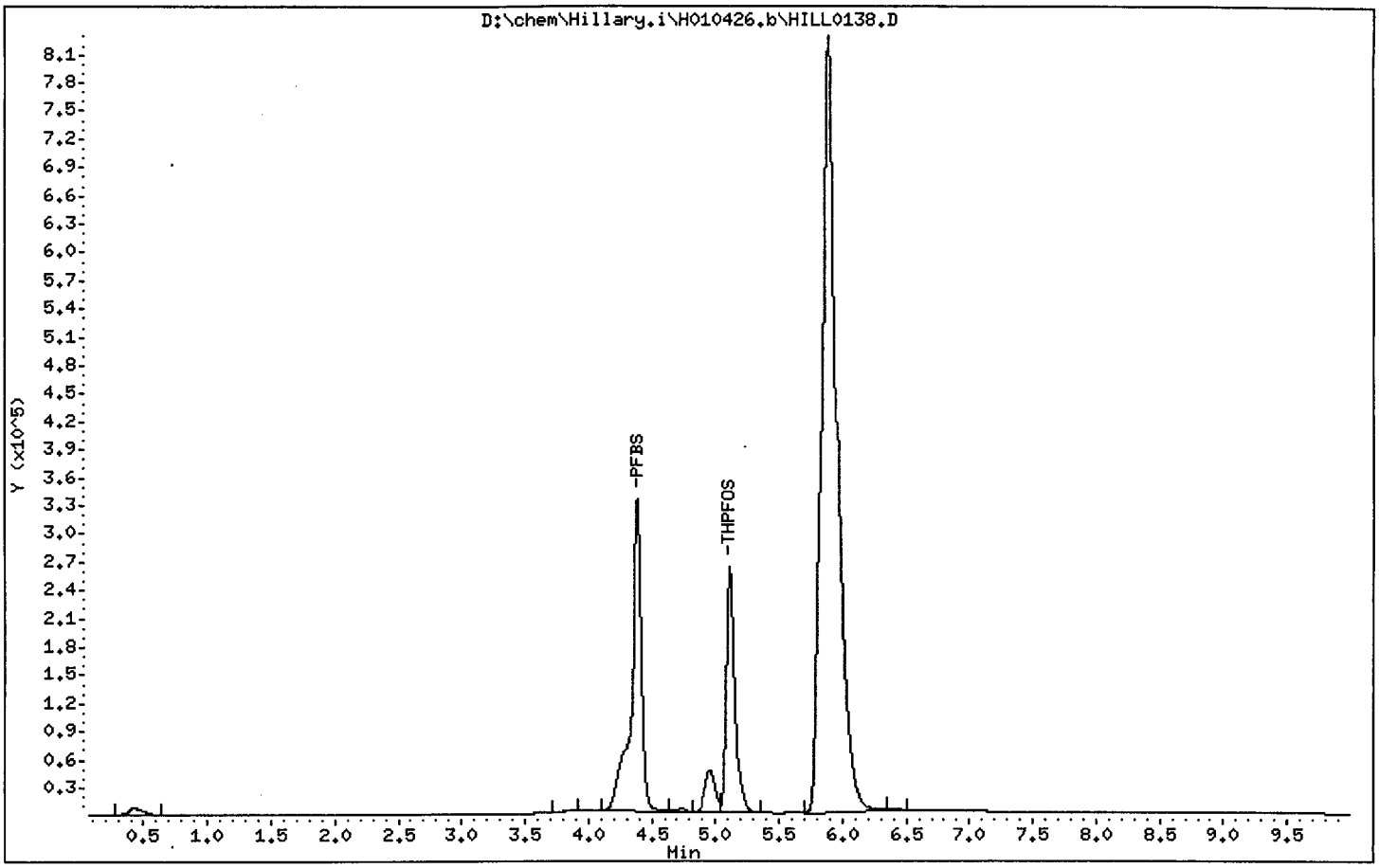
KLT Initial 05/16/01 Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0138.D

3M Environmental Laboratory
Project # E01-0768 Page 2



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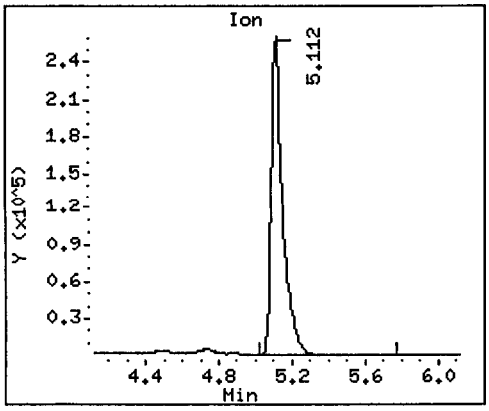

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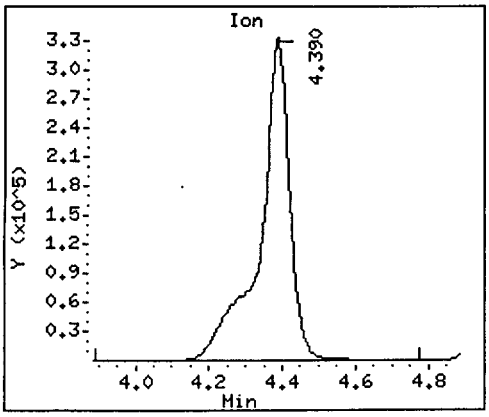

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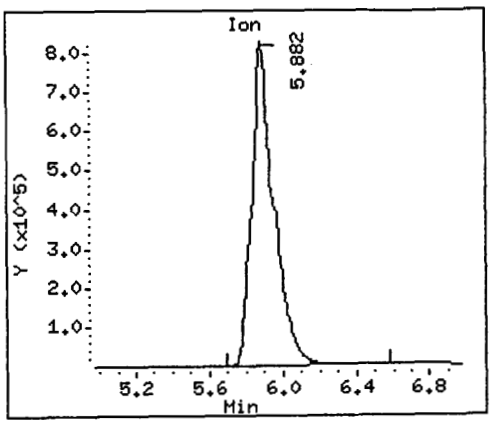
05/16/01
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0138.D

2 FOSA



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Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0152.D
Lab Smp Id: FOSA-2.1.1
Inj Date : 27-APR-2001 21:00
Operator : KLT Inst ID: hillary.i
Smp Info : 10X dil 042301-FOSA-2
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:08 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 75
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.115	5.122	(1.000)	1110585	254.000	
* 7 PFBS	299	4.387	4.387	(1.000)	1783250	254.000	
2 FOSA	498	5.879	5.977	(1.340)	8103858	317.291	317

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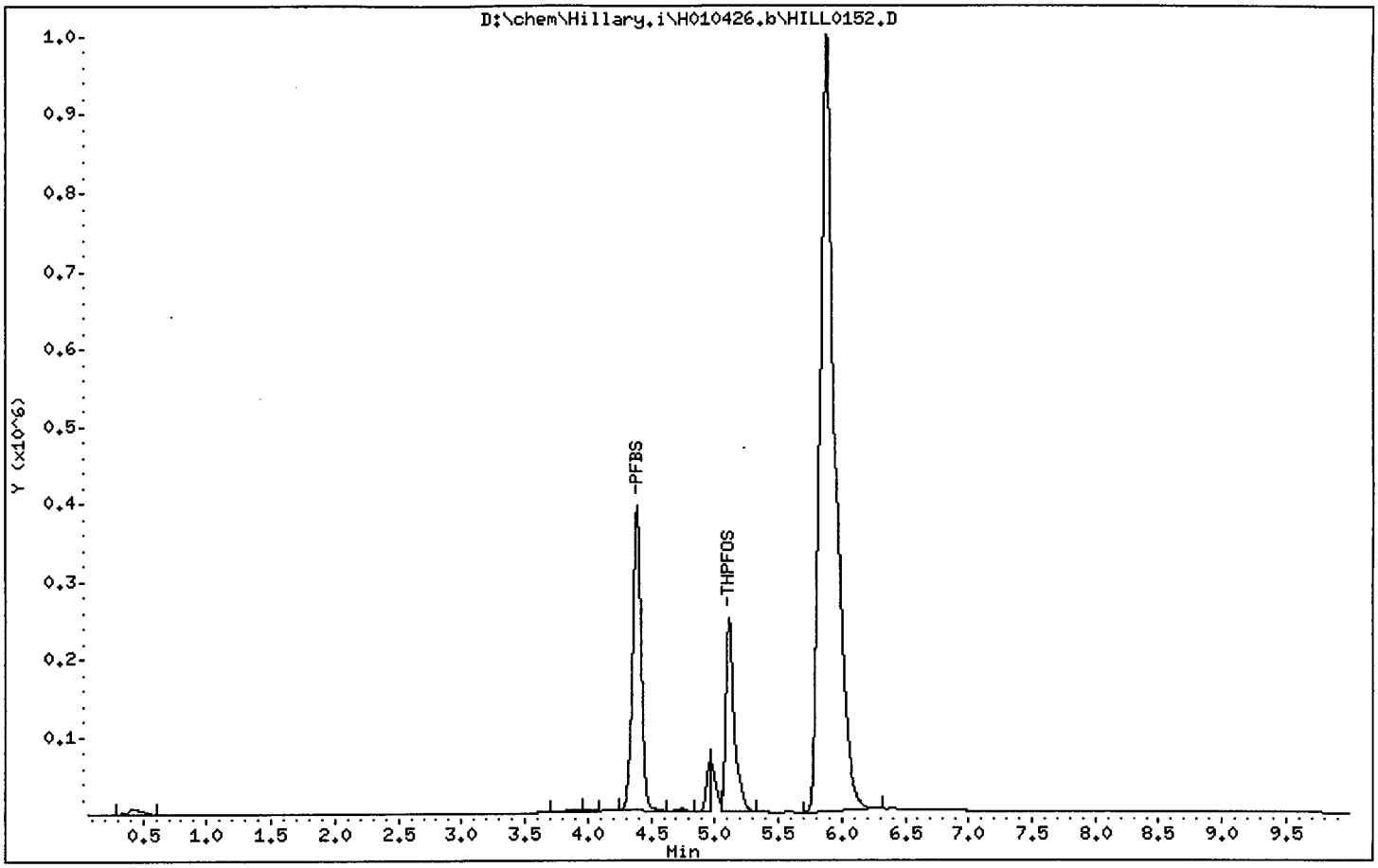
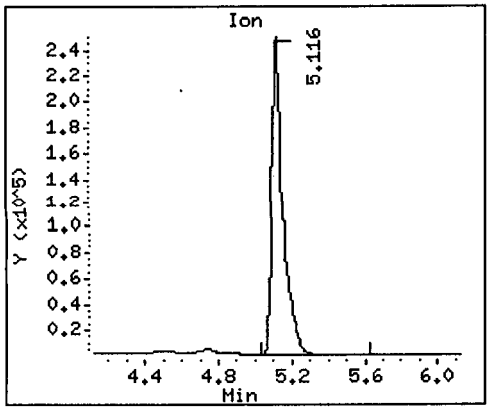
Data File: D:\chem\Hillary.i\H010426.b\HILL0152.D

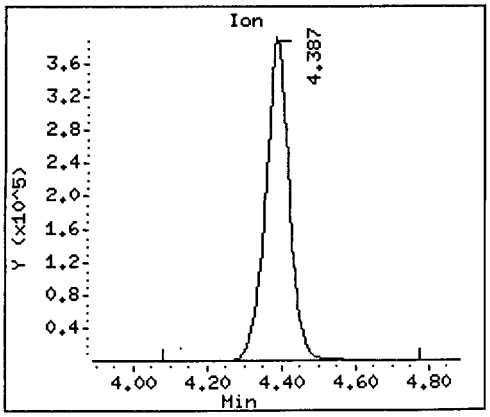
3M Environmental Laboratory
Project # E01-0768 Page 2



* 1 THPFOS



* 7 PFBS



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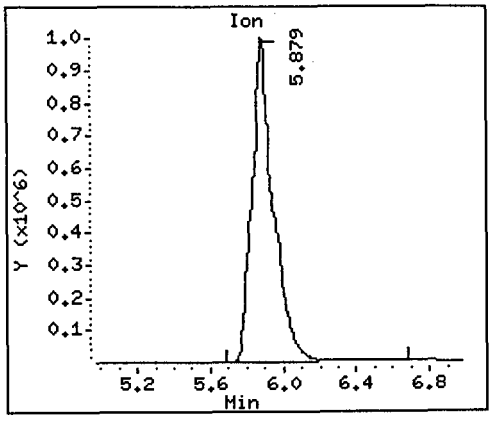
05-16-01
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0152.D

3M Environmental Laboratory
Project # E01-0768

2 FOSA



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Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0157.D
Lab Smp Id: FOSA-2.2.1
Inj Date : 27-APR-2001 21:56
Operator : KLT Inst ID: hillary.i
Smp Info : 10X dil 042301-FOSA-2
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:08 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 79
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.116	5.122	(1.000)	1202436	254.000	
* 7 PFBS	299	4.387	4.387	(1.000)	1863768	254.000	
2 FOSA	498	5.886	5.977	(1.342)	7993752	297.956	298

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Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0157.D

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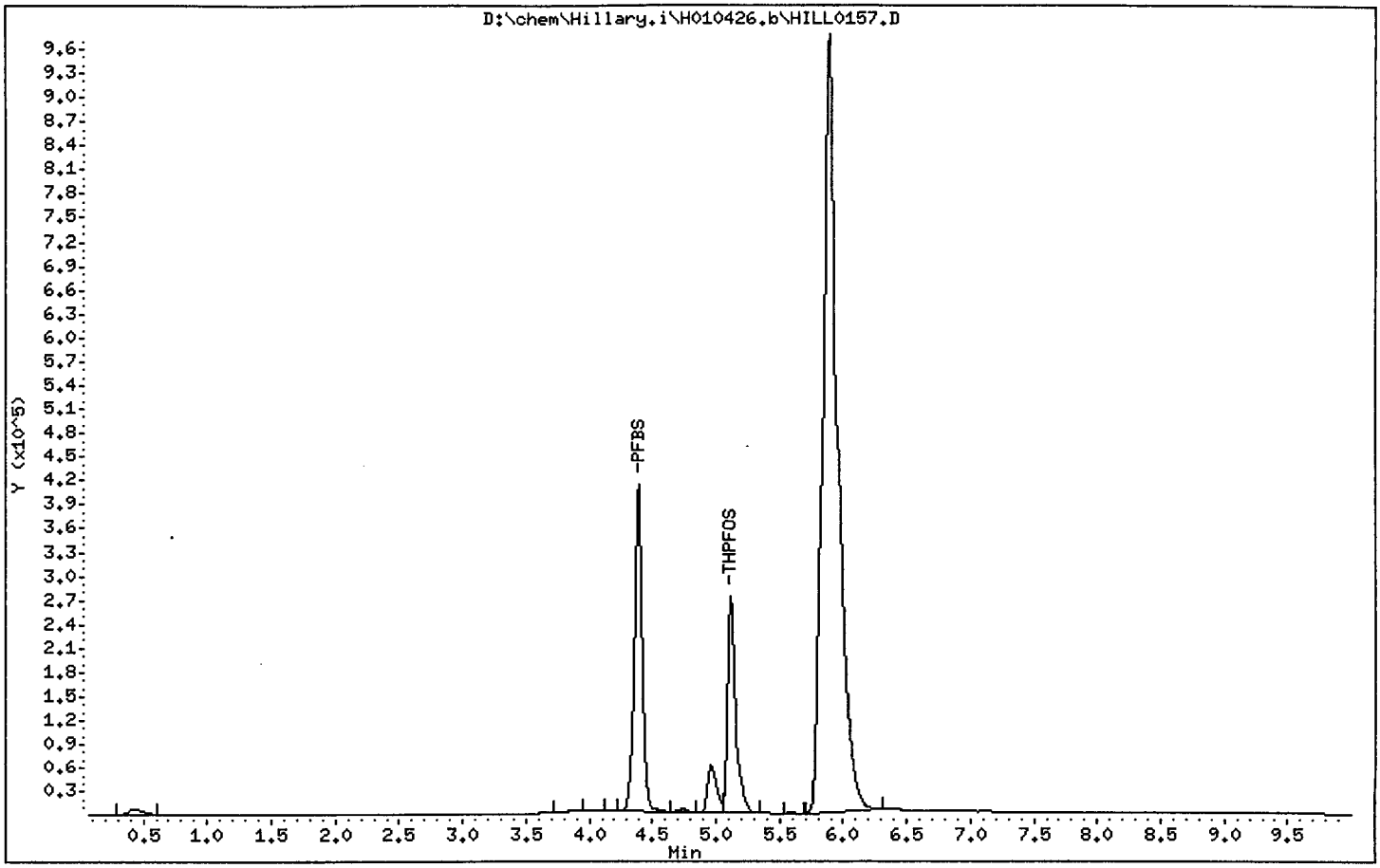
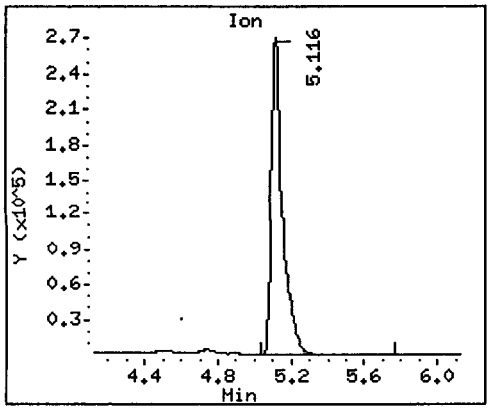
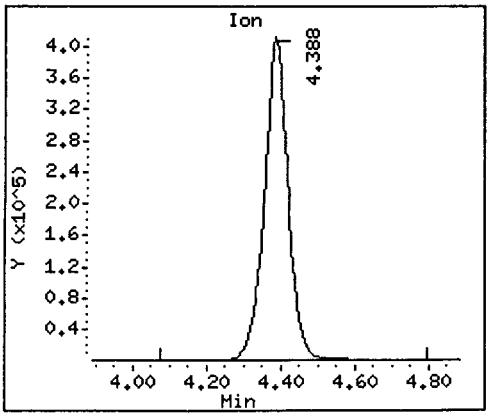
Project # E01-0768 Page 2



* 1 THPFOS



* 7 PFBS



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(Initial) (Date)

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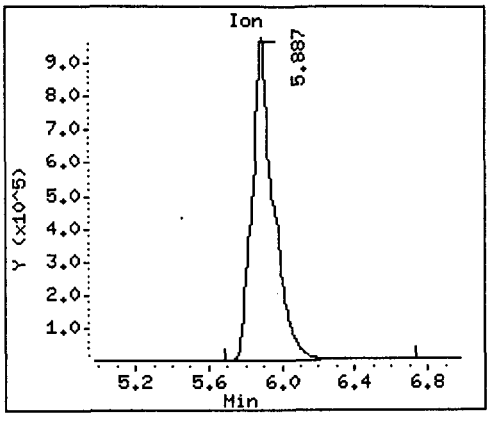
2 FOSA



Original Copy of Signature
- KLT
05/16/01

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0165.D
Lab Smp Id: FOSA-2.3.1
Inj Date : 27-APR-2001 23:25
Operator : KLT Inst ID: hillary.i
Smp Info : 10X dil 042301-FOSA-2
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:08 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 14:17 Cal File: HILL0116.D
Als bottle: 83
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	CONCENTRATIONS FINAL (ng/mL)
* 1 THPFOS	427	5.116	5.122	(1.000)	1158902	254.000	
* 7 PFBS	299	4.394	4.387	(1.000)	1883593	254.000	
2 FOSA	498	5.879	5.977	(1.338)	7850050	288.823	289

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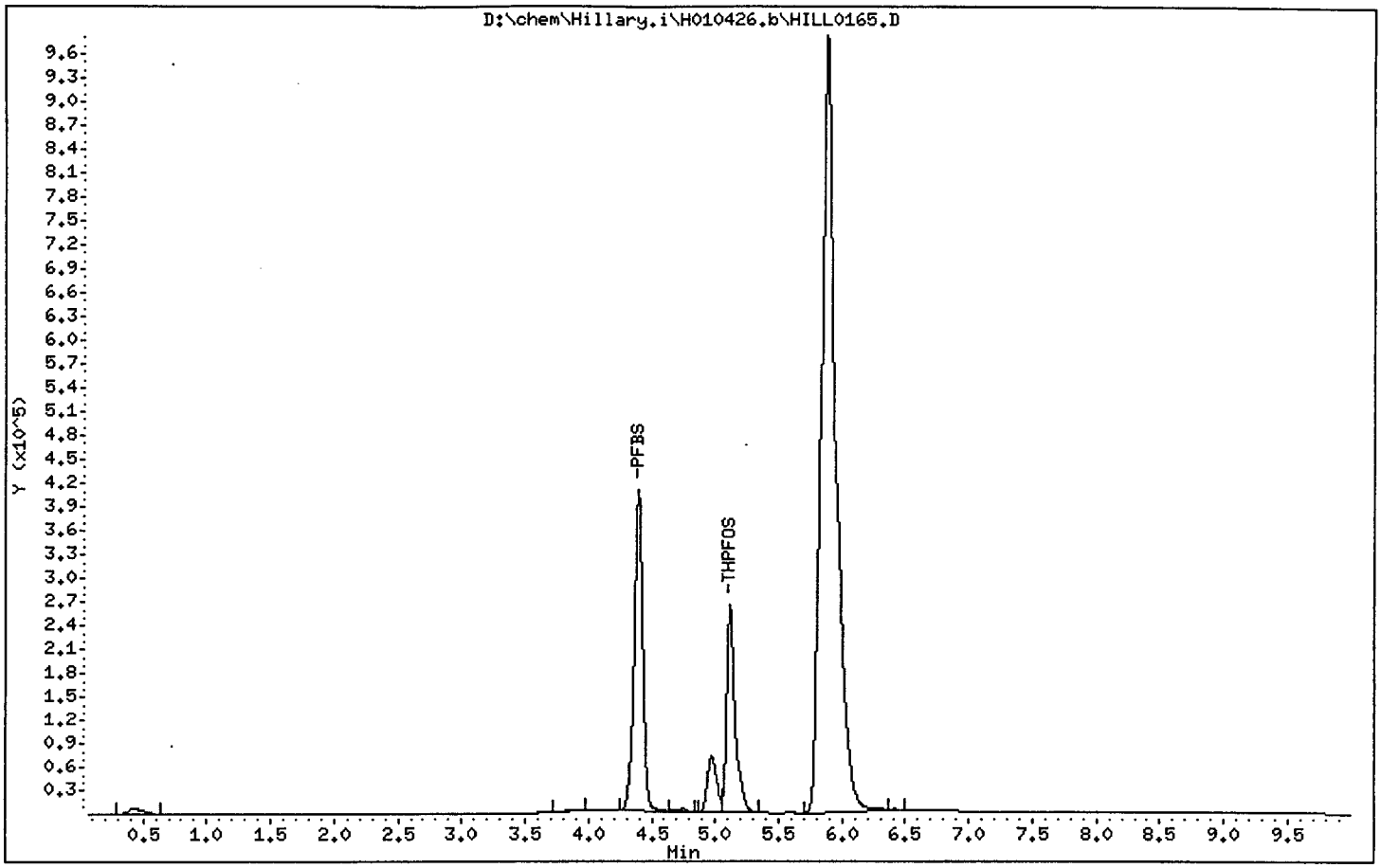
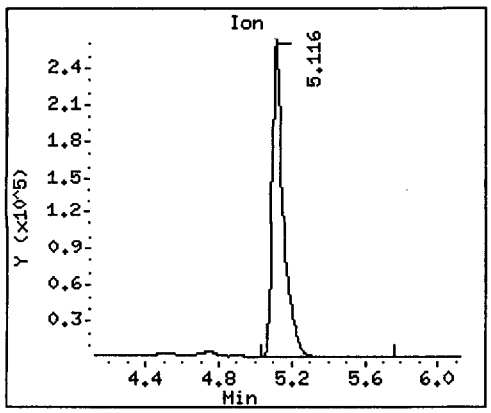
KT 05-16-01
Initial Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0165.D

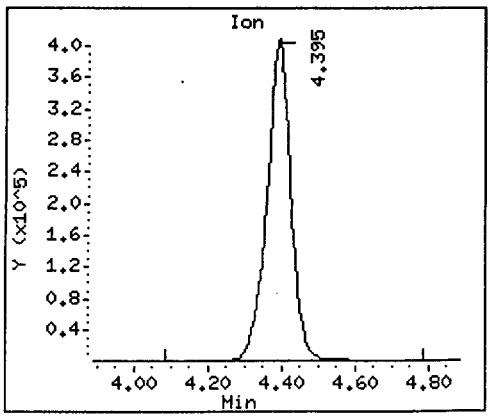
3M Environmental Laboratory
Project # E01-0768 Page 2



* 1 THPFOS



* 7 PFBS



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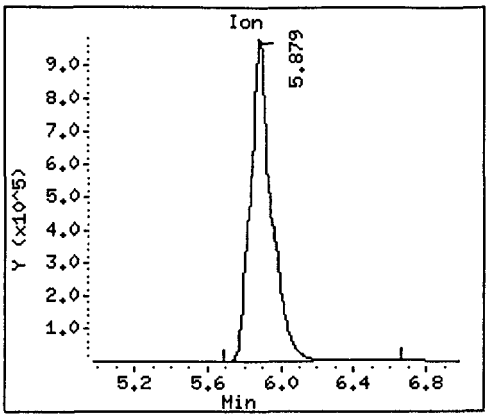
Initial

Date

2 FOSA



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KL 05/16/01
Initial Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0097.D
Lab Smp Id: FOSA-3.1.1
Inj Date : 02-MAY-2001 10:22
Operator : KLT Inst ID: hillary.i
Smp Info : FOSA Urine Solub Day 3 Re-diluted samples 5-01-01
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501a.m
Meth Date : 03-May-2001 11:02 terrell Quant Type: ISTD
Cal Date : 02-MAY-2001 08:53 Cal File: HILL0089.D
Als bottle: 75
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.117	5.118	(1.000)	1186398	254.000	
* 2 PFBS	299	4.403	4.410	(1.000)	1735419	254.000	
3 FOSA	498	5.909	5.993	(1.342)	7201703	268.781	269

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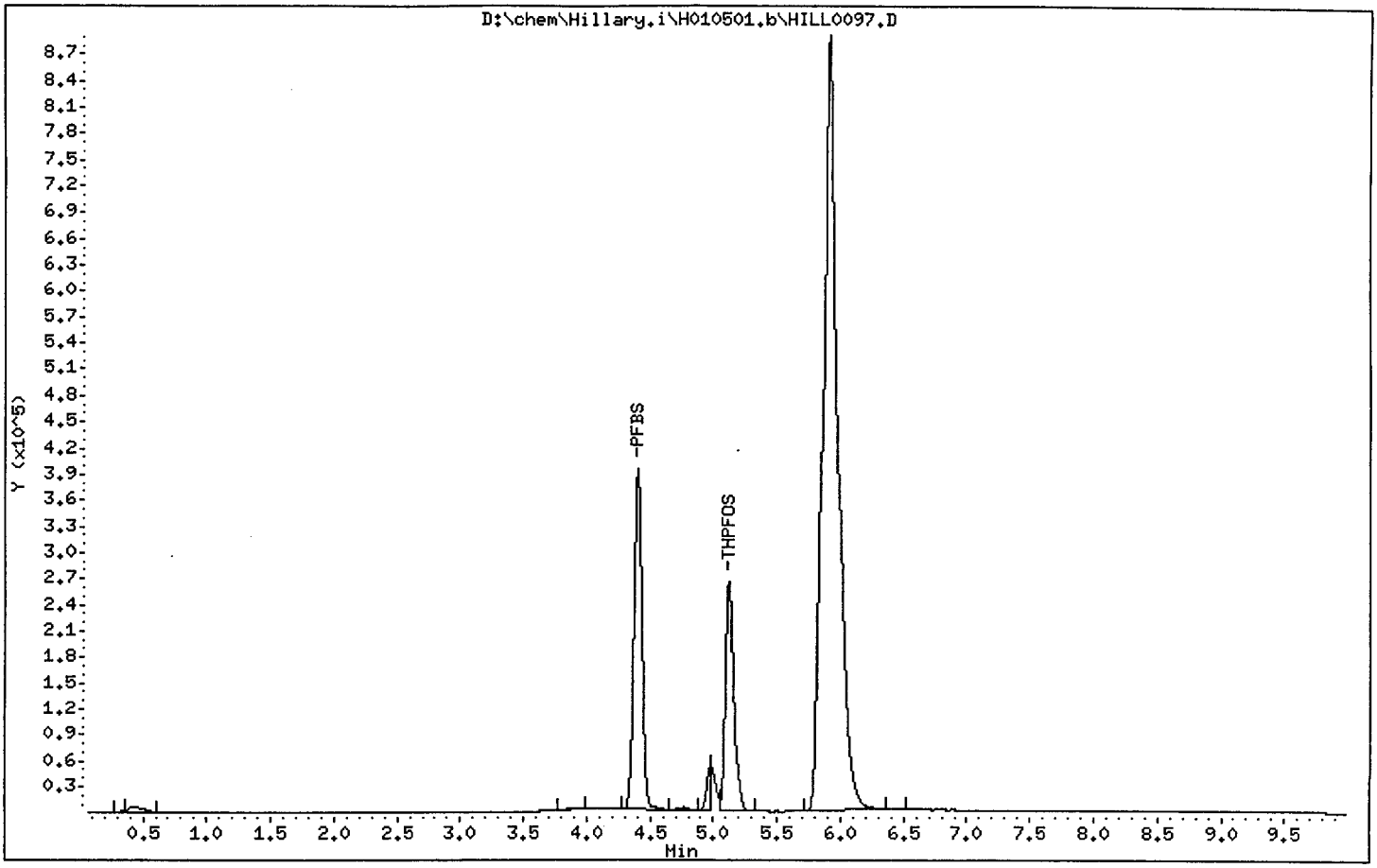
Initial

05/16/01
Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0097.D

3M Environmental Laboratory
Project # E01-0768 Page 2



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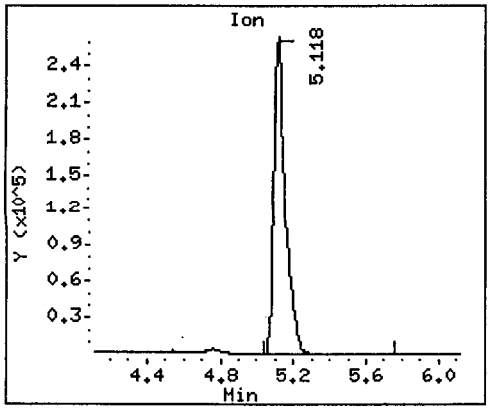

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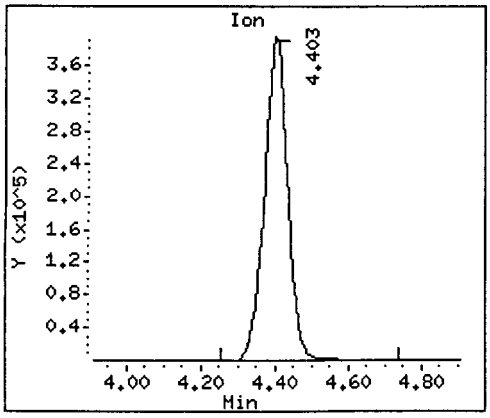

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KH 05-16-01

Initial Date

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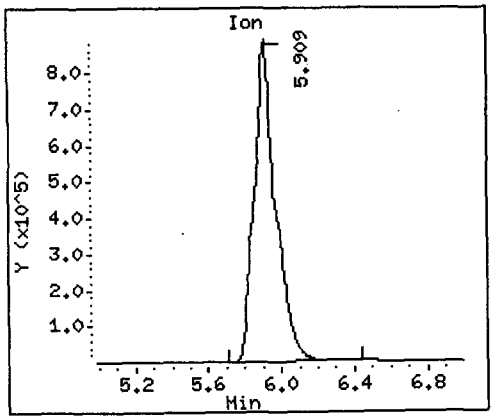
3M Environmental Laboratory
Project # E01-0768

3 FOSA



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05-16-01
Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

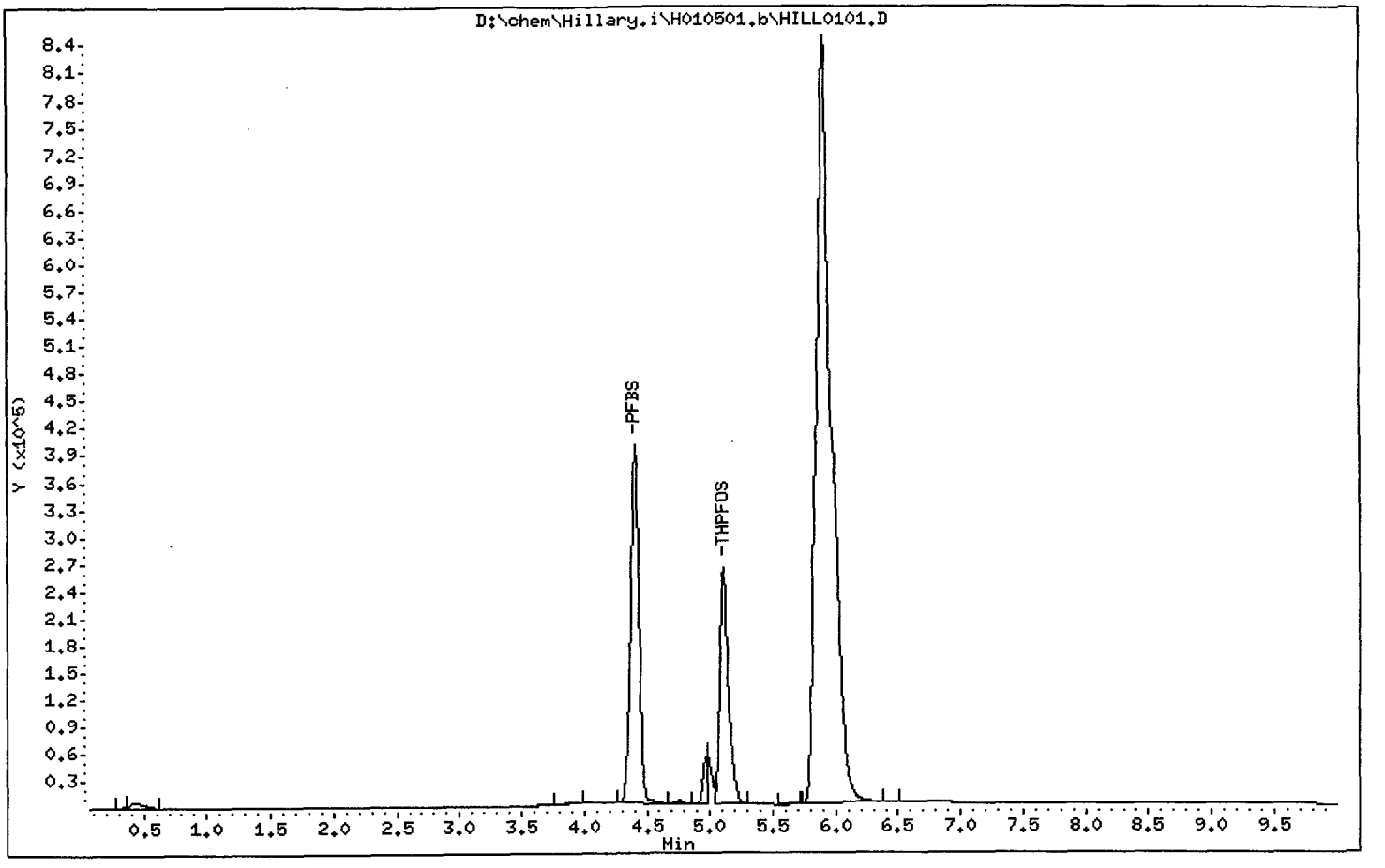
Data file : D:\chem\Hillary.i\H010501.b\HILL0101.D
Lab Smp Id: FOSA-3.2.1
Inj Date : 02-MAY-2001 11:07
Operator : KLT Inst ID: hillary.i
Smp Info : FOSA Urine Solub Day 3 Re-diluted samples 5-01-01
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501a.m
Meth Date : 03-May-2001 11:02 terrell Quant Type: ISTD
Cal Date : 02-MAY-2001 08:53 Cal File: HILL0089.D
Als bottle: 78
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

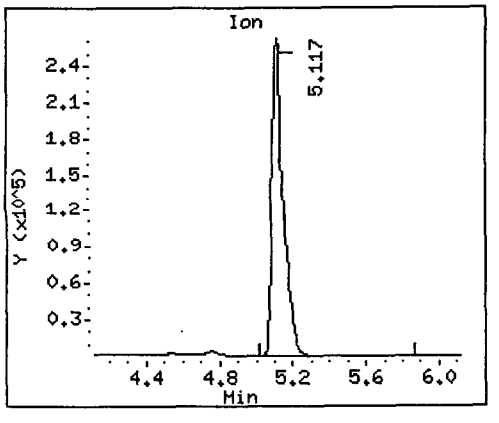
Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 THPFOS	427	5.116	5.118	(1.000)	1170460	254.000	
* 2 PFBS	299	4.409	4.410	(1.000)	1757667	254.000	
3 FOSA	498	5.907	5.993	(1.340)	6990791	256.520	256

Exact Copy of Original

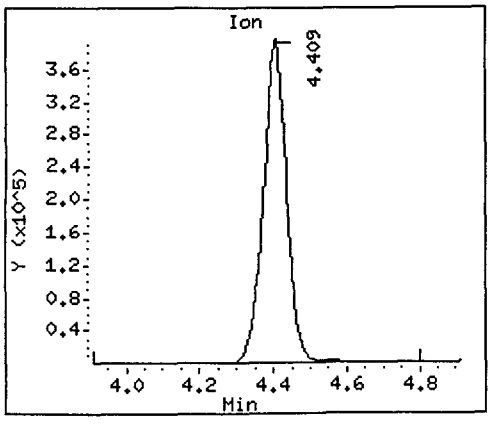
KT 05-16-01
Initial Date



× 1 THPFOS



× 2 PFBS



Exact Copy of Original

KMT 05-11-01
Initial Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0101.D

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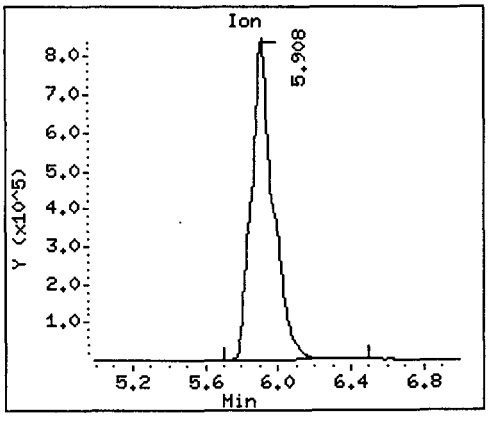
Project # E01-0768 Page 3

3 FOSA



Exact Copy of Original

MG 05-16-01
Initial Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010501.b\HILL0108.D
Lab Smp Id: FOSA-3.3.1
Inj Date : 02-MAY-2001 12:25
Operator : KLT Inst ID: hillary.i
Smp Info : FOSA Urine Solub Day 3 Re-diluted samples 5-01-01
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" H010501.s
Method : D:\chem\Hillary.i\H010501.b\H010501a.m
Meth Date : 03-May-2001 11:02 terrell Quant Type: ISTD
Cal Date : 02-MAY-2001 08:53 Cal File: HILL0089.D
Als bottle: 82
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ng/mL)	CONCENTRATIONS FINAL (ng/mL)
* 1 THPFOS	427	5.124	5.118	(1.000)	1120852	254.000	
* 2 PFBS	299	4.403	4.410	(1.000)	1739019	254.000	
3 FOSA	498	5.915	5.993	(1.344)	7065688	262.600	263

KLT 05-16-01

Data File: D:\chem\Hillary.i\H010501.b\HILL0108.D

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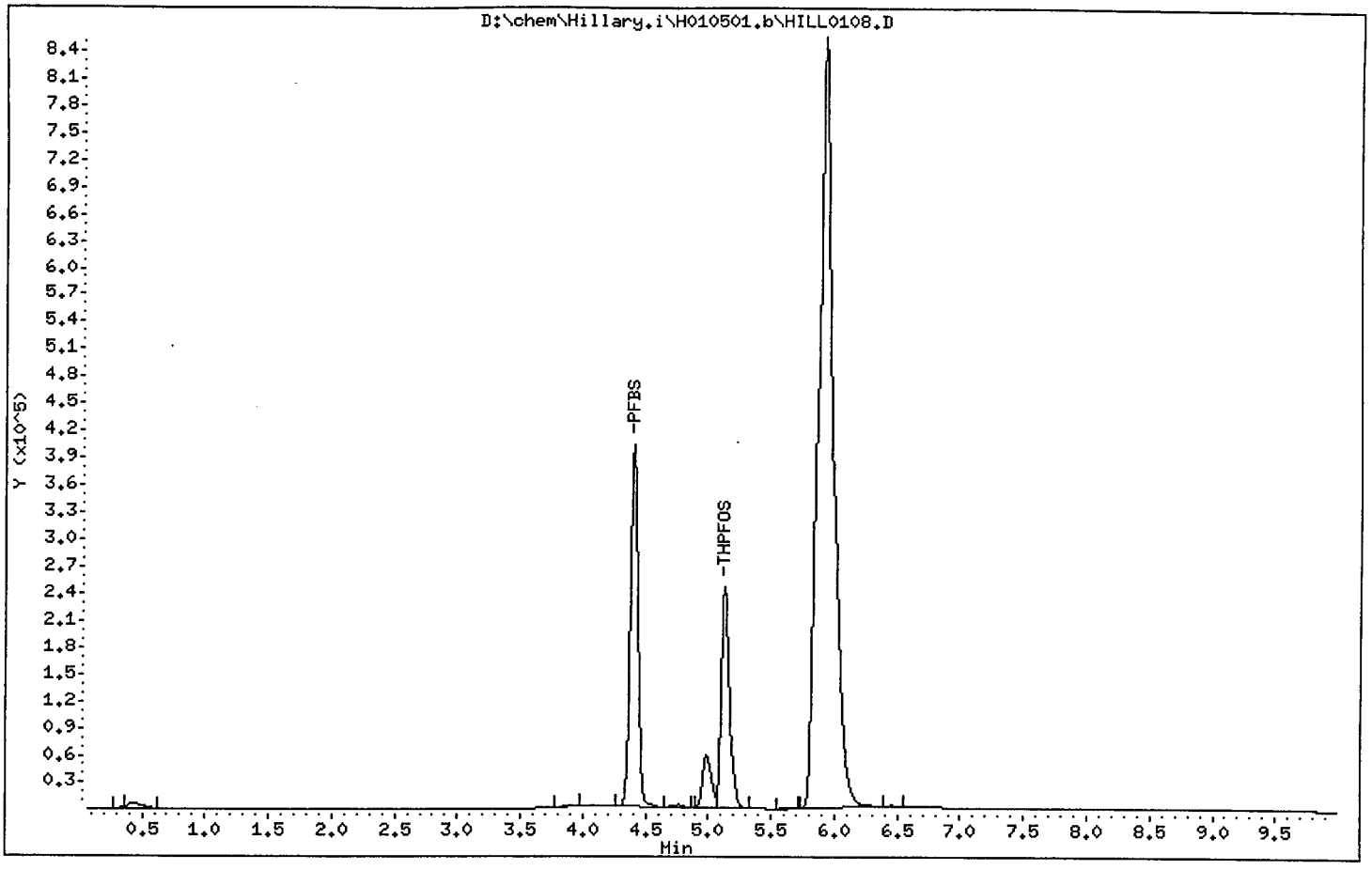
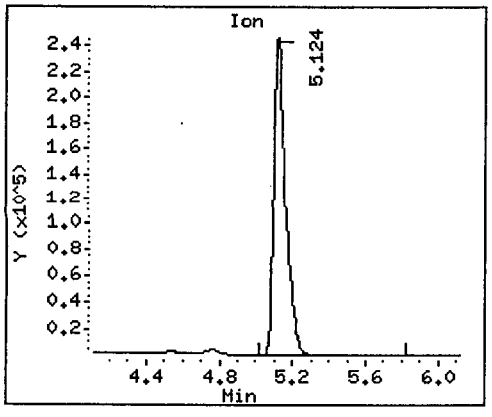
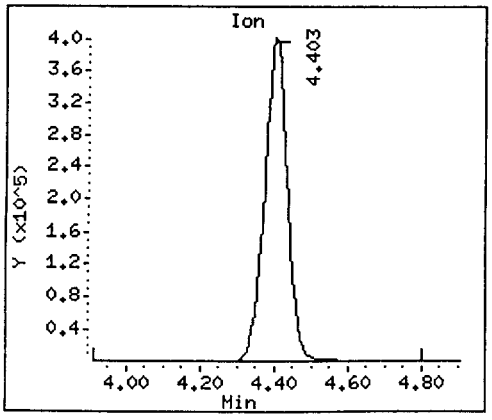


* 1 THPFOS



* 2 PFBS



Exact Copy of Original

KC
Initial

05-16-01
Date

Data File: D:\chem\Hillary.i\H010501.b\HILL0108.D

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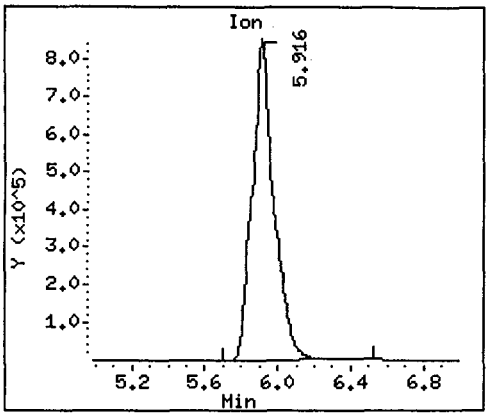
3 FOSA



Original

Initial

Date

3M Environmental Laboratory

FOSA, SE-027, Solubility in Urine

Data file : D:\chem\Hillary.i\H010426.b\HILL0114.D
Lab Smp Id: 01003-25-10
Inj Date : 27-APR-2001 13:55
Operator : KLT Inst ID: hillary.i
Smp Info : 249.6 ng/mL FOSA in MeOH
Misc Info :
Comment : Samples analyzed on HPLC1100 LC/MSD "Hillary" 04-26-01
Method : D:\chem\Hillary.i\H010426.b\H0100426a.m
Meth Date : 30-Apr-2001 14:05 terrell Quant Type: ISTD
Cal Date : 27-APR-2001 13:55 Cal File: HILL0114.D
Als bottle: 10 Calibration Sample, Level: 8
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: all.sub
Target Version: 4.10
Processing Host: W19401

Concentration Formula: Amt * DF * CpndVariable
Cpnd Variable Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ng/mL)	ON-COL (ng/mL)
* 1 THPFOS	427	5.114	5.122	(1.000)	1459549	254.000	
* 7 PFBS	299	4.393	4.387	(1.000)	1999512	254.000	
2 FOSA	498	5.976	5.977	(1.360)	7439185	249.600	256

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KLT
Initial

05/16/01
Date

Data File: D:\chem\Hillary.i\H010426.b\HILL0114.D

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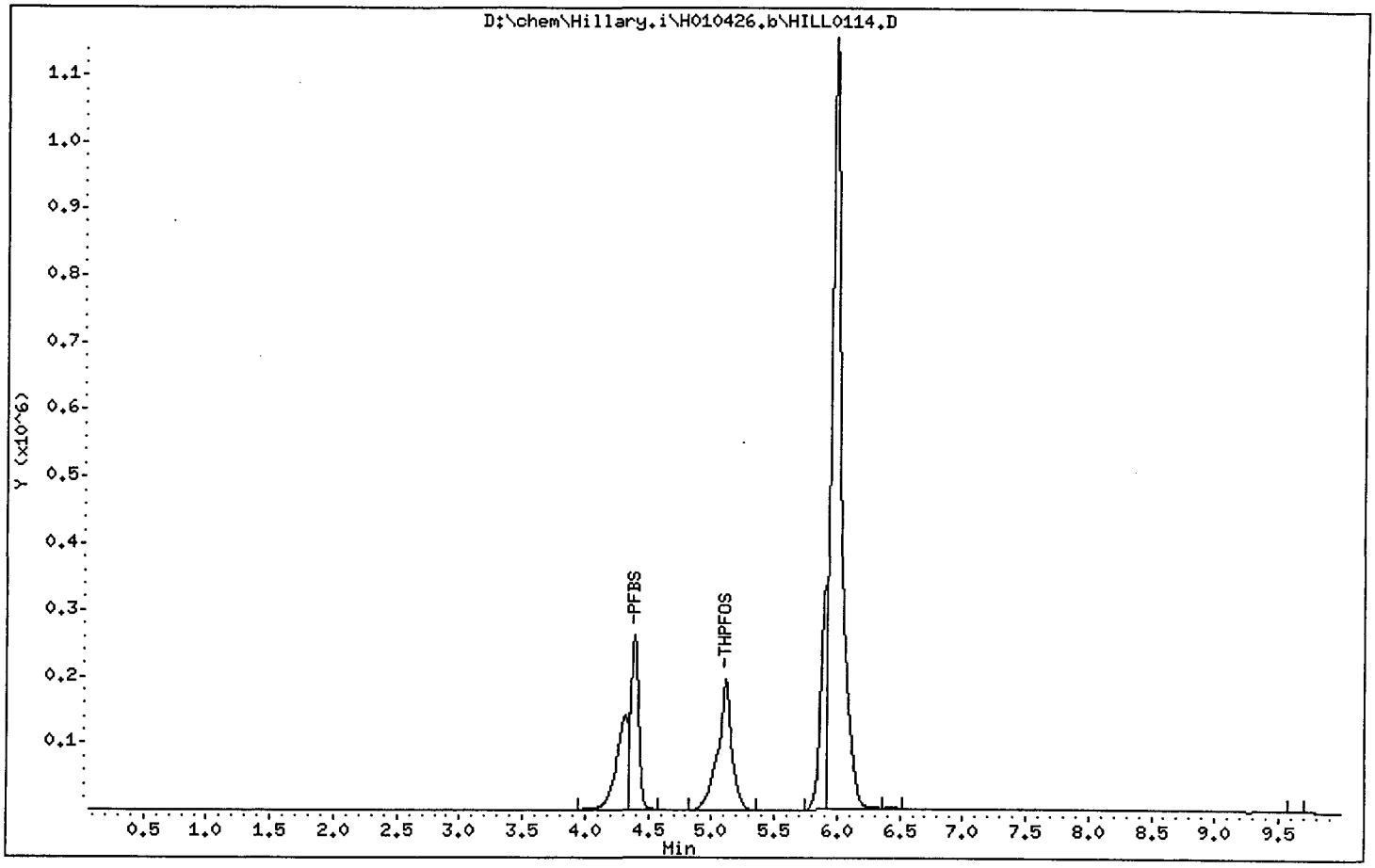
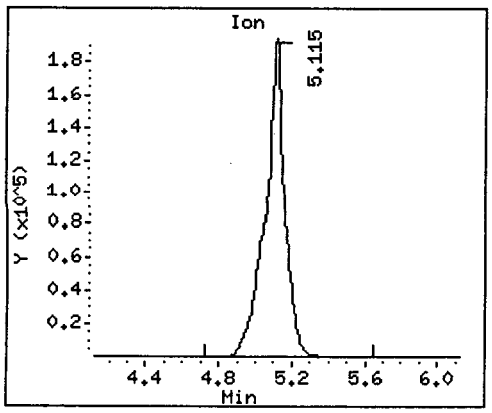
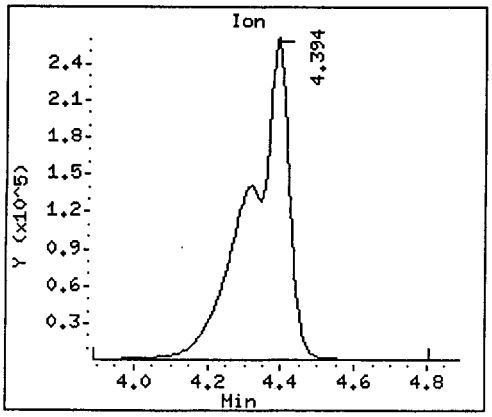


✖ 1 THPFOS



✖ 7 PFBS



Exact Copy of Original

Initial

05/16/01

Data File: D:\chem\Hillary.i\H010426.b\HILL0114.D

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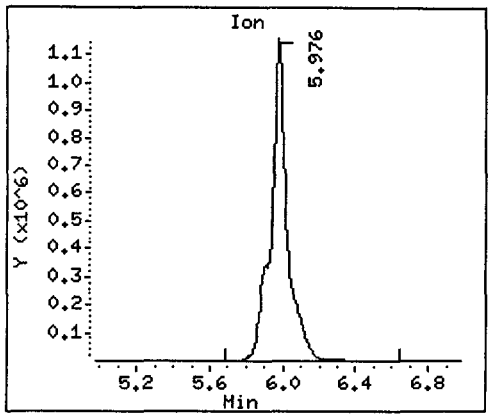
2 FOSA



KLT
Initial

05-16-01
Date

ATTACHMENT D: SAMPLE CALCULATIONS

Purity and dilution correction factors.

- **PFOS, SD-018** 10 µL sample was diluted with 990 µL methanol for a 1:100 dilution, followed by a second dilution to 1:1,000. 100 µL of 1:100 diluted sample was diluted with 900µL methanol. Therefore the on-column concentration was multiplied by a factor of 1,000 for the final reported value. Then, the samples were corrected for the purity of the standards used to analyze the samples. The standard purity was 97.9%. The solubility reported is the amount of PFOS in standard material id# SD-018 that dissolves in water at 23-24 °C.
- **FOSA, SE-027:** 10 µL sample was diluted with 990 µL methanol for a 1:100 dilution. Therefore the on-column concentration was multiplied by a factor of 100 for the final reported value. The standard purity was unknown, therefore no purity correction was done. The solubility reported is the amount of FOSA in standard material id# SE-027 that dissolves in water at 23-24 °C.

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:

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

Average/Standard Deviation /%RSD example calculations:

Sample ID	Concentration µg/mL	Calculations
Rep1	302	Average (302+325+287)/3= 305
Rep 2	325	Std Dev (see above equation)= 19
Rep 3	287	%RSD (19/305)= 6%
Rep 3	146.7	%RSD (19.0/143)= 13.3%

For calculating the precision of two numbers, Relative Percent Difference was used to report the precision of the datum using the following equation:

RPD=(A-B)/((A+B)/2)

Example: Rep1= 2.23, Rep 2=2.13; RPD=(2.23-2.13)/((2.23+2.13)/2) = 28%

Criteria and equations for rejecting suspected outlier values:

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 "$Q_{tabulated}$" with 90% confidence. $Q_{observed}$ = 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. Q-tabulated (90% confidence) for 3 observations (or data points) is 0.886, and 4 observations is 0.679.

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

Description	µg/mL
Sample Conc., Rep1	3.43
Sample Conc., Rep2	2.23
Sample Conc., Rep3	2.13
Range (Rep1-Rep3):	1.3
Gap (Rep1-Rep2):	1.2
$Q_{observed}$ (Gap/Range):	**0.923**
No. observations:	3
Retain?	***No***

Q-Test example conclusion: Since $Q_{observed}$, 0.923, was greater than $Q_{tabulated}$, 0.886, rep 1 may be excluded.